Actual service life prediction of building components

Aagaard, Niels-Jørgen; Brandt, Erik; Hansen, Ernst Jan de Place

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Preface

The ability to predict the service life—the durability—of a new building material is being ardently sought after in many parts of the world. Widespread efforts are correspondingly being made to increase the durability of materials already in wide use. Any estimate of the economic losses resulting from failures in durability lead inevitably to dollar figures so astronomically large that they are usually regarded with incredulity.

R. F. Legget
Proc. of the 1st DBMC

Some 35 years after the 1st DBMC (Ottawa, 1978), sponsored by ASTM, NIST and NRC, durability is an even more important subject. At that time, an accurate service life estimate was considered a condition to life cycle cost (LCC) analysis. Nowadays, service life is also crucial information for the environmental life cycle analysis (LCA). The relevance of durability became widely recognized among scientists: in 1992 the World Business Council for Sustainable Development coined the term “eco-efficiency”, referring to a higher production with fewer environmental and economic resources and less environmental impact. The increase of durability (or service life) of products is one of the critical tools to improve eco-efficiency.

In that period the research community, which was organized into CIB and RILEM working groups, developed a methodology to plan and predict the service life of buildings and constructed assets at the design stage, which was consolidated in the ISO 15686-x standards. As a result of these scientific advances, the degradation factors and mechanisms of most materials and their assemblies are now much better comprehended. Information Technology advances facilitate to mapping the relevant environmental variables by using GIS platforms. Dose-response functions are available, making possible the estimation of degradation rates by combining environmental variables with material characteristics. Even effects of climate change on service life have been a matter of discussion. Altogether, it is an impressive achievement, recorded in the previous 12 DBMC conference. However, despite this scientific progress, the use of these tools in day-to-day production processes, including materials and components R&D, architecture and civil engineering design, and environmental and economic life cycle analysis is still very limited in most countries. Today’s research challenge is not only to increase the knowledge in the field, but also to make this knowledge readily available to the society, by developing more user friendly tools and better educate engineers and architects on the use of these tools.

The 13th edition of the DBMC conference was sponsored by ASTM, NIST, Rilem, CIB and University of Porto. It was joint organized by Polytechnic School of University of São Paulo and Secovi-SP, the most important Brazilian real estate industry’s association, an union that symbolizes our commitment to transfer the knowledge to the society. The support of Saint Gobain Group, Grace Construction Products, Votorantim Cimentos, Gerdau, Fapesp, CNPq and Capes made the conference possible.

We expect that these proceedings will help professionals and academy to incorporate service life planning concepts in their day-to-day decision-making processes.

São Paulo, 25-Aug-2014
Marco Quattrone
Vanderley M. John
Editors
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ABSTRACT

Most of the horizontal surfaces, as roof tops and pavements lose their initial reflectance due to environmental exposure over time. The observed changes are related with microbiological growth and dirt particles deposited on the surface. Those factors cause surface darkening, which is responsible for surface reflection reduction. Biodeterioration and soiling can be partially controlled by periodical cleaning. Cleaning activities are expensive and requires lots of water and detergents. A reflective self-cleaning surface could be a good solution. The aim of this work is to present a literature review of self-cleaning treatment applied on cementitious materials surfaces, as well as is presented a visual qualitative test with methylene blue indicator to show the efficiency of TiO$_2$ as a cleaner agent. The methylene blue solutions were applied over mortar specimens treated with a solution of silane-siloxane overlapped with a 1% and 5% TiO$_2$ solution. The test method was adequate to verify TiO$_2$ efficiency between treated specimens and reference ones by color changes.

1 INTRODUCTION

Cool coatings$^1$ are materials applied on exterior buildings envelope that can contribute to temperature reduction and mitigate the effects caused by the urban heat islands [1]. However, cool materials, as well as conventional materials, modify their properties along the service life. The darkening of the surface, either by soiling and microbiological growth, reduces reflectance performance by absorbing solar radiation leading to surface temperature increase [2].

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$^1$ Are materials with high reflectivity and emissivity.
The semiconductor TiO$_2$ have been investigated as a photocatalytic material capable to maintain reflectance surface due to it self-cleaning properties. When particles with dimensions less than 100nm are achieved by UV radiation, it generates important radical’s reducers that accelerate the degradation of organic material present on the surface. Moreover, it reduces the contact angle between the surface and the rain water drops, turning the washout process easier.

The durability theme is an important issue when using particles of TiO$_2$ as an alternative to extend the durability of cool coatings. In this respect there is few research that includes studies of durability in self-cleaning coatings and/or materials.

The present work proposes an analysis of the literature review on the durability of cementitious cool coatings with TiO$_2$ as a self-cleaning agent. In addition, preliminary results of accelerated aging method using methylene blue to evaluate TiO$_2$ photocatalytic activity.

2 DURABILITY OF COOL COATINGS

The durability of a cool coating depends on the characteristics of the interface between the material and the conditions of exposure and use. Degradation factors of cool coatings are: UV radiation, soiling, temperature and tensions associated with its dimensional variation, winds, rain (which can remove dirt, through leaching or abrasion), moisture and microbiological growth [3]. The relative importance of these factors depends on the nature of the product, as well as the local of exposure.

It is consensus among researchers [3], [4] that there is reduction of reflectance along the years. It is estimated that the loss of reflectance can be in order of 20% in the first year of exposure [4], as a result of soiling.

2.1 Increasing cool coatings durability

To extend the durability of cool coatings there are corrective and preventive methodologies that can be used. Corrective methodology consists in cleaning the coating, to restore the surface properties. The preventive methodology is related to surface treatment, through products capable to prevent and to reduce surface degradation.

2.1.1 Cleaning methods

The degradation can be divided into two categories: (a) reversible by cleaning without damaging the surface removing soiling and microorganisms; (b) abrasive or chemical cleaning that are irreversible, which implies physical and chemical changes of the surfaces.

Cleaning can restore properties like color, texture and even partially surface reflectance [5]. Levinson et al. (2005) concluded that it is not possible to predict the behavior of the reflectance coatings after cleaning because the material deposited on the surface has origins, characteristics, levels and distinct quantities.

2.1.2 Photocatalysis

The photocatalysis can increase durability of cool coatings through self-cleaning action, while maintain the surface reflectance. Self-cleaning materials degrade organic particles on the surface, through the interaction between UV radiation and TiO$_2$. The product of the oxidation and reduction reactions are hydroxyl (OH$^-$) and superoxide (H$_2$O$_2$), responsible for pollutants and organic matter degradation [6]. Additionally, the surface becomes super-hydrophilic, trough the reduction of contact angle between water and the surface. The hydrophilic condition facilitates surface cleaning. This sequence makes the system self-cleaning, deodorizing and antibacterial [7].
2.2 Durability of self-cleaning cool coating

It is well known that TiO\(_2\) (anatase) is stable and long life oxide [8], but applying anatase particles on the surface is certainly less durable than anatase particles incorporated in the coating. The deposits of inorganic pollutants (which cannot be degraded) over the TiO\(_2\) reduce the photocatalysis performance.

Coatings produced by incorporation of anatase into cementitious material can present progressive reduction of performance over time, through overlapping the TiO\(_2\) nanoparticles by cement hydrated products and carbonation products. A fact confirmed by the darkening of the Church Dives in Misericordia facade (Figure 1) in Rome [9]. When TiO\(_2\) is covered it prevent interaction with UV radiation and contact with organic matter to be degraded [10] by photocatalytic effect.

![Figure 1 - Church Dives in Misericórdia after 7 years of exposure (http://kurkista.fi).](http://kurkista.fi)

There is a lack of durability studies to verify photocatalytic effect of self-cleaning cement materials over time. Accelerated aging methods can be used to evaluate materials performance.

3 MATERIALS AND METHODS

3.1 Materials

- TiO\(_2\) from Millenium with an anatase particle dimensions below 40nm.
- Octitrietoxisilano from Dow Corning Corporation.

3.2 Mortar specimens preparation

Expanded polystyrene boards were used as mold of mortar specimens (Size: 9 cm x 9 cm x 2 cm), according with. Mortar mix proportion: dry materials ratio of 1:1:9 (cement: lime: sand) and 21% of water, based on total dry weight. Post treatments were applied, at least, 90 days after molding. The TiO\(_2\) concentration used in dispersion was 1% and 5%, in weight, water or silicone based.

3.2.1 Post treatment procedure

Untreated specimens were used as references. Post treatments were applied by brushing according this sequence: (i) TiO\(_2\) water based dispersion of 1% and 5% (g/L); (ii) Octitrietoxisilano dispersion of silicone based water repellent of 10%, in weight; (iii) TiO\(_2\) plus silicone base water repellent of 1% e 5%, in weight;

For all post treatments the dispersion consumption was 300g/m\(^2\) and 500g/m\(^2\), resulting in a TiO\(_2\) consumption from 3g/m\(^2\) to 5g/m\(^2\) on the specimens at a 1% concentration, and from 15g/m\(^2\) to 20g/m\(^2\) at a concentration of 5%. After the treatments, a quarter of each
specimen was coated with water based dispersion of methylene blue in a concentration of 1% g/L. Another quarter was also stained with methylene blue in alcohol base dispersion, also in a concentration of 1% g/L. The others two quarter were not stained with methylene blue.

3.3 Accelerated aging test

The specimens were submitted to continuous accelerated aging cycles, each of them with twelve hours exposure (four hours of Infrared Radiation (IR), four hours of wetting and four hours of UV radiation). The specimens were placed according to the Figure 2.

![Figure 2](image)

Figure 2 – Schematic representation of specimens placed in the camera.

3.4 Specimens evaluation by visual analysis

The photocatalytic properties were evaluated by the color change\(^2\) kinetics over the 100 cycles of artificial aging (50 days in the chamber). To evaluate color change it was used a color scale reference (Figure 3) to compare to the specimens colors photographed every 25 cycles. For each treatment ten specimens were evaluated. After 50 cycles, a new application of staining was applied. The second methylene blue application was conducted on half of the specimens to evaluate whether there was loss of performance through kinetics reduction. The two color scales normalized were used to identify color changes in specimens with methylene blue in alcohol and in water. Both scales considered 100% when methylene blue was not degraded and 0% when methylene blue was fully degraded.

![Figure 3](image)

Figure 3: Color scale (a and b) used to evaluate the intensity of the methylene blue through the accelerated artificial aging program. (a) dispersed in water; (b) Dispersed in alcohol.

\(^{2}\) The color evaluation is qualitative, based on visual analysis of dye color intensity along the accelerated aging compared to a color scale.
4 RESULTS

Figures 4 and 5 show kinetic of degradation. The dye, diluted in water or alcohol showed similar behavior independent of the initial rate.

The untreated specimens showed discoloration of 25% related with initial color along the 100 cycles used in the experiments. Samples treated with silicone-based water repellent it was observed reduction of dye adsorption. The color measurement was done in methylene blue solution after leaching by water flow of accelerated aging cycles.

It was observed that all specimens treated with TiO$_2$ presented a higher reduction in color intensity. Higher TiO$_2$ concentration (dispersion), higher the staining reduction.

All specimens treated with water repellent and TiO$_2$ showed higher staining reduction and after fifty cycles of exposure, the surface of specimens practically does not show blue color (dye). In addition, the 5% concentration showed a quite similar performance to 1%. The TiO$_2$ used in the study showed low photocatalytic activity and a behavior similar to reference specimens.

![Figure 4: Kinetics of color intensity reduction of substrates treated with dispersions of TiO$_2$.](image1)

![Figure 5: Kinetics of color intensity reduction of the spots on treated substrates with silicone-based water repellent plus TiO$_2$.](image2)
Comparing the results obtained on exposed and unexposed specimens (Figure 8), it was observed significant reduction of color intensity, showing methylene blue degradation, whereas in treatments with TiO$_2$ these differences were smaller, confirming that the photocatalytic processes are still happening. Similar results was also identified by [11].
The reduction is more important in the treatments that did not use the water repellent as bond agent enabling some leaching of TiO$_2$ particles as a result of poor adherence, or even by deposits of hydrated products on the semiconductor particles that creates a barrier to UV radiation.

Specimens treated with silicone-based water repellent as bond agent, did not present differences in intensity of color changes.

![Figure 8: Kinetics reduction of color intensity on specimens previously aged by 50 cycles.](image)

5 CONCLUSION

**Literature review**

1. There are two methods of TiO$_2$ application for self-cleaning purposes. One is to incorporate TiO$_2$ in a coating and the other is applying TiO$_2$ as post treatment.
2. Accelerated tests cannot be correlated to the natural exposure test, which are much more complex, because they present more variables. Accelerated tests use dye as organic matter to evaluate TiO$_2$ photocatalysis. However, those tests do not take into account the microbiological growth that is responsible for surface darkening, which have higher effect on reflectance performance.

**Experimental study**

1. Accelerated program showed to be an effective method to evaluate photocatalytic activity through methylene blue degradation and allowed to differentiate the kinetic of staining reduction, of specimens treated from the untreated ones. Visual color scale demonstrates to be effective as a qualitative and easy to evaluate the photocatalysis performance;
2. Water repellent is an important agent to promote TiO$_2$ particles fixation on the specimens and increase photoactivity durability.
3. Specimens treated with 1 and 5% of TiO$_2$ showed similar performance of methylene blue degradation;
ACKNOWLEDGEMENTS

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EVAULATION OF A MODULAR FRAMEWORK FOR PERFORMANCE-BASED DURABILITY ENGINEERING THROUGH ASSESSMENT OF A COASTAL REINFORCED CONCRETE STRUCTURE

Madeleine M. Flint (1) and Sarah L. Billington (1)

(1) Department of Civil and Environmental Engineering, Stanford University, Stanford, CA – flint@stanford.edu; billington@stanford.edu

Abstract
Traffic emissions caused by corrosion of a hypothetical reinforced concrete composite bridge located in coastal Norway were assessed following a performance-based durability engineering framework. Emissions caused by load rating due to corrosion damage were found significant in the structure’s total life-cycle emissions. Sensitivity and scenario assessments demonstrate that emissions predictions were influenced by model selection and assumed socioeconomic trends. The case study was used to evaluate the usefulness of the performance-based durability engineering framework and to identify aspects needing improvement.

1 INTRODUCTION

Several previous life-cycle assessments indicate that traffic delays due to maintenance, repair and rehabilitation (MR&R) activities may contribute significantly to the environmental impacts of a bridge [1-3]. Given these findings, management plans would ideally account for the traffic impacts of various MR&R strategies to optimize sustainability. However, consideration of the impacts of future MR&R is difficult because of uncertainties related to projections of future population, technologies, and economic trends. In order to avoid unnecessary material use and traffic delays related to MR&R, a durable structure design is desirable; alternately, structures should be designed to need only low-traffic-impact repairs.

Researchers and practitioners have recognized the difficulty of ascertaining which systems will prove durable, and have proposed methods for service life design. Climate change exacerbates the challenge of service life design, as anticipated changes in precipitation, sea level, and temperature may accelerate deterioration such that systems that perform well today may be insufficiently designed for the climate conditions in future years. Modelling the behaviour of a system over its predicted service life can be used to demonstrate its durability. As recognized by Bastidas et al. [4], among others, the complexity of real-world exposure, coupled deterioration mechanisms, and lack of data make such modelling difficult.

A framework [5] was developed by the authors and colleagues for use in sensitivity assessments to identify significant sources of structural performance uncertainty, and to
compare designs and repair strategies. The framework models three stages of interest to structural durability and sustainability: exposure, deterioration and repair, and resulting economic, social, and environmental impacts over the life of the structure. Implementation of this “performance-based durability engineering” (PBDE) framework has indicated its potential usefulness for study of archetypal structures and for identification of research gaps [6]. These studies have also identified problematic computational expense and areas in which scarce data is available to support assumptions.

This paper presents a theoretical case study on emissions caused by load rating of a corroding coastal Norwegian bridge. The case study was used to explore the effects of several sources of uncertainty, including uncertainty in processing of exposure boundary conditions, deterioration model selection, traffic assumptions, and emissions model selection. The influence of boundary condition processing was studied through the extension of a previous study [7], in which permutations of sampling of meteorological data were assessed. This paper runs through the sequential analysis stages of a PBDE framework in order to identify important sources of uncertainty. Uncertainty attributed to each of the analysis stages could be integrated to obtain a full probabilistic description of decision information, as suggested in [5]. This paper evaluates the ability of the PBDE framework to generate research questions through its comprehensive modelling approach. Results demonstrate how modelling, data analysis, and baseline scenario assumptions alter estimates of the sustainability of a bridge or MR&R strategy. Conclusions highlight difficulties that arise when combining models, and suggest future work that may mitigate identified challenges.

2 SELECTED BACKGROUND

The extent to which corrosion of steel reinforcement in concrete impacts the capacity of a structural component and reduces its factor of safety are studied through structural analysis and time-variant reliability assessment. A number of reliability studies predict the time-variant probability of structural failure, defined in terms of service or ultimate limit states, e.g., [4,8]. In practice, structural load-rating or load-limiting of heavy vehicles can ensure safety when a reliability analysis is not performed.

Data from recent bridge life cycle assessments (LCAs) was used in this study [1-3,9-11]. A consistent LCA methodology, assumptions, and phase boundary has yet to emerge. Additionally, existing studies vary in terms of planned lifetime and maintenance and repair activities. To the authors’ knowledge no studies consider load rating and related emissions.

The importance of processing of heat and mass boundary conditions has been recognized in recent studies of concrete infrastructure [4, 6]. A selection of processing schemes of boundary condition data was explored in [7], which found that the heat, moisture, and chloride ion ingress model studied was sensitive to data resolution and processing scheme. Meteorological data taken at permutations of a daily hour resulted in corrosion penetration discrepancies up to 30% from simulations using 3-hour data.

3 METHODS FOR CORROSION LOAD RATING EMISSIONS STUDY

The hypothetical case study structure was a reinforced concrete composite bridge located in coastal Norway. Traffic, structural, and emissions models were linked to predict emissions caused by load rating of the structure after the onset of corrosion. Simulations were carried out using a coupled heat and mass transport model at the original resolution of available regional
meteorological data, three hours, referred to as “3h-o” or “baseline,” and for seven permutations, “P,” where the data point collected at a specified hour was used to represent the conditions for the entire day. Statistics were recorded for the individual permutation simulations. An additional study analysed the baseline data using an effective diffusion model, which did not predict corrosion initiation over the structural lifetime [6].

Several alternate scenarios explored sources of uncertainty. Emissions related to load rating and heavy vehicle detour were compared to repair of the corroded structure by concrete cover replacement. Both alternatives were compared to emissions from other life cycle phases taken from literature. To study sensitivity to assumptions, additional scenario studies assumed constant traffic over time, doubled traffic volume, or a quadrupled detour length. Results from the main emissions model were compared to results obtained using BridgeLCA to explore sensitivity to emissions models [12]. Finally, emissions were re-calculated using time-adjusted warming potential (TAWP), described in [13] and in Section 3.4. Flint [6] contains details on assumptions, models, and parameters, and their effects are discussed in Section 5.

3.1 Structure and location

It was assumed that the structure is located between the Vestvågøy and Flakstad municipalities on the E10 highway, the major route through the Lofoten region of Norway. A hypothetical coastal site was selected where a detour of 4.9 km is possible.

Results from the one-dimensional transport and corrosion analysis were mapped to a structural section with Eurocode 2-based design detailed in [14] and shown in Figure 1. Total deck area for the 200m-long, three-span, two-lane bridge was 2400 m². The concrete deck, part of a composite system, had a concrete composition (C35/45) and cover depth in agreement with the parameters assumed in the deterioration model [8]. It was assumed that the bottom surface of the deck was exposed to sea salt deposition and “ambient” meteorological exposure, leading to corrosion of 16 mm-diameter reinforcement. The cover depth and concrete quality were similar to those used in Norwegian bridges in the 1960s and 1970s; the bridge was assumed constructed in 1960 with decommissioning planned for 2040.

3.2 Traffic data and traffic models

It was assumed that heavy vehicles crossing the structure fell into three classes: trucks on trips originating or ending in the Nordland commune, local public buses, and tourist bus coaches. Data on trucks, buses, and passenger vehicles for Norway, and the Nordland commune, was obtained from Norway's Statistics Bureau. Based on data from 2000-2012, the number of trucks crossing the structure and their capacity distribution was estimated at 2150 heavy trucks per year. Details on the truck and traffic modelling, and subsequent models and data, are provided in [6]. In order to explore the effect of traffic growth, a projection of local bus routes was made based on Norway’s “MMMM” population scenario, which predicts “medium” fertility, mortality, migration, and domestic migration. Assuming that 2/3 of the population growth would be absorbed by existing capacity, a quadratic curve was fit to the 2013 schedule of 50 crossings per week: 55 were predicted in 2020 and 58 in 2040. Data on bus coaches operating in Lofoten was scarce; 280 coach crossings per year were assumed.

3.3 Safety and load rating models

Limit states based on Eurocode 2 were selected to identify when load rating would be undertaken to avoid loss of safety. The design ultimate limit state of the slab in the transverse
direction considered the Eurocode 2 “LM1” load case, which pairs a uniformly-distributed traffic load with in-tandem lane loads representing heavy vehicles stopped on the bridge. This load combination results in the maximum positive bending moment at midspan of the slab between the two girders. Structural safety predictions required analysis of the response of the slab to characteristic permanent dead loads and the LM1 combination. Corrosion was assumed to result in uniform loss of reinforcing area, cracking of the cover concrete, and embrittlement of the reinforcement such that failure was assumed to occur at yield strength. Pitting corrosion and the effects of corrosion on bond strength and confinement were not considered. The equivalent uniform reduction of reinforcement radius at which the structure no longer met the design reliability for the tandem vehicle load case was calculated. A drop below the required capacity is reached at a reduction of 1.5 mm, at which point the structure was assumed closed to buses and trucks with carrying capacity greater than 15 tonnes.

3.4 Emissions models

Two emissions models were used: the detour study used data from Norway’s emissions inventory [15] and considered emissions related to detour travel distance; traffic emissions for routine MR&R and corrosion repair were calculated using BridgeLCA [12]. Both models required assumptions regarding vehicle age, mix of fuel types, fuel efficiency, and emissions factors, which was obtained from the emissions inventory [15] and from recommendations by the US Environmental Protection Agency [16], and in certain cases default values from BridgeLCA. BridgeLCA calculates emissions using empirical equations describing emissions as a function of engine load for various vehicle types [12]. Carbon dioxide, methane, and nitrous oxide were modelled in the detour study, and converted to global warming potential (GWP), CO2e, according to the 100-year factors as in [1,10]. Use of the GWP metric is problematic for bridge assessments because all emissions are assumed to occur at the time of construction, which penalizes MR&R and end-of-life (EOL) emissions. Various adjustments exist; time-adjusted warming potential (TAWP), developed by Kendall [13], was also used.

Emissions related to life cycle phases of interest were obtained from life cycle assessments of other composite bridges and scaled. The phases included construction and construction materials (C&M) [3], planned MR&R actions [3,9,10], corrosion repair [11], and EOL [10]. Scaling factors included bridge deck area, average daily traffic, detour length, and service life. Because the spread in predicted emissions was large, the study most similar to the case study structure was selected as the appropriate value, except for the MR&R phase, for which the literature varied by three orders of magnitude and three studies [3,9,10] were averaged.

4 RESULTS

4.1 Influence of boundary condition resolution and processing on emissions prediction

The time of load rating action and detour emissions for the 3h-o baseline, selected boundary condition permutations, and statistics of the permutations are presented in Table 1. The maximum positive discrepancy in emissions predictions occurred for P00:00 (sampled at midnight); the maximum negative discrepancy occurred for P15:00. The majority of the permutation simulations varied ±10-15% from the baseline, indicating that discrepancies in prediction of deterioration translated to differences in emissions predictions. However, the mean of the individual simulations and the P06:00 and P12:00 simulations had lower relative
difference, within 5% of the baseline result, which was within the margin of error of other parameters, e.g., corrosion rates.

Figure 1: bridge cross-section simplified from [14] and used in structural analysis

Table 1: Time to load rating and detour-related emissions for various processing schemes

<table>
<thead>
<tr>
<th>Study</th>
<th>Predicted load rating [yr]</th>
<th>Emissions [tCO2e]</th>
<th>Relative difference 3h-o</th>
<th>Difference 3h-o [tCO2e]</th>
</tr>
</thead>
<tbody>
<tr>
<td>3h-o</td>
<td>2001</td>
<td>1120</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>P00:00</td>
<td>1996</td>
<td>1250</td>
<td>12%</td>
<td>130</td>
</tr>
<tr>
<td>P15:00</td>
<td>2007</td>
<td>960</td>
<td>-14%</td>
<td>-160</td>
</tr>
<tr>
<td>All P statistics</td>
<td>2003 ± 3</td>
<td>1070 ± 90</td>
<td>-5%</td>
<td>-50</td>
</tr>
</tbody>
</table>

4.2 Comparison of detour emissions to other bridge life-cycle phases

Table 2 presents emissions from different life-cycle phases for the study assuming that heavy vehicles are detoured and the study assuming that the structure is repaired. Emissions for both of these studies were obtained using the 3h-o deterioration analysis. Figure 2 compares data from Tables 1 and 2, and additional simulations based on alternate population trends, traffic scenarios, and emissions models. Corrosion repair, including delays due to speed reduction, was predicted to yield 7% lower impacts over the life-cycle. Detour emissions formed the third largest contribution to the total life cycle impact, and were an order of magnitude larger than emissions related to MR&R traffic and end of life (EOL).

Table 2: Emissions at bridge life cycle phases, adapted and scaled from literature [tCO2e]

<table>
<thead>
<tr>
<th>Study</th>
<th>C&amp;M</th>
<th>MR&amp;R</th>
<th>MR&amp;R Delay</th>
<th>Corrosion Repair</th>
<th>Corrosion Delay</th>
<th>EOL</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>3h-o detour</td>
<td>6600</td>
<td>2070</td>
<td>50</td>
<td>0</td>
<td>1120</td>
<td>190</td>
<td>10,000</td>
</tr>
<tr>
<td>3h-o repair</td>
<td>6600</td>
<td>2070</td>
<td>50</td>
<td>190</td>
<td>160</td>
<td>190</td>
<td>9270</td>
</tr>
</tbody>
</table>

4.3 Comparison of total emissions under different scenarios

Emissions predictions from the alternate scenarios are presented in Figure 2. Of the scenarios studied, the emissions were most sensitive to assumptions of traffic volume and detour length.
Calculation of TAWP as opposed to GWP, or use of the BridgeLCA emissions model, changed the results on the order of ±10%. While the permutation P6 led to a -30% discrepancy in corrosion penetration at 50 years and a -14% difference in detour emissions, the discrepancy in life-cycle emissions was only -2%. Assumption of constant population and bus traffic had negligible effect on total GWP. No scenario preferred load rating to repair.

Figure 2: Full life-cycle emissions over phases described in Table 2 for detour and repair strategies, including various boundary condition processing permutations, population trends, traffic and detour scenarios, and emissions and life-cycle impact models

5 DISCUSSION

5.1 Case study results
Taking the 3h-0 prediction as an accurate and complete description of the safety of the structure, the onset of load rating occurred at a reasonable time considering past experience with similar structures [6]. The late onset of load rating gives some assurance that the assumption of load rating as a possible corrosion response strategy was reasonable.

Emissions related to detour were consequential in the context of the entire life-cycle, and would ideally be considered in environmental impact reports. The difference in repair and detour emissions predictions was small compared to the total life-cycle emissions, and given the assumptions and variance between studies used for the comparison, no general conclusions may be drawn regarding the suitability of one approach over the other.

5.2 Importance of assumptions, exposure resolution, and model selection
5.2.1 Influence of population, technologic, and economic assumptions
While the assumption of constant bus volume herein did not significantly alter the total life-cycle or even the detour emissions, assumption of constant traffic volume may not be reasonable in other contexts. Changes in population and traffic growth are likely to vary regionally, and population sensitivity studies are recommended in LCA as appropriate.
In this study the vehicle mix and fuel efficiency of vehicles was assumed constant, and given the growing popularity of alternative fuel and zero-emissions vehicles in Norway, this assumption could prove conservative. Adoption of new, “clean” technology is sensitive to government policy and mandates; projections of reductions in emissions factors for the vehicle population can likely be improved by integration of policy plans.

On the economic front, the truck model assumed static volume, whereas any trends towards different vehicle types or capacity would alter the projection of trips. Prediction of significant changes in the regional economy would also affect trips and emissions, and could cause larger increases in emissions than the 12% calculated herein for doubled truck volume.

5.2.2 Influence of exposure resolution and model selection

In this study, the processing of boundary conditions had negligible effect on total emissions predictions, although larger deterioration discrepancies from other processing methods suggest that this may not always be the case [6]. Changing the emissions model or the GWP model altered total emissions predictions by a more substantial amount, 10%.

It is likely that changing the structural analysis model, or implementing a time-variant reliability model to determine load rating, would decrease the predicted time to load rating. Studies of time-variant reliability find that estimations of failure probability can double or quadruple when spatial variation and pitting are considered [8].

5.3 Assessment of PBDE framework in the case study context

Aleatoric uncertainty in emissions, exposure, and deterioration was not considered herein and yet the uncertainty in predicted emissions was high due to variation in the LCA data. Use of the full PBDE methodology would require inclusion of these sources of uncertainty, and thereby increase computational demands and the spread of total predicted emissions.

6 FUTURE WORK AND CONCLUSIONS

Three conclusions are drawn for the case study and for the influence of boundary condition processing on emissions predictions. (1) Exposure data processing can and does affect emissions predictions, although to a lesser degree than its impact on deterioration predictions. (2) Model selection is critical: different models can predict divergent emissions outcomes. (3) Load rating should be included in life-cycle assessment when it is a possible response to deterioration, as the contribution to the total GWP can be on par with routine MR&R.

More generally, even simple LCAs like the one studied herein require large amounts of data, which may not be available, and many assumptions, which are not yet standardized. Uncertainty in future socioeconomic and technologic trends may widen the predicted distribution of decision information. Other researchers have recognized these issues and have performed sensitivity assessments; scenario combinations are not regularly considered, and may have nonlinear effects. The development of standardized recommendations for infrastructure LCA, as has occurred in commercial industries through product category rules for environmental product declarations, would ease the comparison of alternative designs. Given the variance in bridge types, use, and planned life, multiple standards are likely needed.

Suggested future work related to the case study includes: implementation of queuing/traffic delay models and mobile emissions models; thorough studies on the impact of population/technologic/economic trends; and a full life-cycle assessment of the case study structure to better assess the relative importance of detour emissions.

Finally, some conclusions may be drawn with regard to the current and potential usefulness
of PBDE. (i) Scenario-consistent studies, in which climate trends and decision information trends are considered concurrently, are of interest, and require alteration of the current approach for integrating the PBDE analysis stages. (ii) More work is required in exposure and impact modelling to make a scenario-consistent approach feasible. (iii) While the drawbacks of data and computational intensity were recognized at the outset of the project, the sensitivity assessments presented here and elsewhere [6] have accentuated these concerns. (iv) Despite the challenges highlighted, PBDE assessments have raised new research questions, suggesting that there is benefit to developing comprehensive frameworks.

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Prediction of Sealant Modulus Change due to Outdoor Weathering

C.C. White\(^{(1)}\), K. T Tan\(^{(1)}\), D.L. Hunston\(^{(1)}\), A. Pintar\(^{(1)}\) and J. Filliben\(^{(1)}\)

(1) National Institute of Standards and Technology, Gaithersburg, MD, USA—christopher.white@nist.gov, kar.tan@nist.gov, donald.hunston@nist.gov, adam.pintar@nist.gov, james.filliben@nist.gov

Abstract

A empirically-based model has been created to predict the change in modulus for a sealant exposed to outdoor weathering. The underlying high precision data supporting this model was obtained using the NIST SPHERE (Simulated Photo degradation by High Energy Radiant Exposure). The NIST SPHERE can independently precisely control each of the four primary components of the outdoor exposure: Temperature, Humidity, Ultraviolet Radiation and Mechanical Loading. The time-dependent modulus of the sealant samples were characterized using ASTM C1735-11 before and after each exposure period. The resulting empirically-based model produces two major results: the statistical significance of each of the weathering components and a prediction of modulus change for any location, provided a historical weather file is available. This sealant model has shown that all four weathering factors, listed above, are significant contributors to modulus change in sealant. Additionally, the geographical predictions allow for independent verification of these models.

1 INTRODUCTION

Verified accurate prediction of in-service performance of sealant materials is critical for sustainability calculations, life cycle cost analysis, performance based building codes, and decreasing barriers to innovation.[1] Currently, sealant materials are evaluated on initial or installed cost due to a lack of predictive methods. Often materials are qualified for use in the marketplace based on passing a consensus-developed threshold test, or long-term outdoor exposures. Neither of these types of tests allow for prediction of actual performance in-service.

The work at NIST has focused on developing methods that have predictive capability. The components of a predictive method include: creating dose-response data, assembling a database of that dose-response data, and generating predictions based on that data. Each of these topics will be covered in sections that follow.

2 CREATING DOSE-RESPONSE DATA

There are two general components necessary for creating a dose-response database: characterizing the property of interest and creating a well-characterized environmental
exposure. These areas have been addressed previously in the literature [2-6], but a brief overview is included here.

The physical property of interest in sealants is time-dependent modulus. The modulus is a ratio of the stress/strain. The strain on the sealant results from changes in the physical dimensions of the sealant relative to the initial dimensions. The stress in the sealant material is the force resisting that dimensional change. The time dependant modulus is obtained from monitoring the modulus over time.[5] The time-dependent modulus is a relatively easy property to measure macroscopically, and changes can be ascribed to specific molecular changes within the material that often precede failure.[7, 8] For example, if the time-dependent modulus increases, the same displacement (i.e., what the building is imposing on the sealant due to thermal changes) increases the amount of stress within the sealant. If that stress becomes greater than either the adhesive bond strength or the tear strength of the material, the sealant will no longer adhere at that location. The time-dependent modulus of the sealant is measured using the ASTM C1735-11 test method illustrated in Figure 1. This is a stress relaxation test method. With an accurate characterization method, the next step is creation of a well-characterized environment.

![Figure 1. Imposed strain on the sealant during the characterization of ASTM C1735-11. The two initial strains are to deal with the Mullins affect.](image)

By consensus of the industry, temperature, humidity (moisture), ultraviolet radiation, and mechanical strain are considered to be the most important weather exposure components.[1, 3, 4] The previously described NIST SPHERE (Simulated Photo degradation by High Energy Radiant Exposure) can precisely control and monitor these four elements of a simulated weather exposure.

A model sealant formulation was supplied to NIST for this work. This formulation has been prepared by the procedure specified in ASTM C719. While the details of this formulation are unknown to us, it is known to be representative of a common commercial sealant formulation. Since the goal is to develop a standard test method, the knowledge of the formulation is not critical.
3 ASSEMBLING A DATABASE OF DOSE-RESPONSE DATA

The model sealant formulation is characterized using the ASTM C1735-11 test method.[9] The sealant is then exposed in the NIST SPHERE to well-controlled temperature, humidity, mechanical strain and ultraviolet radiation conditions. After one month of exposure, the sealant is again characterized using ASTM C1735-11. An example result is shown in Figure 2. Here the average and relative standard error of four samples is shown before and after a one month exposure to 30°C, 25% imposed strain, and 75% relative humidity. From this figure it is clear that the shape of the time-dependent modulus has not changed, but a vertical shift has occurred. The vertical shift in this curve is attributed to a change in the degree of cross-linking of the sealant. An important feature of Figure 2 is that the shape of the curve does not change. With this condition, a single point, arbitrarily chosen to be the modulus at 100 seconds, $E_{100}$, is used to represent the entire curve. A further simplification is to report only the ratio of the $E_{100}$ modulus (after) / $E_{100}$ modulus (before). This data is collected for a wide range of conditions shown in Figure 3. This data is subsequently used to generate the predictions of service life.

![Figure 2](image)

**Figure 2.** Results of the ASTM C1735-11 test before and after a one month exposure at 25% imposed strain, 30°C, and 75% relative humidity.
4 GENERATING PREDICTIONS

The database illustrated in Figure 3 is used to build a empirically-based predictive method. The empirically-based method splits the data into two parts, with UV radiation and without UV radiation. The data in each of these sub-databases is then used to generate a response surface with three independent variables using local regression [10]. Numerous statistical tests were performed on both the data and the response surfaces generated.[11]

Hourly weather data consisting of the recorded temperature, humidity and ultraviolet radiation were obtained from a NOAA database.[12] The imposed strain was calculated based on a maximum rated strain (25%) in tension occurring at the expected highest temperature and equivalent maximum rated strain (25%) in compression at the lowest expected temperature. The strain is expected to be linearly related to ambient temperature. For each hour, the rate of change of the $E_{100}$ modulus was predicted for the recorded climatic conditions at that location using the empirically determined response surface. That rate of change is then scaled appropriately to correspond to one hour of outdoor exposure. The scale factor must be calibrated using outdoor data, which is a continuing effort. This process is repeated for each hour for each of the 29,000 hours from 2007 to 2011. The resulting change in the sealant modulus for each hour is tabulated as a running summation. This summation along with an un-conservative determination (underestimate) of the relative standard error is shown for four locations in Figure 4. The predictions of change in sealant modulus show the biggest change when the sealant is experiencing extreme weather. These predictions are
based on the combination of all four weathering factors, temperature, humidity, UV, and mechanical strain acting on the sealant.

![Graph showing predictions of change in sealant modulus at 100 sec based on statistically interpolated data generated on the NIST SPHERE.](image)

**Figure 4.** Predictions of change in sealant modulus at 100 sec based on statistically interpolated data generated on the NIST SPHERE.

A different prediction is obtained when the United States Department of Energy’s Energy Plus hourly weather data is used. This database has hourly data for a large number of locations across the United States. The data is not for a specific year, but is representative of an “average” year. The same method as above is applied to each station in the Energy Plus database to predict change in $E_{100}$ modulus over one average year. A surface map over the contiguous United States was then generated by spatially smoothing those predictions using local regression. Figure 5 shows that the greatest predicted change occurs in regions with the most extreme weather. This is consistent with the observations from the NIST SPHERE and Figure 4.
5 CONCLUSIONS

The components of a service life prediction model are presented in this paper. These components include characterizing the time-dependent modulus of sealant as a function of dose, assembling the changes in time-dependent modulus into a database, and using statistical methods to generate a response surface for the sealant. This model can then be used to predict changes in the modulus of that sealant at specific locations for over a period of time, or at a wide variety of locations in a given year. The next step in the process of developing service life prediction models will involve validating the predictions with data from outdoor exposures.

ACKNOWLEDGEMENTS

The support from an industry/government consortium on Service Life Prediction of Sealant Materials at National Institute of Standards and Technology (NIST) is greatly appreciated. The participating companies include DAP, BASF, Dow Corning, Dow Chemical, SIKA, Tremco, and Wacker Silicones.
REFERENCES


DURABILITY OF COOL AND CONVENTIONAL COLORED COATINGS AFTER SIX MONTHS EXPOSURE AT DIFFERENT ENVIRONMENTS

Kai Loh (1) Neide M. N Sato (2) Isabela M. S. Liborio (1)

(1) Department of Civil Engineering, University of São Paulo (USP), São Paulo - kai.loh@usp.br; isabela.liborio@usp.br.
(2) Consultant – neidesato@gmail.com.

Abstract

This study investigate the solar reflectance of colored acrylic coatings, "cool" and conventional types, after outdoor exposure at urban, marine and rural environments, all of them located in São Paulo State. The “cool” coatings are paints formulated with infrared reflective pigments and present higher solar reflectance than conventional ones.

The solar reflectance of cool coatings was compared to conventional acrylic coatings, bought in the São Paulo market, with similar colors (gray, white, brown and yellow). The exposure was conducted according to ASTM G-7.

The coatings were applied on asbestos-free fiber cement plates; color and solar reflectance were measured in unexposed specimens and after six months of exposure at the 3 sites. The results showed that the cool paints present significantly higher reflectance in the infrared (NIR) than the corresponding conventional colored paints. The study showed that the “cool” coatings after 6 months of natural aging still present higher reflectance than the conventional paints.

1 INTRODUCTION

The phenomenon of "heat islands" is often observed in metropolitan areas. It is characterized by elevated local temperature, often 10°C higher than the temperature of areas around the city. Several factors contribute to this phenomenon, being mainly replacement of vegetation by buildings and paved streets. The roofing and paved areas are heat absorber of solar radiation and can reach temperatures between 30-45° C, whereas the peripheral areas with vegetation record temperatures between 16-26 ° C, showing differences around 20°C [1].

The "Heat Island Group" at Lawrence Berkeley Laboratories (LBL) is internationally recognized as the highest in this area. This group has conducted many studies on the use of cool pigments. Their studies showed that the use of "cool" materials is a good strategy in
improving the thermal comfort inside buildings in large cities [2]. High reflective “cool” colored coatings are an effective, low-cost and easy to use technique to modify the characteristics of surface materials besides allowing for a variety of colors and meeting aesthetic demands [3].

In Brazil, there are many papers regarding the thermal performance and reflectance of colored coatings from the market [4] [5]. The lack of national data of the use of "cool" coatings motivated this study. In this study the coatings were applied on cement based roofing components, reinforced with fibres commonly used in light constructions, low cost housing and rural constructions for animals [6] so it was selected as substrate for the coating.

2 MATERIALS AND METHODS

2.1 Paint samples

The study was carried out with two groups of acrylic latex coatings with similar colors. One group was formulated with “cool” colored pigments, with formulation typical of commercial coatings for exterior application produced in the laboratory of University of São Paulo, with technical support of a paint specialist. IR reflective pigments: yellow, brown and light gray colors. These pigments are mixed metal oxide (MMO) and known as complex inorganic color pigments (CICPs) [7] [8]. The second group was from São Paulo city market and used for comparative purposes.

The coatings were formulated with semi-gloss finishes and a PVC of 30%. PVC (Pigment Volume Concentration) is the ratio of the volume of pigment to the volume of total nonvolatile material present in a coating and is expressed as a percentage. All samples are based on styrene acrylic copolymer with formula recommended for application on building exteriors. The colors selected were light gray, yellow and brown, common colors for tile products. White was also prepared and was considered cool because of its high reflectivity.

The study was carried out on:
- 3 samples of cool styrene acrylic coatings incorporating infrared reflective pigments, yellow, brown and light gray;
- 1 sample of white styrene acrylic coating, incorporating a high level of TiO₂;
- 4 samples of conventional styrene acrylic coatings collected from the market. Colors: white, yellow, brown and light gray.

2.2 Specimens

The coatings were applied on one face of flat fibercement plates (11.5x8cm), one coat of primer and two coats of paint were applied, using 10ml per coat. Both groups were applied with a brush according to the manufacturer's instructions. A set of uncoated mortars was used as reference. The specimens were dried for several months prior to exposure. Figures 1 and 2 present photos of specimens showing the yellow, gray, brown and white colors, those coatings formulated in the laboratory and those collected from the market with similar colors.
2.3 Methods

Natural weathering test: The coated and uncoated specimens were exposed at an angle of 45 degrees to the horizontal, facing North and 0.45 m above the ground, which was covered with grass, in three locations (ASTM G 7-97): a) Urban atmosphere: São Paulo site, located on the campus of the Department of Civil Construction Engineering, University of São Paulo (USP) in São Paulo city, b) Rural atmosphere: USP campus in Pirassununga, c) Marine atmosphere: located on the Marine Base of the Oceanographic Institute of USP in Ubatuba. A set of uncoated specimen was used as reference. The location and annual weather summary data [9] at the sites are presented in Table 1.

Table 1: Climate summary data at the sites [1]

<table>
<thead>
<tr>
<th>Sites</th>
<th>Latitude</th>
<th>Longitude</th>
<th>Temperature (°C)</th>
<th>Rainfall (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Average</td>
<td>Maximum</td>
</tr>
<tr>
<td>Rural:</td>
<td>21º58'S</td>
<td>47º27'W</td>
<td>19.1</td>
<td>30.1</td>
</tr>
<tr>
<td>Pirassununga</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Marine:</td>
<td>23º26'S</td>
<td>45º04'W</td>
<td>22.4</td>
<td>27.0</td>
</tr>
<tr>
<td>Ubatuba</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Urban:</td>
<td>23º32'S</td>
<td>46º38'W</td>
<td>20.2</td>
<td>25.0</td>
</tr>
<tr>
<td>São Paulo</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Evaluation of weathering effects: The following parameters were measured:

Visual inspection: The appearance is a critical attribute, although subjective and qualitative, because the service life for users is often determined by changes in appearance. The evaluations were carried out with the naked eye based on ASTM standards for coating defects, using a rating system (ASTM D 662 and ASTM D 661).

Color: measured according to ASTM D 2244-89 based on the CIE Lab System (CIE L*a*b*). The L*a*b* color values were measured with a color-guide, Byk Gardner, 45/0 geometry. The axes a* and b* represent the color and axis L* is lightness. (L) is lightness, (a) is red-greenness and (b) is yellow-blueness. The color is established in terms of difference between exposed and unexposed specimens and uncoated reference, rather than absolute values. The change in color ΔE*, is used to represent the color difference according the equation:

\[ \Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \]

Reflectance: measured according to ASTM C1549-09 Standard Test Method for Determination of Solar Reflectance Near Ambient Temperature Using a Portable Solar Reflectometer Model SSR-ER; this instrument measures total hemispherical solar reflectance.
3 RESULTS AND DISCUSSION

3.1 Visual inspection of exposed specimens

The qualitative analysis showed a light deposit of dirt in all specimens exposed at the three sites. Defects such as blistering, cracking, or loss of adhesion, efflorescence or chalk were not observed in the coated specimens.

3.2 Color measurements

Table 2 shows the color measurements of the uncoated and coated specimens, before exposure and after six months at the Pirassununga site. Table 3 shows the color measurements of the specimens, after six months exposure at São Paulo and Ubatuba sites.

Table 2: Color measurements of unexposed and 6-month exposed specimens from Pirassunungau site

<table>
<thead>
<tr>
<th>Pirassununga site</th>
<th>Before exposure</th>
<th>After exposure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cool paints</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brown</td>
<td>L 48 A 18 b 16</td>
<td>L 47 a 16 b 16</td>
</tr>
<tr>
<td>Yellow</td>
<td>L 73 A 17 b 53</td>
<td>L 69 a 17 b 50</td>
</tr>
<tr>
<td>White</td>
<td>L 95 A -1.5 b 2.7</td>
<td>L 90 a 11 b 11</td>
</tr>
<tr>
<td>Gray</td>
<td>L 83 A -0.2 b -0.7</td>
<td>L 76 a 0.4 b 16</td>
</tr>
<tr>
<td>Conventional paints</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brown</td>
<td>L 46 A 18 b 18</td>
<td>L 44 a 17 b 18</td>
</tr>
<tr>
<td>Yellow</td>
<td>L 71 A 18 b 51</td>
<td>L 70 a 17 b 38</td>
</tr>
<tr>
<td>White</td>
<td>L 95 A -1.6 b 1.8</td>
<td>L 90 a 0.6 b 10</td>
</tr>
<tr>
<td>Gray</td>
<td>L 83 A 0.6 b -1.5</td>
<td>L 78 a 0.4 b 6.1</td>
</tr>
<tr>
<td>Reference</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unpainted</td>
<td>L 65 A 0.6 b 4.7</td>
<td>L 54 a 3.5 b 11</td>
</tr>
</tbody>
</table>

The color measurement results showed that the two groups of coatings presented very similar colors. After six months of exposure at Pirassununga, the uncoated specimen presented higher change of color (ΔE: 13) than the coated specimens; probably the porosity of the substrate facilitated retention of dirt particles, except with the gray cool paint and yellow conventional paint specimens, both light colored. The axis L* (lightness) of all specimens presented darkening, but the brown specimens presented less darkening because the particles deposited on their surfaces were probably almost the same color as the paint.

After six months of exposure at São Paulo and Ubatuba sites the uncoated specimens showed higher color change (ΔE: 16 and 11) than the coated specimens. The L* axis (lightness) for all specimens moved towards the darker end of the scale. No great differences in color change between the two groups of paints were observed.
Table 3: Color measurements of specimens exposed at the São Paulo and Ubatuba sites after six months exposure

<table>
<thead>
<tr>
<th></th>
<th>São Paulo Site</th>
<th>Ubatuba site</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>After exposure</td>
<td>After exposure</td>
</tr>
<tr>
<td>Cool paints</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brown</td>
<td>L  46  a  16  b  15  ΔE 2.3</td>
<td>L  45  a  18  b  13  ΔE 3.9</td>
</tr>
<tr>
<td>Yellow</td>
<td>L  67  a  15  b  46  ΔE 9.1</td>
<td>L  70  a  17  b  51  ΔE 3.2</td>
</tr>
<tr>
<td>White</td>
<td>L  87  a  0.2  b  8.4  ΔE 10</td>
<td>L  91  a  -0.6  b  5.9  ΔE 5.5</td>
</tr>
<tr>
<td>Gray</td>
<td>L  78  a  0.6  b  3.5  ΔE 6.2</td>
<td>L  78  a  0.2  b  1.6  ΔE 5.5</td>
</tr>
<tr>
<td>Conventional paints</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brown</td>
<td>L  44  a  16  b  16  ΔE 3.5</td>
<td>L  44  a  16  b  14  ΔE 4.2</td>
</tr>
<tr>
<td>Yellow</td>
<td>L  66  a  15  b  40  ΔE 13</td>
<td>L  68  a  16  b  42  ΔE 10</td>
</tr>
<tr>
<td>White</td>
<td>L  89  a  -0.5  b  6.9  ΔE 8.2</td>
<td>L  92  a  -0.9  b  5.1  ΔE 4.7</td>
</tr>
<tr>
<td>Gray</td>
<td>L  76  a  -0.1  b  2.3  ΔE 7.5</td>
<td>L  82  a  -1.0  b  0.1  ΔE 2.3</td>
</tr>
<tr>
<td>Reference</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unpainted</td>
<td>L  50  a  1.6  b  7.4  ΔE 16</td>
<td>L  55  a  1.6  b  4.1  ΔE 11</td>
</tr>
</tbody>
</table>

The brown specimens presented less color change in Pirassununga than specimens in São Paulo and Ubatuba.

3.3 Solar reflectance

Figures 3, 4 and 5 show the solar reflectance results of uncoated and coated specimens, before and after six months exposure at the ageing sites.

Figure 3: Solar reflectance of uncoated specimens and cool and conventional coatings exposed at the Pirassununga site.
The results showed that the two groups of coatings presented changes in solar reflectance after six months of exposure and the uncoated specimens showed a much greater change in this property. The changes are higher for white and light colors, such as gray and yellow, but a lower change was observed for brown, probably because of its initial lower reflectance and the fact that particles deposited on the surface were almost the same color as the paint itself. The changes were caused probably by dirt retention.

Dark roof materials have reflectance of 0.05 to 0.20 while light-colored roof materials have reflectance of 0.55 to 0.90. The incorporation of brown infrared reflective pigment showed solar reflectance of 0.40, value higher than incorporation of brown conventional pigment, but insufficient to be considered as a cool coating. The initial solar reflectance value required for low-slope cool roof product is greater than or equal to 0.65 and greater than or equal to 0.50 after 3 years ageing [10], so the brown color coating of this study cannot be considered as a cool finishing.
4 CONCLUSION

Exposure to the environment changed the aesthetic and solar reflectance of the coated and uncoated surfaces. Both sets of painted specimens presented changes in color and loss of solar reflectance at similar levels, independent of the environment. With 6 months exposure, the two groups of coatings did not show many differences in behavior.

Durability studies [9] with 7 years of exposure, at the same 3 sites showed that a water cleaning procedure can recover part of the initial qualities. Cleaning procedures changed the axis L* axis (lightness), resulting in recovery of its initial solar reflectance. According to Levinson et al [7] (2005), exposure to the environment can lower initial solar reflectance of roof materials caused by deposition of soot, dust, and fungi or algae; cleaning can restore properties like color, texture and, partially, surface reflectance, but it is not possible to predict the reflectance of coatings because the material deposited on the surface has various origins, characteristics, levels and quantities.

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IMPACT OF DIFFERENT EUROPEAN LOCAL CLIMATE CONDITIONS ON THE MOISTURE PERFORMANCE OF WOODEN COMMODITIES

Bornemann, T. (1), Brischke, C. (1), Alfredsen, G. (2), Humar, M. (3) and Larsson-Brelid, P. (4)

(1) Leibniz University Hannover, Faculty of Architecture and Landscape Sciences, Institute of Vocational Sciences in the Building Trade (IBW), Hannover, Germany – bornemann@ibw.uni-hannover.de; brischke@ibw.uni-hannover.de

(2) Norwegian Forest and Landscape Institute, Ås, Norway – gry.alfredsen@skogoglandskap.no

(3) University of Ljubljana, Biotechnical Faculty, Department of Wood Science and Technology, Ljubljana, Slovenia – miha.humar@bf.uni.lj.si

(4) SP Technical Research Institute of Sweden, Dept. of Wood Technology, Borås, Sweden – pia.larsson.brelid@sp.se

Abstract

One key issue in wood construction is durability which is determined by construction details and the resistance of the material against biological deterioration and wetting. The service life of wooden commodities is dominated by occurring moisture loads on the one hand and their capability to withstand moisture on the other hand. Hence, climatic factors have a direct impact on the material climate and thus on service life. However there is still a lack of knowledge about the relationship between material climate and local weather conditions. Therefore the aim of this study was to quantify this interaction.

Within this study wood moisture content and wood temperature were recorded for combined façade-decking elements made from Douglas fir heartwood and Scots pine sapwood exposed at eight different locations in Europe, namely Bergen (Norway), Ås (Norway), Borås (Sweden), Ljubljana (Slovenia), San Sebastian (Spain), Ourense (Spain), Portsmouth (Great Britain) and Hannover (Germany). The moisture performance of the wooden materials was set into context with local weather parameters.

Orientation of the facades had a major impact on the moisture performance. Moreover the horizontal exposition experienced higher moisture loads compared to the vertical. Differences between test sites were determined and could be explained with different climatic loads impacting on the construction.

Keywords: Moisture monitoring, climatic parameters, outdoor performance
1 INTRODUCTION

Wood shows a number of advantages in comparison to other building materials like being a natural and renewable resource or providing high strength related to its own weight which makes it easy to handle and process [1]. Therefore wood is increasingly used in many outdoor applications. On the downside, outdoors wood is exposed to a variety of influences, which might reduce its durability against wood degrading organisms [2]. Therefore, one key issue for wooden constructions in outdoor applications is their durability.

Besides the material inherent resistance, the service life of wood is also influenced by environmental conditions which are more or less favourable for decay organisms. In this context the climate plays a vital role and Brischke and Rapp [3] distinguished between different climate levels: The macroclimate (weather data of a certain site), the mesoclimate (climatic conditions at the site) and the microclimate (surface conditions). Service life analyses of building constructions as well as the expectations of engineers, architects and consumers will need more data on durability of products, not only on the wooden material itself. The first step to evaluate the exposure conditions are the macroclimate conditions. Driving rain, moisture, temperature and also the solar radiation are the most important factors [4]. In this respect Scheffer [5] made a first attempt to find a correlation between climate parameters and the decay rate in different parts of the United States. He developed a climate index that described the potential for decay in above ground structures based on temperature and rainfall data. Brischke [6] identified the “material climate”, wood moisture content (MC) and wood temperature, as the most important factor for degradation by wood destroying fungi and bacteria. Therefore the material climate and its dynamics should be considered for estimation of decay hazards in first instance [7].

Climatic parameters can vary strongly for different locations. Brischke and Rapp [8] investigated differences in material climate under various microclimatic conditions at four different test sites in Germany. Their results showed that microclimatic parameters like shading had a direct impact on degradation processes. In addition, Frühwald Hansson et al. [9] presented a climate model transferring macro climate data into wood climate data for calculating the relative risk for decay caused by climate variability in Europe. A decay hazard map was drawn and illustrated the climate induced variability within the European continent. Nevertheless, the results were based on horizontal double layers which made them not necessarily transferable to less severe exposures.

It has been shown that site-specific climatic factors have a direct impact on the material climate and an indirect impact on service life respectively. However there is still a lack of knowledge about the relationship between material climate and climatic parameters and the performance of wooden components under different climate regimes.

In this respect the aim of this study was to investigate the influence of local weather parameters at different test sites on the moisture performance of combined facade-decking elements made from Scots pine sapwood (Pinus sylvestris L.) and Douglas fir heartwood (Pseudotsuga menziesii Franco). Therefore test elements have been exposed outdoors above ground at different locations in Europe. The moisture performance of the materials and test site dependent differences were studied.
2 MATERIAL AND METHODS

2.1 Moisture and temperature recording

The measurement system applied in this study was described in detail earlier by Brischke et al. [10] and can be summarized in brief as follows: electrodes of polyamide coated stainless steel cables were conductively glued in the specimens. The electrodes were connected to small data loggers (Materialfox Mini, Scanntronik Mugrauer GmbH, Zorneding, Germany) that recorded the electrical resistance of the wood. Wood species-specific resistance characteristics were developed [10, 11]. Minimum and maximum temperatures were recorded daily using Thermofox Mini data logger (Scanntronik Mugrauer GmbH, Zorneding, Germany) and used to calculate the average daily temperature. The measurement points were placed in the centre of the specimens.

2.2 Combined facade-decking element

Combined facade-decking elements (Figure 1, left), which had been designed as a reference object for UC 3.1 and UC 3.2 [12], had been equipped with samples made from Scots pine sapwood and Douglas fir heartwood. The element had a total height of 1510 mm and was 1570 mm wide. Board-like specimens were submitted to the following three exposure situations:

- **Vertical cladding (oriented north and south):**
  Boards (25 x 100 x 500 mm³) were mounted horizontally on a combined facade-decking element (Figure 1, left) and carried out as board-on-board cladding.

- **Horizontal single layer (decking, oriented south):**
  Boards (25 x 100 x 500 mm³) were exposed vertically on two bearings of a combined facade-decking element (Figure 1, left).

![Combined facade-decking element](image)

Figure 1: Combined facade decking element with sandwich test at the test site in Ljubljana, Slovenia (left), Sandwich test set up (taken from [13]) (right)
In addition, Sandwich test-setups for UC 3.2 [13] made from the same material were exposed horizontally to the North on two bearings of a combined facade-decking element (Figure 1, right). The size of the specimens were 200 (ax.) x 49 x 25 mm$^3$ (a, Figure 1, right) and 200 (ax.) x 100 x 25 mm$^3$ (b, Figure 1, right).

For each exposure type three replicate specimens were provided with electrodes for daily moisture content and temperature recordings. The measuring points were in each case located on the three cover boards of the cladding plus the bottom side of the decking and the sandwich specimens.

2.3 Location of the test sites in Europe

Combined facade-decking elements were exposed at different sites in Europe (Figure 2):

1. Norwegian Forest and Landscape Institute Fana, Bergen, Norway (60°25’99.77”N; 5°34’83.81”E). Exposed March 2012.
2. Norwegian Forest and Landscape Institute, Ås, Norway (59°66’71.45”N, 10°77’34.23”E). Exposed March 2012.
3. Technical Research Institute of Sweden (SP), Borås, Sweden (57°71’49.65”N; 12°88’74.40”E). Exposed June 2012.
4. To be exposed at University of Portsmouth in the South of Great Britain close to the English Channel.
5. Institute of Vocational Sciences in the Building Trade, Hannover, Germany (52°39’29.87”N; 9°69’59.58”E). Exposed April 2010
6. Test site of Ljubljana University, Slovenia (46°04’87.56”N; 14°47’86.12”E). Exposed September 2012.
8. Tecnalia, Sustainable Construction Unit, Timber in Construction, Ourense, Spain (42°17’44.49”N; 7°48’00.77”W). Exposed December 2013.

Figure 2: Location of the test sites in Europe (1 Bergen, 2 Ås, 3 Borås, 4 Portsmouth, 5 Hannover, 6 Ljubljana, 7 San Sebastian, 8 Ourense)
For all locations climatic parameters (temperature, relative humidity, precipitation) were recorded by the closest weather station to the test site.

3 RESULTS AND DISCUSSION

3.1 Climatic conditions at the test sites

Table 1 shows the mean values of temperature and mean precipitation for the eight different test sites split in time periods and the mean annual temperature as well as the annual sum of precipitation.

Table 1: Mean values of temperature (Temp. [°C]) and precipitation (Prec. [mm]) for the 8 different test sites (based on data from the World Meteorological Organisation (WMO)) separated in time periods: spring (March till May), summer (June till August), fall (September till November) winter (December till February) and mean annual temperature and annual sum of precipitation. Mean values were calculated using average daily (24h) values of temperature and precipitation per month in the period 1961-1990.

<table>
<thead>
<tr>
<th>Location</th>
<th>Spring</th>
<th>Summer</th>
<th>Fall</th>
<th>Winter</th>
<th>Annual values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>[°C]</td>
<td>[mm]</td>
<td>[°C]</td>
<td>[mm]</td>
<td>[°C] [mm]</td>
</tr>
<tr>
<td>Bergen</td>
<td>6.6</td>
<td>130</td>
<td>13.9</td>
<td>157</td>
<td>1.7 192</td>
</tr>
<tr>
<td>Ås</td>
<td>4.6</td>
<td>49</td>
<td>15.3</td>
<td>77</td>
<td>-4.3 46</td>
</tr>
<tr>
<td>Borås</td>
<td>5.3</td>
<td>38</td>
<td>15.1</td>
<td>76</td>
<td>-1.4 51</td>
</tr>
<tr>
<td>Portsmouth</td>
<td>9.3</td>
<td>35</td>
<td>16.1</td>
<td>32</td>
<td>6.0 43</td>
</tr>
<tr>
<td>Ourense</td>
<td>13.3</td>
<td>53</td>
<td>21.2</td>
<td>18</td>
<td>8.4 98</td>
</tr>
<tr>
<td>San Sebastian</td>
<td>11.4</td>
<td>116</td>
<td>18.0</td>
<td>101</td>
<td>8.8 117</td>
</tr>
<tr>
<td>Ljubljana</td>
<td>10.4</td>
<td>80</td>
<td>19.4</td>
<td>112</td>
<td>0.8 60</td>
</tr>
<tr>
<td>Hannover</td>
<td>8.6</td>
<td>44</td>
<td>17.0</td>
<td>39</td>
<td>1.9 55</td>
</tr>
</tbody>
</table>

The test sites in Scandinavia and Ljubljana showed lowest mean temperatures during the winter period, highest were found for the South European sites. The test site in Bergen with 1.7 °C reached highest temperatures during the winter among the Scandinavian sites. This indicates the very prominent influence of the Gulf Stream in this region leading to very mild temperatures during wintertime due to the transport of warm water off the coast of Western Norway. Prominent were the high differences in average precipitation. The rainfall in Bergen was more than 1.5 times higher during summer and fall compared to Ljubljana (112 mm (summer), 135 mm (fall)) and San Sebastian (101 mm (summer), 153 mm (fall)). Also compared to all other test sites the amount of precipitation was exceptional high during the whole year (2250 mm) which is again due to the influence of the Gulf Stream. High mountains near the coast led to continuously high amounts of precipitation in the area of Bergen. Also the test sites in Ourense and San Sebastian show high amounts of precipitation in combination with high temperatures during fall and winter. This can be explained by the
influence of the Azores Current transporting warm and humid air of the coast line of Western Spain. Hannover has a more continental and dryer climate compared to the other test sites, which are more situated in a maritime climate zone. Each of the locations have different, site specific climate conditions and were therefore selected for investigating their impact on the moisture performance of wooden commodities.

3.2 Moisture performance at different test sites

The impact of different climate conditions on the MC is exemplarily shown for Douglas fir at four different locations in the horizontal exposition in Figure 3. Significant differences between the locations became obvious. Highest MC was recorded for the decking in Bergen which can be explained by the high values of precipitation impacting on the decking. Also for the decking exposed in Borås and Ås high MC were recorded although the amount of precipitation was significantly lower compared to Bergen (Table 1). Nevertheless the amount of precipitation was high enough that it allowed for long periods of standing water on the decking surface leading to a constant wetting and high moisture ingress into the specimens respectively. In Hannover Douglas fir showed the lowest MC over the measuring period (arrow Figure 3) which coincides with low values of precipitation and higher temperatures which allowed for better redrying during dry periods. Also Viitanen et al. [3] reported that macroclimatic loads were lower in the continental and northern part of Europe compared to zones near the Atlantic Ocean. Furthermore Frühwald Hannsson et al. [9] stated that there is a general trend of higher decay hazard within maritime climate and decreasing decay hazard towards the east which has more continental and drier climate.

![Figure 3: Moisture course of horizontal decking made from Douglas fir at 4 different test sites.](image)

The impact of orientation on the moisture performance is exemplarily shown for a vertical cladding made from Scots pine sapwood for North and South orientation in Bergen and Hannover (Figure 4). Significant differences between the test sites became visible. Interestingly there were almost no differences between the North and South orientation in Bergen although South West is the weather side in central Europe. This can be explained by
constant high amounts of precipitation during the whole year leading to constant moisture loads on both sides of the facade. Also Rapp et al. [14] and De Windt et al. [15] concluded that the duration of continuous rain is more relevant than the amount of rainfall. In contrast, in Hannover the South oriented facade showed higher MC compared to the North. This was also described by Nore et al. [16] who reported that test elements oriented to the south showed higher moisture contents in terms of high wind-driven rain loads from southwest and high relative humidity close to the surface due to evaporation of surface water in times of re-drying. Also Auld et al. [17] supported these results by describing wind driven rain as the moisture source that can impact the most on the performance of a building envelope. On the other hand, solar irradiation and higher wind loads on the south oriented facade fostered re-drying.

![Figure 4](image)

Figure 4: Moisture course of North and South oriented vertical cladding made from Scots pine sapwood at 2 different sites.

The impact of climatic loads on the moisture performance of wooden claddings was also described by Engelund et al. [18]. They reported that the annual variation of weekly averages of the MC of coated wooden claddings appeared to follow the variation of temperature and relative humidity [18]. No evidence of a significant effect of precipitation on the weekly average MC of coated claddings was found [19] but for the untreated reference racks the precipitation attributed significantly to the average weekly MC.

4 CONCLUSION

It became apparent that climatic parameters differed significantly between various locations. Furthermore design and exposure conditions determine the respective microclimate, which can have an overruling effect on the moisture performance. To get a deeper insight into the relationship of climate and moisture performance of wood exposed outdoors future studies are foreseen. In this context test-site comparisons with a referential test object could serve to investigate the influence of various factors in different regions with preferentially more extreme climate conditions (mountain and coastal areas, humid and arid regions, etc.).
REFERENCES

NO CONCRETE IS SUSTAINABLE WITHOUT BEING DURABLE!

Geert De Schutter(1)

(1) Magnel Laboratory for Concrete Research, Ghent University, Ghent, Belgium – Geert.DeSchutter@ugent.be

Abstract
Sustainability of concrete is a hot topic nowadays, not only in research but also in practice. The production of Portland clinker is responsible for a significant amount of CO$_2$-production worldwide. Partial replacement of Portland cement by supplementary cementitious materials should indeed be encouraged. A win-win situation can be obtained by implementing waste materials in concrete, reducing the cement content and avoiding huge amounts of waste disposal. However, no concrete structure can be sustainable without being durable. Even while having an excellent carbon footprint performance per unit of volume, it could be prone to a more accelerated degradation under severe environmental conditions, needing earlier repair or replacement, and thus increasing the cumulated carbon footprint calculated over the entire service life. Performance of alternative concrete mixtures should not only be checked in terms of strength classes, but also in terms of durability performance. It is shown in this paper that alternative mixes with similar strength as a reference mix can show inferior durability performance. As a consequence, sustainability comparisons based on equal strength considerations can be misleading towards the real sustainability over the entire service life of a concrete structure.

1 INTRODUCTION
In Wikipedia, a good description can be found on sustainability. The word sustainability is derived from the Latin sustinere (tenere, to hold; sus, up). Dictionaries provide more than ten meanings for sustain, the main ones being to “maintain”, ”support”, or “endure”. However, since the 1980s sustainability has been used more in the sense of human sustainability on planet Earth and this has resulted in the most widely quoted definition of sustainability as a part of the concept sustainable development, that of the Brundtland Commission of the United Nations on March 20, 1987: “sustainable development is development that meets the needs of the present without compromising the ability of future generations to meet their own needs” [1].
Contrary to sustainability, **durability** is not that well explained in Wikipedia. *Durability is the ability to endure. It can refer to: Durable goods, goods with a long usable life in economics; Durability (database systems), one of the ACID properties; In safety and technology: Dust resistant, Fire resistant, Rot-proof, Rustproof, Thermal resistant; Impact resistant; Waterproof [2].*

Durability as well as sustainability is thus referred to as a property or ability to endure. However, sustainability clearly involves also aspects of ‘preservation’, not compromising future generations. Nevertheless, a lot of confusion exists while discussing sustainability and durability. To many, sustainability and durability are synonyms, or at least do mean something very similar. In some languages there is not even a possibility to make a distinction (e.g., ‘Duurzaamheid’ in Dutch). On internet forums, some people do raise the question “What is the difference between sustainability and durability?” hoping to get some better guidance [3]. However, only one short answer seems to be added: “Sustainability is something's ability to last. Often, the durable things are able to sustain longer because they are tougher, more hardy, built to last.” Unfortunately, this answer is also not very clear, and seems to relate the two words to ‘the ability to last’ or ‘sustain’. The good point, although somewhat hidden, is the statement that durable things are able to sustain longer, which can be interpreted as a statement that durability is part of sustainability (although it is not clear whether this is what was meant to be said within the reply given on the forum). However, as defined by the Brundtland Commission, sustainability is about more than just sustaining or lasting for a long time.

Along these lines, a good opinion has been published by Peter Yost in the Magazine of Sustainable Design in 2009: “Sustainability requires durability!” [4]. The strong title of his opinion is nicely illustrated by the statement: “If you double the life of a building, no matter what the building is made of, you halve the environmental impact of its construction”. He explains that the search for energy saving has led to increased moisture problems in buildings, leading to mold growth, rot, corrosion, air-quality problems and even structural failures. He concludes: “I am not saying we need to back off on the energy efficiency of our buildings: green buildings absolutely need to be energy misers. But to be green and durable and energy efficient, we need a new mantra: Manage energy and moisture with equal intensity”. Peter Yost is astonished to see “that most versions of LEED are silent on the critical matter of durability and its dependence on the relationship between energy and moisture control”.

A similar opinion has been published by Eric Wilson in 2011: “Durability is the Key to Sustainability” [5]. “Goods that need replacing every few years, what is known as planned obsolescence, deals a serious blow to the goals of sustainability. When a poorly made object fails, ends up in the waste stream, and leads a consumer to purchase a new widget, there are environmental ramifications for the planet and economic ones for the individual.”

In the context of concrete structures, it is clear that technical durability is a prerequisite for environmental sustainability. Technical durability can be seen as the resistance to degradation processes [6], while environmental sustainability is – following the Brundtland Commission – linked to the preservation of natural sources and limitation of waste etc. Unfortunately, sustainability of concrete is often limited to carbon footprint per cubic meter of concrete, or carbon footprint to build a structure. However, when a cubic meter of concrete with reduced carbon footprint needs to be replaced much sooner due to a reduced resistance to degradation processes, the environmental benefit can be totally lost or even become negative. A better
approach is to perform LCA analysis, duly considering the technical durability of the concrete. In this respect, another common simplification with potential negative consequences is the (erroneous) idea that concrete strength is fully determining the concrete durability. Nevertheless, two different concretes with equal strength do not necessarily have the same technical durability. These aspects will be illustrated further on in this paper.

2 STRENGTH VERSUS DURABILITY

Many standards, including the European concrete code EN 206-1, consider three important parameters in order to guarantee durable concrete structures: water/cement ratio, cement content, and strength. It has been illustrated previously that each of these parameters can be criticized [7]. As an example of the debatable correlation between concrete strength and durability, Figure 1 shows the carbonation depth after 50 years of exposure to a natural environment for 14 different concrete compositions. The results have been obtained for limestone filler based self-compacting concrete with different cement and limestone filler content, with different water/cement ratio, with different cement/powder ratio, and with different strength class [8]. The carbonation depths after 50 years have been estimated based on experimentally obtained diffusion coefficients.

![Figure 1: Carbonation depth after 50 years of 14 different SCC mixtures as a function of 28 days’ concrete strength.](image)

While limestone filler is accelerating the hydration of Portland clinker due to improved nucleation possibilities, it is not chemically active (except for a minor percentage which could be chemically active in the formation of carboaluminates) [9-11]. In this way, limestone filler improves the strength development, while the improvement by limestone filler of the pore structure and the long term durability performance is not always of the same degree [12]. In ternary blends however, there could be additional synergetic effects between limestone filler
and the aluminate phase of a third powder, e.g. slag, fly ash or calcined clay, leading to improved microstructural properties [13,14]. As the effect of addition of powders can be manifold (chemical, physical, filler, dilution) [15], the correlations between simple mix design parameters, strength and durability are not always straightforward. In the example of Figure 1, the difference in mix design is leading to a difference by a factor of three in carbonation depth for concretes having similar strength.

Following typical Fickian diffusion laws, increasing the CO$_2$ diffusion coefficient by a factor of three is leading to a reduction of the time to reach a certain carbonation depth by a factor of nine! In case the service life of a structure is defined as the time needed for reinforcement steel to be depassivated by carbonation, the service life could thus be reduced by a factor of nine when relying on strength alone as a durability indicator for alternative “sustainable” concrete compositions!

This is illustrated in Figure 2 for the case of a structure with concrete cover 20 mm and intended service life of 50 years. Due to the insufficient technical durability (insufficient resistance against carbonation, with three times higher carbon diffusion coefficient), the alternative composition with similar strength, and which at the same time might have a considerably lower carbon footprint per cubic meter of concrete, will in that case not result in a more sustainable solution!

Figure 2: Effect of a tripled diffusion coefficient on the service life (time needed to reach a carbonation depth of 20 mm), following Fick’s diffusion law.
In spite of the clear message of manifold trivial examples similar to the one given in Figures 1 and 2, many research papers in international journals only focus on concrete strength when reporting studies showing the feasibility to develop eco-friendly, sustainable mixtures, typically implementing alternative waste materials. Some simplistic reasoning can sometimes be found in these kind of papers. On the one hand, while calculating or estimating the carbon footprint of a cubic meter of concrete, no footprint is considered for the waste materials “as they are available anyway, and should be landfilled otherwise”. This is not fair, as some of these waste materials are resulting from a polluting industry, e.g. coal power plants. On the other hand, they consider the alternative concrete as sufficient when reaching similar strength values as needed for the reference concretes, implicitly accepting that “same strength will guarantee same durability performance”, which of course is not valid at all.

3 DURABILITY VERSUS SUSTAINABILITY

As shown before, wrong choices could reduce the service life of the resulting concrete structure by a factor of nine. In literature, the carbon footprint of alternative ‘green’ mixtures is typically 30% to 60% lower in comparison with reference mixtures. Even keeping only one third of the carbon footprint per cubic meter of concrete, a reduced service life by a factor of nine still means a net increase of the environmental loading by a factor of three in view of a predefined service life (e.g. 50 years), as illustrated in Figure 3. A concrete mixture with a reduced carbon footprint will not lead to sustainable solutions when the concrete does not prove to be durable!

In order to make a reliable evaluation of the ‘green’ character of a concrete structure with alternative binder systems, a fair analysis has to be made, duly considering the technical durability performance of the ‘green’ concrete, and not merely the carbon footprint per cubic meter of concrete. In literature, good examples can be found on how to evaluate the real environmental impact of ‘green’ concretes, based on a detailed life cycle assessment (LCA). Based on literature review and theoretical calculations, Van den Heede and De Belie [16] conclude the following: “The adopted functional unit for which the environmental impact is calculated, influences the outcome significantly. When comparing different concrete compositions, this unit should incorporate differences in strength, durability and service life. Hence, a cradle-to-grave or modified cradle-to-gate approach is advised as system boundary. When using industrial by-products as cement replacing material in ‘green’ concrete, an economical allocation of impacts is recommended.”

One of the major pitfalls, is to blindly rely on similar concrete strength. This misconception is still too widely spread within concrete practice. The erroneous idea saying that “when it is strong enough, it is durable enough”, is getting more and more risk-full when considering alternative ‘green’ binder systems. While traditionally, strength is the first and most important concrete property to be considered, a new approach should be followed in which “strength follows durability”. It should first (or at least simultaneously) be checked how the ‘green’ binders should be defined in order to be technically durable. The structural design should afterwards be based on the resulting strength values. This of course raises the question on how to check the durability of alternative ‘green’ concretes.
4 HOW TO EVALUATE DURABILITY?

Current standards, such as the European EN 206, consider durability performance following ‘deemed to satisfy’ rules. Mainly based on long-term practical experience, minimum cement contents and maximum water/cement ratios are defined in order to obtain durable structures. This approach is practical and is quite reliable in case of well-known traditional binder systems (e.g. based on Portland cement or blast furnace slag cement), at least, on the condition that the casting, placing and curing of the concrete is also following the state-of-the-art rules.

For alternative binder systems, the European Standard defines the ECPC-concept (Equivalent Concrete Performance Concept). Following this concept, the durability performance of an alternative concrete has to be experimentally verified and compared with the durability performance of a well-accepted reference composition. Although the European Standard is not providing detailed prescriptions for the application of this ECPC-concept, some countries have adopted local standards giving more details, as is e.g. the case for Belgium with the Standard NBN B15-100 [7]. The ECPC-concept is considered to be more promising than the traditional k-value concept [17].

Figure 3: Combined effect of carbon footprint per m³ of concrete and increased carbonation rate on the carbon footprint during the entire service life.
A better solution would be to define absolute durability performance criteria, so that the durability performance of alternative concrete compositions can be directly verified without the need of performing comparative tests with reference mixes. The absolute durability performance could be estimated in a non-destructive way on site, on a finalized concrete structure, by special test methods, and following well-defined durability indicators [18,19]. This is a most-promising approach which needs to be further developed.

5 CONCLUSIONS

- Based on available test results for alternative concretes based on blended binders containing alternative materials, it is illustrated that the technical durability of the concrete can be very variable even when the concrete has similar strength.
- For the case of carbonation, based on Fickian diffusion, it is shown that the service life of a concrete structure could be significantly shortened in case of alternative binder systems, even when having similar strength.
- It is further illustrated that the carbon footprint of the concrete structure over the entire service life can be substantially higher in case of reduced technical durability, even if the carbon footprint per cubic meter of concrete is significantly lower.
- As a main conclusion it can be stated that the sustainability of concrete structures should be carefully investigated by duly considering the technical durability of the structurer. New approaches are available, including the ECPC-concept (Equivalent Concrete Performance Concept) and the concept of durability indicators.
- No concrete can be sustainable without being technically durable!

REFERENCES


TOWARDS PERFORMANCE BASED DURABILITY STANDARDS FOR WOOD IN CONSTRUCTION – PART 1: DELIVERING CUSTOMER SERVICE LIFE NEEDS

Christian Brischke (1), Ed Suttie (2), Finn Englund (3), Eric Heisel (4), Jöran Jermer (3), David Lorenzo (5), Marek Polášek (6), Sven Thelandersson (7) and Joris Van Acker (8)

(1) Leibniz University Hannover, Faculty of Architecture and Landscape Sciences, Institute of Vocational Sciences in the Building Trade, Hannover, Germany, brischke@ibw.uni-hannover.de
(2) Building Research Establishment (BRE), Watford, United Kingdom, suttiee@bre.co.uk
(3) SP Technical Research Institute of Sweden, Stockholm, Sweden, finn.englund@sp.se; joran.jermer@sp.se
(4) Technological Institute FCBA Laboratory of Biology, Bordeaux, France, eric.heisel@fcba.fr
(5) TECNALIA, Spain, david.lorenzo@tecnalia.com
(6) VVUD Timber Institute, Prague, Czech Republic, polasek@vvud.cz
(7) Lund University, Faculty of Engineering, Division of Structural Engineering, Lund, Sweden, sven.thelandersson@kstr.lth.se
(8) Ghent University, Faculty of Bioscience Engineering, Laboratory of Wood Technology, Ghent, Belgium, joris.vanacker@ugent.be

Abstract
There is an increasing need for consideration of performance classification for wood products in construction, as evidenced by the European Construction Products Regulation, and by warranty providers and end user demands for information. As a consequence this requires radical consideration how test methods to determine wood durability (in particular biological durability) can inform on service life and how they might be translated into a performance classification system. This paper describes the changes that have occurred in the past 5-10 years in Europe and how the trajectory of standards development is now on a different pathway. The collaborative European project PerformWOOD is described. It is one of the main responses to the classification and service life demands and is considering key issues such as material resistance, moisture risk and adaptation of existing standards. The current thinking on how to bring together these critical issues to inform end users of performance classification, and ultimately on service life, will be discussed in detail.
1 INTRODUCTION

The building sector is strongly requested to improve its cost effectiveness, quality, energy efficiency and environmental performance. At the same time the use of non-renewable resources needs to get reduced. In this respect wood and wood-based products can play a key role since they are generally low in embodied CO$_2$ and can be gained from sustainable forest resources. Wood has numerous further advantages compared to other building materials such as a high strength-weight ratio, good thermal insulation, and appealing aesthetics. However, its durability against different biological agents is limited and requires consideration when wood is exposed to moisture and thus favourable conditions for decay.

There is an increasing need for consideration of performance classification for wood products in construction, as evidenced by the European Construction Products Regulation, and by warranty providers and end user demands for information. A key issue for the competitiveness of wood is the delivery of reliable components of controlled durability with minimum maintenance needs and life-cycle costs. The development of performance-based design methods for durability requires that models are available to predict performance in a quantitative and probabilistic format. The relationship between durability during laboratory and field testing and the performance under in-service conditions needs to be quantified in statistical terms and the resulting prediction models need verification and adaptation according to performance in real life.

Research on the performance of wood and treated wood ranges from early predictive methodology [1], predictive models [2], service life prediction research [3-5] to current advanced modelling [6] and research [7-11]. There is a considerable wealth of existing research and knowledge [12].

The majority of existing durability test standards was developed for the assessment of wood preservatives. Now there are numerous additional ways of enhancing durability of a wood product (e.g. wood modification, water repellents, coatings, design). The tests are collectively used to determine if a treatment is fit for purpose in a specific end use, but they are in many cases ill fit to accommodate novel treatments and modifications of wood. More recent activity in the PerformWOOD project has concentrated on an accelerated programme of activity to tackle:

- Material resistance and classification, where the on-going revision of EN 350 [13] has enabled consideration of including permeability to water moving closer towards material resistance [14].
- Moisture dynamics and time of wetness, where on-going work seeks to gather considerable new data on moisture regimes in wooden test specimens [15-17].
- Data handling and variability, as different statistical tools are applied to biological test data to begin understanding best way to present variability and distribution data [18-20].
- Modelling and estimation of service life approaches, considering engineering approaches and combinations of key parameters [12, 21, 22].

2 MATERIAL RESISTANCE

The existing classification system for wood durability in Europe is based on EN 350 [13], where five classes between ‘very durable (DC1)’ and ‘non-durable (DC 5) are distinguished. The classification is based on relative values, e.g. the service life of specimens in field tests relative to non-durable references, or the relative mass loss of specimens in laboratory decay.
tests. However, from the standard it is so far not clear where the classification was derived from. An ongoing revision of EN 350 [13] aims at increasing the transparency of the classification system by indicating the respective data source. Besides ‘pure’ resistance against wood-degrading organisms the revised standard will also consider the ability of wood to withstand wetting (wetting ability). Within the PerformWOOD project, Round Robin tests are focussing on laboratory test methods to determine the wetting ability of wood. Based on the idea of the factor method [23] set values have been used for service life calculation for different wood-based materials by Thelandersson et al. [9]. Therefore material resistance classes had been defined and assigned to resistance indices (Table 1). This idea has been followed by Isaksson et al. [24] for an improved design guideline, where various factors to be considered for PSL have been quantified using performance models and data from long term field monitoring. Material resistance classes as consequence of durability against organisms and resistance against wetting are consequently foreseen for a new concept of performance classification within the PerformWOOD approach (see Figure 1).

Table 1: Resistance classification of selected wood materials and corresponding design resistance index $I_{Rd}$ (taken from the WoodExter Design Guideline, [9]).

<table>
<thead>
<tr>
<th>Material resistance class</th>
<th>Examples of wood materials</th>
<th>$I_{Rd}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Heartwood of very durable tropical hardwoods, e.g. Afzelia, Robinia (Durability class DC 1) Preservative-treated sapwood, industrially processed to meet requirements of use class 3</td>
<td>10.0</td>
</tr>
<tr>
<td>B</td>
<td>Heartwood of durable wood species, e.g. Sweet chestnut (DC 2)</td>
<td>5.0</td>
</tr>
<tr>
<td>C</td>
<td>Heartwood of moderately and slightly durable wood species, e.g. European larch and Scots pine (DC 3 and 4)</td>
<td>2.0</td>
</tr>
<tr>
<td>D</td>
<td>Slightly durable wood species having low water permeability, e.g. Norway spruce</td>
<td>1.0</td>
</tr>
<tr>
<td>E</td>
<td>Sapwood of all wood species (and where sapwood content in the untreated product is high)</td>
<td>0.7</td>
</tr>
</tbody>
</table>

3 MOISTURE RISK IN WOODEN COMPONENTS

Apart from in-ground exposures, where adverse moisture conditions can be assumed to exist at any time, a further distinction of fungal hazard is made regarding the respective moisture conditions and corresponding potential decay organisms. This has been done for defining use classes [25] as well as for the differentiation of climate-induced decay hazards [e.g. 26]. However, the variety of existing above ground test methods – representing very different moisture regimes - is also linked to limited comparability of the obtained results. Moisture content measurements could therefore serve as cross linking element between test methods, test sites and other boundary conditions for comparative studies. However, they are still sparsely used, and especially for real assemblies and commodities [e.g. 16, 27] moisture data are rare.
A second aspect of wood moisture content and its dynamics is its contribution to wood resistance. Besides biocidal ingredients of wood, hydrophobic substances and anatomic peculiarities have a significant impact on the moisture dynamics of timber and thus on its durability. Furthermore, impregnating timber with water repellents as well as modifying the wood cell wall aims on reducing the water uptake [28]. The capability of a wooden material to take up and accumulate moisture must consequently be seen as a second component of wood resistance (maybe the more important one). At least for the less severe UC 2 and UC 3.1 [25] moisture performance tests in the field might be seen as appropriate and time-saving alternative to long-term decay tests. The ‘time of wetness’ concept will therefore serve as key element in a dosimeter approach for performance classification in a revised EN 460 standard [29].

4 DATA HANDLING AND VARIABILITY

Paying attention to variability in test methodologies and data analyses is essential, especially for heterogeneous biological materials such as wood [18]. Increasing the number of test specimens usually improves the reliability of test results, but also increases their cost. Therefore it is important to optimize the amount of delivered data and to select the most relevant approaches making the characterization of the products possible, without presenting unnecessary cost burdens on those procuring the tests.

The variability which is often observed in the results of durability tests results from the variability of both the operators and the tested material itself. Variability among laboratories and/or technicians performing the same tests is frequently reported [e.g. 30, 31]. Although the same standards and protocols are used, their levels of complexity and the fact that the assessment criteria are often qualitative, and thus more subjective than quantitative criteria, explain why different results may be obtained. The variability of the tested material can be due to sampling, but also due to its intrinsic characteristics, which are influenced by the origin and the age of the tree and the position the test samples are taken from within the tree. When selecting a set of samples to be as similar as possible, the variability of the test is accounted for, but cannot be fully prevented. On the other hand a certain number of sources (assortments) should be considered to assure representativeness of the test results.

The classification of natural durability or biocidal efficacy is generally based on the mean or median values of the recorded data. However, the spread of individual values is sometimes wide, which makes the interpretation of the data complicated. Expressing the results in a way that will provide customers with relevant information regarding the level of natural durability is one major challenge for the future. The major risk of underestimating the importance and the impact of high variability, and thus of neglecting implementation of robust statistical approaches, is that manufacturers, users and authorities may be supplied with data whose reliability is doubtful. The statistical approach in CEN/TC 38 standards is being strengthened in order to deliver robust methodologies to the laboratories performing durability tests and, consequently, deliver more reliable information on wood-based products to the market.

5 PERFORMANCE MODELS

For service life planning and performance classification of buildings, building assets, and building products well-functioning ‘performance models’ are absolutely essential. The term
‘performance model’ is ambiguous in a double sense: On the one hand ‘performance’ needs to be carefully defined, because it can have very different meanings depending on the respective type of material, product, commodity and its application. On the other hand the general meaning of ‘model’ is the ‘schematic description of a system, theory, or phenomenon that accounts for its known or inferred properties and may be used for further study of its characteristics’, but it is not settled which factors will describe the command variable. Building components that are exposed outdoors to the weather are mainly affected by moisture and temperature related effects. In addition moisture and temperature can also play an important role indoors and in the building envelope. In particular for bio-based building materials such as wood biological agents require consideration for predicting their service lives. In contrast, other degradation processes such as corrosion, erosion, and hydrolysis play a minor role.

Efforts in developing performance models for both fungal decay and mould growth have been intensified in recent years. Various approaches have been followed and different models have been proposed to be implemented in design guidelines [12]. However, a high heterogeneity among these attempts became visible and different strategies have been followed.

The modelling attempts can be roughly distinguished according to the following criteria:

- **Objectives**: precise service life estimation (ideally in years) or comparative evaluation (to quantify effects, e.g. of design details; to compare alternatives, e.g. materials or design solutions)
- **Command variables**: mass loss, strength loss, remaining strength, decay ratings, decay depth, service life according to different limit states, aesthetic appearance, etc.
- **Data sources**: laboratory test data, field test data, survey data, expert opinion, reality checks, multi-source approaches, etc.
- **Level of accuracy/reliability**: Use and durability classes, dosimeter approaches, combined approaches

Furthermore from an engineering point of view decay and biological degradation in general are only part of the overall performance of wooden structures. A comprehensive engineering model can therefore be dominated by the effect of crack formation, ageing, UV degradation, corrosion of fasteners, or building physical phenomena.

With respect to the objectives of PerformWOOD it became clear that a lot of approaches for modelling service lives and performance of wood and wood products are already available. Many approaches have been implemented into design guidance documents already. Consequently, a framework of how exposure, dimension, design details, and the material-intrinsic ability to take up and release water can be linked to model the moisture risk in wood products is in principal available. In particular, the various dosimeter models could serve as reliable tools to quantify the effects of different construction details.

### 6 QUASI-CONTINUOUS CONCEPTION OF EXPOSURE DOSE - PERFORMANCE MODEL

The future sustainable use of the European forest resource lies with enhancing durability with environmentally improved and targeted systems to meet service life expectations. Work is progressing to take the first steps towards meaningful performance classification for wood
products so end users can be assured that wood is a reliable and thus low impact material. The outcomes of the project PerformWOOD are essential in moving forward to:

- Confirm a material resistance measure
- Confirm a moisture risk measure
- Kick start refinements to TC 38 standards and improve test methods
- Provide a draft standard (EN 460 [29]) for consideration of service life

As a major challenge we identified the complex task to unify a continuous approach for quantifying exposure dose and material resistance on the one hand and to provide guidelines that meet the user expectations on the other hand. The latter comprises a scheme with well-defined performance classes which can be applied during daily practice in the building trade. Therefore a quasi-continuous concept has been proposed, as illustrated in Figure 1. Dose and resistance are both considered to be continuous without defining static limit states. Nevertheless, discrete levels of performance can be defined and used for practical planning of buildings and constructed assets. For timber and wood-based building materials this requires considering exposure separately for the different potential degrading agents such as fungi, insects, termites, marine borers and wetting.

![Figure 1: Conceptual chart for determining ‘performance classes’ for a combination of material and exposure parameters.](image)

7 CONCLUSIONS

Project PerformWOOD has formalised and drawn together the on-going research to focus on generation of a material resistance factor for performance classification and alongside developing the first moisture dynamic test protocols in the history of CEN TC 38. A draft standard (EN 460) for consideration of performance classification of wood in construction is underway and relies on concepts that are being road-tested with industry, construction professionals, researchers and the general public. The work in the Working Groups of CEN TC 38 now really begins to continue to revise and sharpen the tests and methods needed to support such a performance classification framework as well as build the technical reports to underpin the future EN 460.
8 ACKNOWLEDGEMENTS

The authors gratefully acknowledge the support of the European Commission through the 7th Framework programme for funding project PerformWOOD a Coordination and support actions (Supporting) grant number 319132. Work programme topics addressed: NMP.2012.4.0-1 Support for standardisation needs to consolidate the technical background for standardisation to deliver new documents on the service life performance of wood in constructions.

9 REFERENCES


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TOWARDS PERFORMANCE BASED DURABILITY STANDARDS FOR WOOD IN CONSTRUCTION – PART 2: CONSIDERING MOISTURE RISKS IN WOODEN COMPONENTS

Christian Brischke (1), Linda Meyer (1), Ed Suttie (2)

(1) Leibniz University Hannover, Faculty of Architecture and Landscape Sciences, Institute of Vocational Sciences in the Building Trade, Hannover, Germany, brischke@ibw.uni-hannover.de, meyer@ibw.uni-hannover.de

(2) Building Research Establishment (BRE), Watford, United Kingdom, suttiee@bre.co.uk

Abstract

In the frame of the collaborative European research project “Performance standards for wood in construction – delivering customer service life needs – PerformWOOD” the immanent need for improvement of European standards dealing with wood durability has been identified. Potential of existing test protocols, but also challenges to enhance their capability to deliver performance and service life data have been investigated. One major task was a stronger emphasize on the role of moisture risk in wooden building components. A Europe wide survey among experts has been conducted to gather information about the significance of quantifying the moisture induced risk in wood durability testing and service life prediction of wooden components. Hereby the focus was on degradation by fungi and bacteria as well as on aesthetic aspects related to moulds.

This paper gives an overview of the results of this expert survey. On the one hand moisture was still felt to be inadequately considered through existing test protocols. On the other hand the outstanding role of moisture and wetting ability of wood-based products has been pointed out without fail. An inventory of standard but also non-standard laboratory and field test methods has been made. It became apparent that there exists a variety of test methods – mainly non-standardized ones – that act as makeshift to consider adequately different exposure to moisture of wood in the outdoor environment. Recommendations for future implementation of measuring moisture content and time of wetness in test standards have been developed.

1 INTRODUCTION

In principal, the natural durability of wood and wood-based products can be determined either in the field or in laboratory decay tests according to different standardized and non-standardized methods [1-3]. It is generally accepted that field tests provide more realistic test conditions, but often suffer from unacceptably long test durations. While in-ground tests with buried stakes need at least five years to give an indication on the effectiveness of wood preservatives [4-6], the onset of decay in above ground trials will take place significantly later, and service lives cannot be calculated before decades have passed [7, 8]. For these reasons results from laboratory decay tests as well as field test data from in-ground graveyard tests can be found quite frequently, but natural durability studies with respect to above-ground exposures are rare, although they play the more important role in timber engineering.
Moisture monitoring might serve as a cross linking element between long-term field exposure and short term tests. Apart from in-ground exposures, where adverse moisture conditions can be assumed to exist at any time, a further distinction of fungal hazard is made regarding the respective moisture conditions and corresponding potential decay organisms. Furthermore, the ability of wood to withstand wetting should be considered as an element of the material-intrinsic ‘resistance’, which might be implemented in the upcoming revision of the European standard EN 350 [9,10]. To allow also for short-term results laboratory methods are sought to provide information about the ‘wetting ability’ of wood-based materials.

The suitability of a moisture content measurement method depends on the respective study objectives. For determining the time of wetness (in terms of critical moisture conditions for the onset of decay) the accuracy of methods needs to be high below and above fibre saturation. For very low and very high moisture contents less accuracy can be accepted. More critical is the impact of other parameters affecting the measurement results such as temperature, density and altered substrate properties (modification, impregnation).

Within the European research project “Performance standards for wood in construction – delivering customer service life needs – PerformWOOD” an immanent need for improvement of European standards dealing with wood durability has been identified. Potential of existing test protocols, but also challenges to enhance their capability to deliver performance and service life data have been investigated. One major task was a stronger emphasize on the role of moisture risk in wooden building components. A Europe wide survey among experts has been conducted to gather information about the significance of quantifying the moisture induced risk in wood durability testing and service life prediction. Hereby the focus was on degradation by fungi and bacteria as well as on aesthetic aspects related to moulds.

2 SURVEY ON “MOISTURE RISK AND WOOD DURABILITY TESTING”

A web-based survey on “Moisture risk and Wood Durability Testing” was carried out among in total 177 scientists from 37 European countries (see Figure 1).

Figure 1: Web questionnaire on ‘Moisture risk and wood durability testing’ – welcome page.
The survey aimed on gathering information about the significance of quantifying the moisture induced risk in wood durability testing and service life prediction of wooden components. Hereby the focus was on degradation by fungi and bacteria as well as on aesthetic aspects related to moulds. The participants had been asked to provide information about their practice in durability testing. The questionnaire was therefore structured according to laboratory tests, field tests, monitoring of structures and finally development of models and service life prediction. According to the participants’ answers the questionnaire was adapted to their respective activities in wood durability research. The estimated time for processing the questionnaire was 20 min.

3 OUTCOME OF THE SURVEY

In total 45 replies from 18 European countries were received which is equivalent to a percentage return of 25.4 %. In total 44 questions were raised with regard to different issues related to durability and moisture performance testing. In the following selected findings are presented and discussed. A more detailed description of the survey has been published by Brischke et al. [11].

The majority of respondents are conducting durability tests as well as moisture related tests under laboratory conditions and in the field (Figure 2). However, the lowest value (65 %) was obtained for MC measurements in the field.

![Figure 2: Percentage of test types performed.](image)

It was further asked which materials are tested with respect to their moisture performance (Figure 3). Native solid wood ranked highest followed by thermally modified wood. Chemically modified wood, preservative treated wood and panels had similar percentages; wood polymer compounds were tested by only 23 % (field: 12 %) of the respondents. Furthermore, single institutions are testing the moisture performance of wood treated with
water repellents, bio-based insulation materials, concrete, finger jointed and laminated components, windows, claddings, and coated wood.

For moisture monitoring and performance testing in the field different MC measurement methods are in use. Most frequently used are manual gravimetric MC measurements and electrical resistance measurements (Figure 4). Less frequently used are capacitive and hygroscopic measurements, which might be explained by their limitations regarding accuracy and measurement range respectively. Automated gravimetric measurements using load cells are also rather rarely applied (7 %) – seemingly due to high investment costs. Among those, who replied that they are not performing MC measurements in the field, the most often mentioned reason was that this kind of measurements is simply not requested (19 %).

As expected not only type of MC measurement and the applied methods differed between laboratories, but also the aims of moisture performance testing were divergent. This becomes
evident from the ‘kind of moisture’ the different respondents were interested in as shown in Figure 5.
While gravimetric MC measurements do not allow determining ‘local MC’, the resistance measurements do not register the bulk moisture of a specimen or building component (‘global MC’), but critical details and their effect on MC can be examined. Various resistance based sensors also allow detecting MC gradients from the surface to the interior of a wooden piece [12, 13].

Figure 5: ‘Kind of moisture content’ of interest.

In addition to a questioning on test methodology, it was also asked how and for which purpose the collected data are used and will be further processed. In particular we were
interested to which extent data are used for modelling the service life of wooden components. Since 30% of the respondents were developing models to predict performance and/or service life of wood products it was also of interest which factors they consider as dose parameters and which for the corresponding response (Figure 6 and Figure 7).

Interestingly, numerous factors, direct and indirect factors (Brischke et al. 2006) were recorded and analysed to determine the dose for wood degradation. The most prominent factors were air temperature, relative humidity, wood moisture content and temperature, time of wetness and rain. In the face of these dose parameters the following response measures were mentioned nearly equally often: Service life, mass loss, strength loss, decay rating, and aesthetic appearance (mould growth).

Finally, the interviewees have been asked to comment on several statements with respect to moisture performance testing. Therefore a six-step rating scale was used (0 = ‘I do not agree’ till 5 = ‘I fully agree’). In Table 1 the overall replies are presented as percentage of the single ratings as well as an average rating. The majority agreed wetting ability is an essential parameter to determine wood resistance and should be determined in addition to traditional decay resistance. Discordance became evident with the question if suitable methods to determine wetting ability do already exist or are even already established. While the majority again would appreciate if MC measurements were more frequently applied in the field, the respondents were apparently not so sure the added value can justify the extra time and cost. Finally, a remarkable percentage of the respondents were not convinced that the reliability of MC measurements is sufficient to provide performance data.
Table 1: Response on statements between ‘0 = I do not agree’ and ‘5 = I fully agree’.

<table>
<thead>
<tr>
<th>Statement</th>
<th>Percentage of replies [%]</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>Mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wetting ability is an essential parameter to determine wood resistance against decay</td>
<td></td>
<td>2</td>
<td>0</td>
<td>2</td>
<td>14</td>
<td>33</td>
<td>49</td>
<td>4.3</td>
</tr>
<tr>
<td>In addition to traditional decay resistance tests, wetting ability should be determined</td>
<td></td>
<td>2</td>
<td>0</td>
<td>7</td>
<td>19</td>
<td>33</td>
<td>39</td>
<td>4.1</td>
</tr>
<tr>
<td>Suitable methods to determine wetting ability of wood are available</td>
<td></td>
<td>9</td>
<td>7</td>
<td>28</td>
<td>37</td>
<td>14</td>
<td>5</td>
<td>2.6</td>
</tr>
<tr>
<td>Suitable methods to determine wetting ability of wood are already established</td>
<td></td>
<td>16</td>
<td>19</td>
<td>35</td>
<td>18</td>
<td>12</td>
<td>0</td>
<td>2.0</td>
</tr>
<tr>
<td>MC measurements have the potential to reduce test durations of field tests</td>
<td></td>
<td>7</td>
<td>12</td>
<td>28</td>
<td>21</td>
<td>25</td>
<td>7</td>
<td>2.7</td>
</tr>
<tr>
<td>Continuous MC measurements in the field should be performed more frequently</td>
<td></td>
<td>5</td>
<td>0</td>
<td>12</td>
<td>21</td>
<td>32</td>
<td>30</td>
<td>3.8</td>
</tr>
<tr>
<td>Added value by performing MC measurements does not justify extra time and cost</td>
<td></td>
<td>23</td>
<td>28</td>
<td>16</td>
<td>26</td>
<td>5</td>
<td>2</td>
<td>1.7</td>
</tr>
<tr>
<td>Reliability of MC measurements is insufficient to provide performance data</td>
<td></td>
<td>16</td>
<td>23</td>
<td>16</td>
<td>26</td>
<td>12</td>
<td>7</td>
<td>2.2</td>
</tr>
</tbody>
</table>

4 CONCLUSIONS

In summary, different methods for moisture risk assessment have potential to be applied in the field. Accompanying measurements in addition to traditional field durability testing can provide valuable extra information in particular with respect to materials showing improved wetting resistance such as many modified and water repellent treated wood products. Since long monitoring intervals are usually required and moisture levels far above fibre saturation can be expected the use of automated electrical resistance measurements turned out to be the most promising method. Other methods such as gravimetric systems suffer from being more time and cost consuming, but can provide an indication of moisture risks even when they are applied not continuously.

The use of methods to determine moisture content and time of wetness as an alternative rather than an additional feature to traditional decay assessment requires generally different qualities: The outcome of the PerformWOOD survey already showed that A. the reliability of MC measurements needs further exploration and B. the existing data base is not sufficient to convince the majority of experts.

The need to consider the wetting ability of wood as self-contained parameter which has an essential effect on wood durability has been recognised by scientists as well as responsible experts in standardisation bodies. A first step towards new test protocols for determining the wetting ability of wood under laboratory conditions has been made by initiating a round robin trial in liaison with CEN task group TG EN 350. Preliminary results indicated that both adsorption and desorption as well as short, medium and long-term water uptake need consideration. A draft standard test protocol for determining the wetting ability of wood is...
under progress, but further data – in particular from long-term field measurements are needed before it will be established.

5 ACKNOWLEDGEMENTS

The authors gratefully acknowledge the support of the European Commission through the 7th Framework programme for funding project PerformWOOD a Coordination and support actions (Supporting) grant number 319132. Work programme topics addressed: NMP.2012.4.0-1 Support for standardisation needs to consolidate the technical background for standardisation to deliver new documents on the service life performance of wood in constructions.

6 REFERENCES

REPEATED AUTOGENOUS HEALING IN CEMENTITIOUS COMPOSITES WITH MICROFIBRES AND SUPERABSORBENT POLYMERS

Didier Snoeck (1), Stijn Debaecke (1) and Nele De Belie (1)

(1) Magnel Laboratory for Concrete Research, Ghent University, Belgium –
didier.snoeck@ugent.be; nele.debelie@ugent.be

Abstract
Cementitious materials are sensitive to crack formation and it would be beneficial if the material could reach again the original strength. Autogenous healing is an already-present feature in cementitious materials, but it is an inferior mechanism as it can only heal cracks up to 50 µm in the presence of water. Therefore, a cementitious material with synthetic microfibres and superabsorbent polymers (SAPs) is proposed. The microfibres will cause multiple crack formation and will result in a vast amount of small healable cracks. If superabsorbent polymers (SAPs) are also included, the self-healing can be promoted as SAPs are able to extract moisture from the environment and to provide it to the cementitious matrix for autogenous healing. But, if the building blocks are exhausted due to a first healing cycle, healing at second reloading may be less efficient. A crack can thus possibly heal only once. In this study, the ability of (promoted) autogenous healing to repeat itself is investigated by comparing the mechanical properties after performing repeated four-point-bending tests. Specimens are able to heal and to regain some of the mechanical properties after being preloaded and pre-cracked under four-point-bending. Even if those healed samples are reloaded for a second time, there is some regain in mechanical properties. If SAPs are added, there is even healing in an environment without liquid water (relative humidity of more than 90%), also in repeated healing actions. The cementitious composite with microfibres and SAPs thus shows partial repeatability of self-healing.

Keywords
Self-healing, four-point-bending, durability, hydrogels, CaCO₃
1 INTRODUCTION

Cracks occur in concrete due to its low tensile strength. These cracks have a negative effect on the durability of the structure because harmful substances may enter and deteriorate the concrete. This results in repair works which are labour-intensive and expensive. Concrete, however, has the ability to heal itself by closing small cracks, referred to as autogenous healing.

Two mechanisms are mainly responsible for this form of passive healing: the continued hydration of unhydrated cement grains and the precipitation of calcium carbonate (CaCO₃) [1, 2]. In the last case, CO₂ dissolves in water to react with Ca²⁺ ions present in the mortar matrix (Fig. 1). A detailed study on the autogenous healing of cracks in concrete was performed by Edvardsen [3]. She found a reduced permeability in time due to autogenous healing and performed an intensive study on the laws which govern the nucleation and crystal growth processes. The decrease in permeability demonstrates the potential of the autogenous healing as there is less intrusion of durability-decreasing substances.

![Figure 1: Autogenous healing mechanism by further hydration (a) and calcium carbonate crystallization (b). Figure redrafted after ter Heide [4].](image)

A small crack, with a crack width smaller than 50 µm, is needed for high-strength concrete to be able to heal the crack completely. If not, the products available in the mortar mix are consumed before the crack is effectively closed. The wanted cracking behaviour is obtained by adding microfibers to the mortar mix, typically 2 vol%. This results in a concrete with a high tensile ductility, strain hardening and small crack widths in the range of 20–80 µm [5, 6]. Cracks smaller than 50 µm show complete healing and cracks up to 150 µm show partial healing [5]. Without the presence of water, the composite does not heal. Recent studies aimed at a crack width of maximum 30 µm to ensure a complete healing of the crack by means of autogenous healing [7].

The self-healing capability of concrete can be improved by adding superabsorbent polymers (SAPs). SAPs have the ability to absorb and retain a vast amount of liquid without dissolving (Fig. 2). The use of SAPs to promote the self-healing is dual. SAP-particles swell during the mixing process and shrink during the hardening of the concrete, leaving behind macropores [7, 8]. These macropores act as initial flaws and promote multiple cracking. Secondly, SAP-particles are very useful during the self-healing as they absorb water during wet periods and slowly release it during dry periods. Water is thus continuously available for
the self-healing process [7] and the composite showed a complete regain in mechanical properties, with healing of cracks up to 130 µm. Also, as the SAPs swell, they will initially seal a crack from intruding fluids, thus increasing the durability [9]. In this way, a smart cementitious material which is reliable and independent from external conditions is acquired.

Figure 2: Visualization of a dry and saturated superabsorbent polymer particle.

If we consider Fig. 3a, the durability performance of a construction is higher than the required performance. But, if there is excessive loading and/or cracking, this durability parameter may shift to lower values and degradation may occur. If one does not notice the failure and does not repair, the construction may decline. The dashed curve shows the costs. The costs will increase stepwise in time due to manual repair and maintenance monitoring [10].

Figure 3: Durability performance and costs as a function of time for manual repair (a), and for a self-healing material (b). Figure redrafted after van Breugel [10].

If we would use an ideal self-healing material, the durability would be guaranteed (Fig. 3b). But, in case of autogenous healing, the amount of available building blocks is consumed in time, thus limiting the possibility of renewed crack healing. So, the question rose whether this autogenous healing could be repeated. What will happen when autogenously healed specimens are subjected to cracking again? In this paper, the possibility of a repeated autogenous healing is investigated by performing four-point-bending tests on self-healing materials.
2 MATERIALS AND METHODS

2.1 Materials

The mortar mixtures contain 608 kg/m³ CEM I 52.5 N, 608 kg/m³ Class F fly ash, 426 kg/m³ silica sand ($D_{50} = 170 \mu m$; Sibelco, Belgium), 365 kg/m³ water, a polycarboxylate superplasticizer (Glenium 51, conc. 35%), 2 vol% of Polyvinyl-Alcohol (PVA) fibres (Kuraray, Japan), and a varying amount of SAP expressed as mass-% (m%) of cement weight. Two types of SAP from BASF were used: SAP A being a copolymer of acrylamide and sodium acrylate (particle size $100.0 \pm 21.5 \mu m$); SAP B, a crosslinked potassium salt polyacrylate ($476.6 \pm 52.9 \mu m$).

To assess the sealing capacity of SAP, the swelling capacity was calculated from the volume increase between the vacuum dried state and the saturated state. A fluid was added to vacuum dried SAP particles and the whole was filtered after one day. The amount of filtered fluid was recorded. To ensure there was no influence of the filter paper, the latter was saturated with the fluid prior to filtration. The measurements were performed with de-ionized water and filtered cement slurry (obtained by mixing 10 g CEM I in 100 g of de-ionized water). The absorption capacity is $305 \pm 4$ g de-ionized water/g SAP A, $283 \pm 2$ g de-ionized water/g SAP B, $61 \pm 1$ g filtered cement slurry/g SAP A and $58 \pm 2$ g filtered cement slurry/g SAP B. The SAP particles are also able to extract 4 times their weight in moisture from an environment at 98% RH.

2.2 Methods

2.2.1 Mixing, casting and storage procedures

First, the solid components (cement, fly ash and SAPs) were equally distributed with a mortar mixer. Then, water and superplasticizer (30 s at 140 rpm) were added. Silica sand was added for the next 30 s at 140 rpm. To ensure a homogeneous dispersion of all components, the speed was increased for the following 30 s to 285 rpm. The edges of the bowl were scraped and there was a total resting period of 90 s. Next, at a speed of 140 rpm, microfibres were slowly added during 30 s. The final step was mixing for 60 s at 285 rpm.

A comparison of the flow value of mixtures with and without SAPs was used to reflect the absorption by the SAPs in the mortar mixture [11]. The material was hereby spread by jolting a plate 15 times. The amount of additional mixing water was increased several times in several mixes with the same amount of SAPs until the flow value corresponded to the flow value of the same mixture without SAPs.

Moulds were filled and the samples were compacted by jolting a plate 60 times. The samples were demoulded after 24 h and were stored at a relative humidity of 95 ± 5% and 20 ± 2°C until the age of 28 days. Series used for four-point bending tests (Table 1), consisted of minimum three $160 \times 40 \times 10$ mm³ samples with 2 vol% of microfibres.

2.2.2 Four-point-bending and curing conditions

Cracks were created in the specimens by a four-point-bending test at the age of 28 days. A servo hydraulic testing system (Walter+Bai DB 250/15) ensured a displacement-controlled test (0.0015 mm/s to imitate a quasi-static load). The strain at the bottom side of the specimen was limited to 1%. This strain is lower than the maximum strain, so the service cracks could be studied. After cracking, the samples were cured at 20 ± 2°C by applying wet/dry cycles (alternatively stored in water for 12 h, and at a relative humidity (RH) of 60% for 12 h), or by placing them in a room with a RH > 90% or RH = 60%.
Table 1: Explanation of the different samples codes.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>m% [%]</th>
<th>SAP type</th>
<th>Curing</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 - 60</td>
<td>0</td>
<td>-</td>
<td>RH = 60%</td>
</tr>
<tr>
<td>0 - 90</td>
<td>0</td>
<td>-</td>
<td>RH &gt; 90%</td>
</tr>
<tr>
<td>0 - wd</td>
<td>0</td>
<td>-</td>
<td>wet/dry</td>
</tr>
<tr>
<td>0.5A - 60</td>
<td>0.5</td>
<td>A</td>
<td>RH = 60%</td>
</tr>
<tr>
<td>0.5A - 90</td>
<td>0.5</td>
<td>A</td>
<td>RH &gt; 90%</td>
</tr>
<tr>
<td>0.5A - wd</td>
<td>0.5</td>
<td>A</td>
<td>wet/dry</td>
</tr>
<tr>
<td>0.5B - 60</td>
<td>0.5</td>
<td>B</td>
<td>RH = 60%</td>
</tr>
<tr>
<td>0.5B - 90</td>
<td>0.5</td>
<td>B</td>
<td>RH &gt; 90%</td>
</tr>
<tr>
<td>0.5B - wd</td>
<td>0.5</td>
<td>B</td>
<td>wet/dry</td>
</tr>
<tr>
<td>1B - 60</td>
<td>1</td>
<td>B</td>
<td>RH = 60%</td>
</tr>
<tr>
<td>1B - 90</td>
<td>1</td>
<td>B</td>
<td>RH &gt; 90%</td>
</tr>
<tr>
<td>1B - wd</td>
<td>1</td>
<td>B</td>
<td>wet/dry</td>
</tr>
</tbody>
</table>

After a period of 28 days, the specimens were reloaded in four-point-bending and the mechanical properties obtained during the first and second loading cycle were compared. These are the first-cracking-strength and the regain in first-cracking-strength. The first-cracking-strength is obtained just before the first drop in stress due to an unstable extension in the matrix fibre tunnel. Again, the strain was limited to 1%. After another 28 days of curing, the specimens were reloaded for a second time, till failure in the third loading cycle. From the obtained stress-strain curves, the first-cracking-strength and regain in first-cracking-strength were compared once again.

Figure 4 represents the stress-strain curves of a specimen healed in wet/dry-cycles and three parts are visible. The first loading part is the preloading (strain from 0% until 1%), the second part is the first reloading (an additional strain of 1%) and the third part is the second reloading (until failure).

During the healing periods, microscopic observations were performed at regular time intervals by means of a stereo microscope (Leica S8 APO with DFC 295 camera). The visual closure hereby served as a qualitative measurement for the autogenous healing capacity.
3 RESULTS

Due to the addition of microfibers, multiple cracking was observed, giving all samples a ductile strain-hardening behaviour. The crack width is hereby a very important parameter. If large cracks are formed, the building blocks will be consumed after some time and the autogenous healing will no longer be optimal. In this research, the crack widths were limited to 30-50 µm, as was also the case in [5]. Due to the addition of SAP particles, this multiple cracking behaviour was enhanced. The macropores formed by the SAPs act as crack initiators, increasing the ductile behaviour. Also, the crack width tended to decrease slightly, but the difference was not statistical significant. Due to partial reopening of the cracks at first reloading, the crack width increased slightly. Again, the differences were not significantly different. This was confirmed by microscopic observations and analysis of the crack widths.

By examining the crack width before and after curing, the percentage of crack closure could be quantified. Figure 5a shows the visual crack closure in function of the initial crack width after the first healing cycle. Results show that cracks up to 30 µm heal completely and up to 150 µm heal partly when specimens are subjected to wet/dry cycles. Also Yang [5] and Li et al. [6] had similar results, but their limit of total crack healing was 50 µm instead of 30 µm. They also showed that, when stored in ambient air (without any liquid water available), samples cannot heal. It is clear that specimens healed under wet/dry cycles exhibit the best healing as a fair amount of water is available for autogenous healing. If superabsorbent polymers are added as well, the amount of healing is slightly higher. This is due to the fact that SAPs are able to seal a crack from intruding fluids, reducing the flow and thus establishing better circumstances for autogenous healing. Secondly, water is also available during the dry periods of the wet/dry cycle, thus increasing the possibility of autogenous crack healing. At a relative humidity of more than 90%, the healing is far less. However, specimens with superabsorbent polymers show partial healing. As SAPs are able to extract moisture from the environment, they will swell to a small extent, and then they will provide the water for further hydration of unhydrated binder particles in the crack. At a relative humidity of 60%, only the mixtures with SAPs show partial healing.

![Figure 5a](image1.png)

**Figure 5a:** Crack closure [-] after the first healing stage as a function of initial crack width (a).

![Figure 5b](image2.png)

**Figure 5b:** Crack closure [-] after the first healing stage as a function of initial crack width (b).
After the first healing period, most of the building blocks for self-healing, i.e. unhydrated cement particles and Ca^{2+} ions are consumed, reducing the possibility for autogenous healing. Specimens with SAPs in the second wet/dry cycle for example show an inferior visual closure of the crack compared to the same specimens in the first healing cycle (Fig. 5b).

Cracks may even be closed with deposited crystals after the two healing cycles. The strength of the new material was investigated by analysing the results from the loading experiments. The first-cracking-strength of all studied mixtures is shown in Fig. 6a. The first-cracking-strength for the mixtures without SAPs is higher than for the mixtures with SAP A. Due to the macropores in the mixtures with SAP-particles, the active cross-section is reduced, resulting in a lower first-cracking-strength. The macropores formed after emptying the water-filled SAP inclusions may promote multiple cracking behaviour as they serve as crack initiators. The strength is not reduced significantly when using the larger SAP B particle.

Samples with SAPs show a higher regain in first-cracking-strength than the SAP-free samples in all studied healing conditions (Fig. 6b). The regained first-cracking-strength for the samples healed in wet/dry-cycles is higher than for those healed in an environment with a RV > 90% which, in turn, is higher than for those healed in an environment with a RV = 60%. Healing in wet/dry-cycles provides the necessary water for the hydration of cement particles and the puzzolanic reaction of fly ash. This results in a regain in first-cracking-strength. The regain is higher for mixtures with SAPs than for mixtures without SAPs. In mixtures with SAPs, more water is available for the continued hydration of cement, puzzolanic activity and precipitation of calcium carbonate due to the absorption capacity of the SAP-particles. Increasing the amount of SAP from 0.5 m% to 1 m% also increases the regain in strength.

Ca^{2+} ions are consumed at the crack faces after the first healing period. This is visible in the results on visual crack closure and the regain in mechanical properties. The visual crack closure is about 20% lower when the specimens are reloaded for a second time. During the first reloading, only a limited number of new cracks were formed. So mostly the previous healed cracks reopen and there is a limited possibility for a renewed autogenous healing of the
crack. Most unhydrated cement particles are now hydrated and the Ca\(^{2+}\) ions are mostly consumed to form calcium carbonate crystals. In the second healing stage, Ca\(^{2+}\) ions present in the interior of the matrix need to diffuse towards the crack face. So, the crystallization becomes diffusion-controlled instead of surface-controlled as free Ca\(^{2+}\) ions in the matrix need to travel towards the crack faces. This limits the speed of possible crystallization and thus autogenous healing and regain in mechanical properties.

SAP particles not only tend to increase the healing capacity at the first healing stage (from 46\% to 75\% in wet/dry cycles), but also at the second healing stage. The regain is still 66\% at second reloading. Without the superabsorbent polymers, this value was only 28\%.

The overall healing and closure of a crack may lead to less ingress of potentially harmful substances, thus increasing the durability and service life of civil structures.

4 CONCLUSIONS

SAP particles promote self-healing by providing water upon crack formation and this results in more visual crack closure and more regain in mechanical properties. The main mechanisms of self-healing are further hydration of unhydrated particles and the precipitation of CaCO\(_3\) on the crack faces. To a certain degree the autogenous healing capability of cementitious materials is maintained during subsequent loading cycles. Cracks heal and close during a second healing cycle and there is a regain in first-cracking-strength of specimens stored in wet/dry cycles. Even in an environment with a RV > 90\%, there is a non-negligible healing capacity noticeable. Even though the active cross-section is reduced due to the macropores resulting in a smaller first-cracking-strength, this effect is not significant when using larger SAP particles. The smart material with SAP B is thus an excellent material to use in future building applications.

ACKNOWLEDGEMENTS

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STRUCTURAL RECYCLED CONCRETE SCIENCE AND MARKET: A SUCCESS STORY

Marco Viviani (1)

(1) University of Applied Sciences Western Switzerland – HEIG-VD, Yverdon les Bains – marco.viviani@heig-vd.ch

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Author contacts HEIG-VD, Rte de Cheseaux, 1 Yverdon-les-Bains, Switzerland – marco.viviani@heig-vd.ch

Abstract
Recycled concrete is possibly one of the best examples of the impact of a single word on commercial success. A product should be evaluated for its performances and added value rather than for its name. Against all odds and researches, recycled concrete is often refused because is not “new” but is “recycled” and many people do not want to pay the same money for a second-hand product. Naturally, “rules” has always been the best incentive for recycled concrete (typically set like: either you use recycled concrete or your construction will not meet the environmental standards). But can a civil engineer be blamed if he fears the use of recycled concrete? This article describes the reasons for fearing and the facts for rely on recycled structural concrete.

1. STATE OF THE ART
When it comes to design a structure, few details are as important as the materials that will be specified. Often the shape and size of the structural element can be little modified due to the requirements of the architects, the owners, the laws, the codes and construction methods. Theoretically the engineers have a wide choice of construction materials but, in the real world it is not easy to obtain whatever type of material is wanted. Concrete is a remarkable example: if a C50/60 certified concrete is required, it will not be available in many regions. The reason is simple: due to the low demand by the market of this specific concrete type, no ready mix plants will invest to certify it according to the code EN 206 [1]. According to the European Ready Mixed Concrete association, in the 2012 the amount of concrete with strength class ≥
C30/35 was 11%, 2% lower than in 2011 [2]. This statistic is made on approximately 360 million cubed meters produced by the ERMCO associates. Furthermore, few engineers will take the responsibility of using a concrete that is not certified, since they will be legally responsible of whatever problem of durability and strength. According to the code EN 206 [1] and SIA 262/2013 [3] the engineer is responsible to prescribe the performances of the concrete he desires. Often, the engineer is also responsible of preparing a specification document that will be used for bidding, and definitely to define a budget. If a concrete C50/60 certified EN 206 is required by the engineers, most of the producers will respond that they will indeed supply a concrete with mean (cube) strength of 70 Mpa but they will definitely not supply a certified concrete. At this point, the engineer can accept the risk of liability or pay. Pay for the time and the costs of certification of a concrete, pay because he has stalled the project and for the time he will spend in modifying the project.

The same situation occurs if the engineer requires without a specific reason that a standard, certified concrete (for example a common C30/37) has one or more properties that are different from the ones that are usually set by national codes (flexural strength, elastic modulus, shrinkage, etc). Asking for a certified concrete C30/37 to have an elastic modulus of 45000 might generate troubles to find a supplier. The explanation for this is that the norm SN EN 262 [3] links the elastic modulus to the strength and the nature of the aggregates. Therefore to have a specific elastic modulus on a specific concrete strength class possibly means to abandon the usual mix-design and have different aggregates supplied (maybe from another country). But in this case the producer will be obliged to recertify the concrete and pay for it. If this is true for the mechanical properties, it is not different for the durability of a specific concrete. A regular, normal concrete have some durability performances guaranteed i.e is sold as fit for an exposure class. A ready-mix plant might sell a concrete that is, for example, resistant to the sulfate attack and therefore to the exposition class XS. Traditionally, all performance-based codes require, for a certain level of SO42- ions in the soil or groundwater, that some mix design parameters and some physical properties are guaranteed (by instance a low permeability of the concrete). These requirements are deemed satisfied if a lower w/c ratio, a minimum cement quantity , a cement sulfate resistant i.e. with low C3A content, etc. is used [4-6]. Sometimes it is demanded to isolate the concrete from the aggressive environment (with a waterproof membrane). All the mentioned actions are required against the external sulfate “attack”. When it comes to the internal sulfate attack, the chemical composition of the aggregates is of capital importance. Aggregates should not be polluted or contain more than a “critical” quantity of sulfate ions. Researchers agree that, in the “ideal hydration process” all sulfate used to regulate the setting time of cement is consumed during the early phases of the reaction, and therefore there should not be any pollution due to the gypsum (set regulator). Furthermore, the use of composite cements reduces the overall need of set regulator due to the lower clinker factor. Therefore the recycled concrete aggregates (hydrated concrete crushed) should not have any particularly
high quantity of sulfates due to the hydrated cement they contain and the fact that the sulfate ions content into recycled aggregate is higher than in normal concrete is considered reasonable and the soil of accepted sulfate extracted by acids is 0.8%. In the real world, however, it is always better to test the sulfate quantity, since the hydration process is rarely “ideal”. Recycled concrete aggregates cannot (or should not) be demolished and immediately used in a new construction unless their properties are carefully tested (according to the SN EN 206 standard, all components of a certified concrete should be certified). The right procedure is thus to stock and subsequently process and homogenize large amounts of recycled concrete aggregate and then to characterize it. By this way it will be known if the recycled concrete aggregate will be of suitable quality to build structures.

The structural response of a RC building it is certainly influenced by the durability and by the mechanical characteristics (such as compressive strength) but it is also deeply influenced by the concrete stress-strain behavior. Since the early studies on the RC concrete design (Hennebique system), it has been clear that properties of a RC structural element (such as the bending moment capacity) depend also on the shape of the uniaxial compression stress-strain curves of concrete. As far as today, the accepted shapes of stress-stress curves are substantially three, the most exact a sort of parabolic curve. SIA 262 proposes a parabolic rectangular curve with this shape:

![Figure 1 a parabolic-rectangular stress-strain curve as for the SIA 262/2003](image)

Some authors [7,8] investigated the fundamental stress–strain curve of recycled concrete under uniaxial compression and its features. One of the main results of Xiao’s research [7] was that recycled concrete has a slightly fragile behavior after the peak compressive strength, fragile behavior that increases with the (recycled aggregate) substitution ratio. This matter is of extreme importance, because most software used in structural calculation uses the stress-strain model proposed by codes such as the SIA 262/2013 [3] and user-defined curves are not largely used by professional engineers. Furthermore, even if an engineer was willing to insert a user defined stress-strain curve of recycled concrete, the data available would be too few to be generalized. The question is therefore, if the usual stress-strain models are appropriate for recycled concrete and if the norms should take into account this aspect.

Recycled concrete is an unfortunate name for a product, because too many people recall something “used” of lower quality. Eco-concrete is a name with a good marketing potential but is disputed by whose maintaining that recycled concrete requires “more cement” to have
“the same” mechanical properties and therefore it is not “Eco”. The paradigm that recycled concrete “needs” more cement is not true and is often accepted on the conviction that a “recycled concrete” can be obtained just swapping some natural aggregate with some recycled aggregate, keeping the same mix design. This is rarely possible and, especially in concretes with low paste content [9]. Some eco-balances of recycled concrete have set as hypothesis that it is indeed necessary to add cement [10]. Therefore, under this assumption, if recycled concrete is still interesting because less natural resources need to be used, it is, however, not interesting because of the energy necessary for this extra cement. In this article it will be shown that recycled concretes does not need “more cement” to have suitable mechanical properties.

2. MATERIALS AND METHODS

Recycled concrete aggregate samples were collected according to the norms on the large stockpiles set aside to form the stock for recycled concrete. The materials used in this research are available on the local market (concrete, recycled and standard aggregates are sold also separately). The concrete used in this study is a SN EN 206 certified concrete, available on the local market for a price lower than the ordinary concrete of the same strength class. The mix design of the standard (SC) and recycled (RC) concrete is shown in Mix design of RC and SC concrete 1-3 concretes 1.

Table 1 Mix design of RC and SC concrete 1-3 concretes

<table>
<thead>
<tr>
<th>Composition</th>
<th>Recycled Concrete RC C30/37 XC4 Cl 0.20 Dmax32 C3</th>
<th>Standard Concrete C30/37 XC4 Cl 0.10 Dmax32 C3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement :</td>
<td>350 kg/m3</td>
<td>350 kg/m3</td>
</tr>
<tr>
<td>Water :</td>
<td>168 kg/m3</td>
<td>168 kg/m3</td>
</tr>
<tr>
<td>Plasticizer :</td>
<td>0.9 %</td>
<td>0.9 %</td>
</tr>
<tr>
<td>aggregates :</td>
<td>28-30 % recycled</td>
<td>0 % recycled</td>
</tr>
<tr>
<td>price :</td>
<td>Fr. 188.50</td>
<td>Fr. 190.00</td>
</tr>
</tbody>
</table>

Table 1 does not clarify a simple truth: Recycled concrete can rarely be obtained by swapping a part of the standard aggregate with some crushed concrete. Apparently the mix designs are the same but, the way in which the recycled concrete is produced and processed as well as the proportion of the aggregates fractions and their nature (partly round, partially crushed, in this case), creates two completely different mix designs. Therefore a certified and performing recycled concrete can be obtained only if its mix design is carefully studied.

All concrete was produced in a stationary batch plant, mixed for 60 seconds in a two shafts 3 m3 (max capacity) mixer. This was done to test a recycled concrete available on the market.
and not a “labcrete”. Each lot of fresh concrete used to prepare the specimens was taken from a batch that was on delivery to a client. Specimens were marked with a code and tested in different laboratories. Stress-strain curves were obtained by strain-driving of a common compression machine.

A comparative eco-balance has been performed for the two concrete in table 1. The methodology applied in this eco-balance complies with ISO 14’040 and 14’044 standards. All data concerning materials and processes to be analyzed were obtained directly from the ready mix concrete supplier and included the raw materials, the type and distance of transport, the amount of energy used etc. The environmental datasets were taken from the Ecoinvent database version 2.2 [11].

4 TEST RESULTS

Recycled concrete is being actually widely used also in public and private works in Switzerland. Eco-labels as the Minergie, require that at least 50% of the total volume of all concretes used is recycled. This is an “exclusive clause” requirement and must be satisfied unless there is no supplier within 25 km or it is not advisable to use recycled concrete. One of the most used “technical reasons” to avoid the use of recycled concrete is that internal sulfate attack (ISA) might occur. A serious weakness with this argument, however, is that if a concrete is certified, all components must be certified and the certification includes the testing for the percentage of sulfur or water-soluble sulfate (WS) and acid-soluble sulfates (AS). Limits for recycled concrete for WS and AS are respectively 0.2% and 0.8%. Below these levels it is considered that the aggregate will not promote ISA. Of course also the others components must be below the maximum sulfate content. Figure 2 and 3 shows the results for the extraction of water soluble and acid soluble sulfate on specimens taken from large recycled concrete aggregates stockpiles within a period of about 3 years. Sulfate content of acid-extracted sulfate was always much lower than 0.8% as well as the water sulfate was constantly a fraction of the 0.2% permitted by the EN norms.
The delay of the setting time provoked by an extract of water of recycled aggregate was analyzed as well. The maximum delay observed was of about 3.5% which meant 7 minutes delay on a setting time of 205 minutes. The tests were able to show that recycled concrete can be used in structural concrete. However, as for ordinary aggregate, it is necessary to collect representatives’ specimens of the material and regularly test the characteristics. It is not advisable to crush and immediately reuse a lot of concrete as it is not advisable to use supply of aggregates coming from a construction site without testing them. Stress strain-behavior of RC and ordinary concrete of table 1 was tested under uniaxial compression. The results are shown in figure 4. The 28 days strength of both concretes is within the strength class under which these concretes are classified. For these six specimens, the fact that the strength of recycled concrete is slightly higher than ordinary should magnify rather than moderate a fragile behavior.
A detailed eco-balance has been prepared on the concretes of table 1 within the frame of the same research project. Different scenarios including concrete made with natural aggregates supplied from different sources located at different distances from the ready mix plant and the field and different methods of concrete production (in field and in stationary batch plant) were analyzed in the eco-balance. All the machinery necessary in the process and transportation was included. The eco-balance is actually being peer-reviewed. Figure 6 shows the results of one of the scenarios studied: the production and delivery of a volume of 7 m$^3$ which corresponds to the typical quantity of concrete transported by a drum truck. The scenario of truck mixing has not been studied, since truck mixers are rare in Switzerland and the truck serve only to keep the concrete perfect during transportation (the drum is not designed for mixing). Recycled concrete has always lower impact and the rate of impact reduction vary.

A further impact reduction, which is difficult to quantify but self-evident is not included in the eco-balance: all demolished material is a waste of considerable volume. A single plant in the region produces about 18000 m$^3$ of recycled concrete of different types per year. This means that this single plant permits avoiding the disposal of more than 2200 tons of demolished material per year.
5 CONCLUSIONS

Based on the evidences presented in this article, it can be concluded that:

- Recycled concrete might be certified and fits the principles of performance-based prescriptions.
- As soon as a recycled concrete is certified, it can be safely used for structures.
- The use of recycled concrete aggregates does not increase the possibility of ISA occurrence.
- The use of recycled concrete aggregates does not imply the use of larger cements rates if a specific mix design studied.

Further testing is necessary to confirm the stress-strain behavior is not sensibly modified by higher rates of recycled concrete aggregates.

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IMPROVING THE RESILIENCE AND DURABILITY OF NEW ZEALAND’S BUILDINGS

Mark S. Jones (1), Nick J. Marston (1), Patricia Shaw (1) and Zhengwei Li (1)

(1) BRANZ Ltd, Wellington, New Zealand – Mark.Jones@branz.co.nz; Nick.Marston@branz.co.nz; Patricia.Shaw@branz.co.nz; Zhengwei.Li@branz.co.nz

Abstract

Although considerable research has been carried out into the resilience of buildings, materials and components, there are still substantial gaps in knowledge and the information available. Such areas include how these factors are influenced by extreme events, how buildings can be made more resilient and how maintenance can extend the service life of materials and buildings.

Over recent years, New Zealand has experienced a number of extreme events, which have led to increasing costs to the building industry, homeowners, councils, government and the insurance industry. Despite being built in areas of risk, New Zealand’s housing stock has limited resilience, which is further compromised by poor maintenance and repair of our existing buildings. This puts the existing stock even further at risk and there is a strong need for our buildings to be made more resilient to such events.

Building on the development of the BRANZ Durability Verification and Residual Service Life tools, current research is delivering risk profiles and developing guidelines on materials and design that can inform stakeholders about actions that can be taken to improve the resilience of both new and existing dwellings. The research involves robust investigations into the resilience of buildings and structures, taking into consideration the material and building characteristics, property characteristics, external elements, geographical location and combinations of hazards.

1 INTRODUCTION

New Zealand is an island nation on the boundary of the Pacific and Indo-Australian tectonic plates, straddling a mid-latitude zone of strong westerly winds, and as a result is susceptible to a wide range of natural hazards and extreme events. Over recent years the country has experienced a number of these extreme events, which have led to increasing costs to the building industry, homeowners, councils, government and the insurance industry. Earthquakes account for the most significant losses, which is not surprising, given the Canterbury earthquakes in 2010 and 2011 were amongst the most significant natural disaster events in the world at that time. The insurance losses for these events alone is estimated at tens of billions of dollars. Some of the secondary effects of the earthquakes are only just becoming evident, for example, the repeated flooding in certain areas of Canterbury.
Prior to the Canterbury earthquakes in 2010-2011, approximately 94% of adverse event insurance claims in New Zealand were for storms, severe floods, snow and landslides. Storm and flooding damage alone is estimated to have cost the country hundreds of millions of dollars over recent years. To provide a representative history of losses, Figure 1 provides a breakdown of natural hazard losses with the recent Canterbury earthquakes removed.

![Figure 1. Insurance losses from natural hazards in NZ from 1968 – 2012.](image1)

It is a similar situation in Australia, where cyclone, storm and flood damage have had a devastating effect on the country. Figure 2 shows the insurance losses for natural disasters in Australia from 1967 to 2013, based on 2011 normalised dollars [1]. In 2011, extreme weather events, including Cyclone Yasi and the Queensland floods, caused damage to over 4000 homes and 3000 commercial properties, estimated at over $5 billion.

![Figure 2. Insurance losses for natural disasters in Australia from 1967 - 2013 [1](image2)
Throughout the world, the insured losses from climate-related disasters have increased substantially. Estimates demonstrate an increase from approximately US$5 billion to US$27 billion in annual insured losses over the last 40 years [2]. Furthermore, it is estimated that only 25 per cent of the total losses from a natural disaster are borne by the insurance industry with the remainder being absorbed by individuals and governments [3]. Data from the International Disaster Database shows that although earthquakes are low frequency events, they have the highest economic loss. However, taking into consideration the number of occurrences of different types of natural hazard events per year, flood and storm events clearly outweigh other events and this may be an important consideration when focusing on the wider community impacts, rather than economic losses.

Valued at over $600 billion, New Zealand’s housing stock is valuable both socially and economically. The building and housing sector is a key component of the New Zealand economy and is central to New Zealand’s continued growth. Construction activity contributes four to five per cent of gross domestic product (GDP) and the sector employs eight per cent of New Zealand’s workforce [4]. However, despite being built in areas of risk, New Zealand’s housing stock has limited resilience, which may in turn compromise their ability to meet current durability requirements within the New Zealand Building Code (NZBC). Poor maintenance and repair of our existing buildings, as shown within the BRANZ House Condition Surveys [5, 6], also puts the existing stock even further at risk and there is a strong need for our buildings to be made more resilient to such events.

2 BUILDING RESILIENCE RESEARCH

2.1 Resilience Assessment Tools and Methodologies

There are a number of assessment tools and methodologies in use, or under development in different countries across the world. The initial review work has focused on tools and other information that is relevant to any developed country [7]. Although a number of tools have been identified, most research and information has been in the form of guidelines or standards, rather than tools [8-9]. These have generally adopted a systems approach to specifying construction details and types that offer more resilience, usually focusing on only one particular natural hazard such as flood, storm or bushfire. A number of other organisations have funded research investigating resilience and climate change adaptation [10-11]. There are few resilience tools that tend consider the materials that the building components are manufactured from and attempt to take account of a range of natural hazards.

In Australia, the increased frequency and the destructive effects of hazard events has led to the Insurance Council of Australia (ICA) investing in the development of the Building Resilience Rating Tool (BRRT), identifying the need to improve the resilience of Australian residential properties against natural weather hazards. As a result, the ICA and Edge Environment formed the Australian Resilience Taskforce (ART) to develop and implement the BRRT [12]. The BRRT is intended to inform homeowners, local authorities, planners, building professionals and insurers of the resilience of homes to a broad range of hazards, as well as being designed to encourage homeowners, homebuyers, homebuilders and property professionals to adopt improved material selection and design. The BRRT tool aims to measure the resilience of an individual building to a range of extreme weather hazards. The BRRT
calculates the resilience taking into account the hazard profile, location of the house, section details, house type and the individual building materials used in construction.

2.2 BRANZ Resilience Research

BRANZ has recently been working with the Centre for Research Evaluation and Social Assessment (CRESA), to assess resilience of buildings and communities in order to understand the impact of incorporating resilient materials and design on New Zealand’s dwellings [13]. Better understanding of the effects of extreme weather events on materials and buildings, and of building design and materials selection provides the following benefits:

- Information on better design for durability and robustness;
- Better options for mitigation strategies on existing houses;
- Easier and cheaper repair of buildings after an extreme weather event;
- Ability to inform stakeholders of the risks of extreme weather events;
- Understanding of the benefits of maintenance of houses; and
- Understanding of the cost-benefit of different infrastructure and materials choices.

All of these considerations are important to a range of stakeholders including home-owners, designers, insurance industry and regulatory authorities. Assessing and understanding resilience has the potential to improve individual and community outcomes after an extreme natural event.

From the review of the material databases currently available, it is evident that data regarding the durability, environmental performance and safety of materials is very fragmented and there is no one-stop-shop for information on materials resilience. This highlights the need for a robust and transparent database like the BRKD, which would provide consistent information on materials durability, its resilience against natural hazards and its environmental performance.

BRANZ has previously developed a Durability Assessment and Verification Database to provide a more robust durability assessment framework for the New Zealand building industry [14, 15]. The database aggregates existing durability knowledge and verification methods, identifying critical knowledge gaps to guide future research, and provides useful durability information in a convenient manner. This Durability Verification framework has subsequently been extended to include a Residual Service Life Assessment Tool for New Zealand, a broadly-applicable tool guiding the day-to-day decisions of building industry practitioners concerning residual service life of buildings and building elements [16].

As part of the project, BRANZ has been developing a hazard risk rating for the most common building materials and elements found in New Zealand houses. This is based on testing the materials performance when exposed to the effects of extreme natural events. As flooding and storms were the most frequently occurring natural events in New Zealand, and responsible for the largest proportion of insurance losses, the initial focus has been on the effects of water damage.

The resilience of materials and assemblies are being assessed by testing the effects on relevant properties to different hazard exposures under standardised conditions. The results of these tests are used to provide a qualitative assessment of the resistance of different materials to a particular hazard exposure. Initially the performance of individual materials is studied. Once this behaviour is understood, materials are tested in combination, such as those typically found in a building element. For example, the performance of wall insulation and plasterboard is being assessed individually and also when assembled to simulate a wall element. The materials are given relative ratings, as a 1-5 score, where a higher rating indicates a higher
resilience to the specific hazard. This approach was influenced by FEMA’s Technical Bulletin on Flood Damage-Resistant Materials [17].

Initial testing was carried out to assess the effects of fresh water flooding, starting with flooring materials and wall materials. For example, a variety of typical wood-based flooring materials were selected and immersed in water for different times at ambient water temperature and tested in accordance with appropriate standard methodologies, such as AS/NZS 4266.5. Parameters measured included dimensional stability, weight, modulus of rupture (MOR) and modulus of elasticity (MOE). Most loss of strength (decrease in MOR/MOE) occurred after 3 hours of immersion and then levelled off up to 7 days of immersion (Figure 4). After 3 hours immersion, only the tongue & groove (T&G) flooring and plywood still met the selected flooring specification, based on NZS 1860.1:2002, indicated by the red dotted line in the figures.

![Figure 4. MOE for wood-based flooring materials after water immersion for specified times.](image1)

![Figure 5. Recovery of strength (MOE) after immersion and re-drying.](image2)
Additional work demonstrated that all the materials showed good strength recovery on re-drying after short immersion times (Figure 5). This is particularly useful in determining the resilience of materials subjected to short and long term flooding events and assessing whether building components require replacement. The next stage of work was then to attribute relative ratings to the flooring materials, based on their performance in these tests, as shown in Table 2.

Table 2. Indicative Resilience Ratings for Wood-Based Flooring Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>BRANZ Rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particleboard</td>
<td>1</td>
</tr>
<tr>
<td>Strandboard</td>
<td>2</td>
</tr>
<tr>
<td>Plywood</td>
<td>3</td>
</tr>
<tr>
<td>MDF</td>
<td>1</td>
</tr>
<tr>
<td>T&amp;G</td>
<td>4</td>
</tr>
</tbody>
</table>

2.3 Materials and Systems Approaches

A materials approach to resilience considers the durability and performance of individual materials in isolation, usually based on testing and evaluation of small samples. A systems approach considers all the materials present, their interactions, and how they are used in the building. In reality, a successful and useful resilience rating tool will draw on aspects of both approaches. Any resilience tool will always need to consider the relative performance of materials. Some materials will perform better than others when faced with certain events and any tool will need to guide users towards favouring these materials.

However, the development of the rating tool has shown in some cases that assessing single building materials does not give a systems perspective, i.e. the use of several building elements together, and how compatibility and performance may change if just one of these elements is substituted or replaced. Taking a materials approach has the potential to overlook the use of several building elements together. To explore this, it is useful to consider how building elements fail during extreme events. For example, a roof is likely to fail catastrophically through the detachment of the cladding or through the detachment of the roof substructure. These failure mechanisms are mostly independent of the material from which the roof cladding is made. That said, under the first failure mechanism it may be argued that the material from which the roof cladding is made exerts some influence over the failure. If the roof structure fails, then the roof cladding material has had little bearing on the performance of the roof. Therefore, it is unlikely that a reliable assessment of the likely performance of a roof will be made by looking at the roof cladding material in isolation. The cladding fasteners, roof substructure material and roof attachments must also be taken into consideration. Ultimately, the design of the whole roof system is likely to be the key determinant of the overall performance.

Further, the design and complexity of the roof, and the roof geometry, will have a significant bearing on the consequences of failures. For a simple hip roof, any blocking of the gutters could resulting in wetting of the cladding. It is not likely to lead to extensive water penetration into the roof space or to water entering the living space. In contrast, a roof with blocked, near horizontal, valley gutters could more easily have water penetrating the roof space or even the living space.

This leads to the conclusion that a resilience tool driven by collecting information on the individual materials used would still need to be consider system factors. This is because all
consequential impacts would need to be accounted for. This dependency, of the exposure of one material upon the performance of another, is currently involving three sequential phases:

- Firstly, what is the probability of the exposed material failing due to a hazard (e.g. wind lifting the roof cladding, hail breaking tiles, hail clogging gutters).
- Secondly, which of the underlying materials will be exposed to (the same or) any additional hazard(s) (e.g. rain entering a roof space or through a clogged gutter will affect other internal components).
- Thirdly, what is the extent of failure of the underlying material to (the same or) the additional hazard(s) (e.g. once exposed to rain, different components will prove more or less resilient to rainwater).

To implement this in a tool, it would then become necessary to attempt to incorporate the influence of the risk of failure of a given material on all the other materials that are protected by it. This is further complicated by the risk of failure of a given material being the product of the probability of different hazards attaining different levels of severity and the probability of failure of the actual material when exposed to that severity of hazard. Simply, using a materials based approach to resilience assessment is extremely complex and the likelihood of a key consequential impact on the performance of the building being omitted is high.

Examples of some the difficulties associated with a materials approach for resilience assessment include:

- Materials are often difficult to tell apart once incorporated into a building – hard to tell if what was specified or reported was actually used,
- Performance of a specific material relies on consistency of manufacture and supply
- Performance of a material often depends on installation
- Ignores quality of construction
- Ignores maintenance

3 CONCLUSIONS

By assessing the frequency of natural hazards and losses in New Zealand, it confirms the common beliefs on key natural hazard risks in New Zealand. Key findings from analysis and tool development include:

1. Earthquakes exceed all other hazards in New Zealand in terms of financial loss, having the potential to cause catastrophic damage and costly, but are reasonably well dealt with through building regulations.
2. Flooding is New Zealand’s most frequent hazard, followed by storm
3. New Zealand has a similar hazard profile to Australia allowing similar scoping and development of resilience rating tools.

From the current study, it is concluded that a holistic assessment methodology and assessment tool are needed to assess the resilience of typical building element junctions as well as the resilience of the building elements and construction materials. This approach is also most likely to allow translation of short term tests to the behaviour of a real building. Ultimately, a more robust assessment methodology will be in place that evaluates the resilience and cost
benefit of different infrastructure, building and material choices that can drive an increase in resilience and protection. Further development of assessment methodologies tools and solutions to evaluate the impact of adverse events on the durability and resilience of materials and buildings will better inform key stakeholders.

ACKNOWLEDGEMENTS

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REFERENCES

[1] Insurance Council of Australia, Historical Disaster Statistics


RE-ASSESSMENT OF NEW ZEALAND’S ATMOSPHERIC CORROSIVITY

Nick J. Marston (1), Zhengwei Li (1), Mark S. Jones (1) and Patricia Shaw (1)

(1) BRANZ Ltd, Wellington, New Zealand - Nick.Marston@branz.co.nz; Zhengwei.Li@branz.co.nz; Mark.Jones@branz.co.nz; Patricia.Shaw@branz.co.nz

Abstract

Atmospheric corrosivity maps are fundamental to the specification and selection of materials for buildings in different geographic regions to ensure that they meet the durability requirement of the performance based New Zealand Building Code. Recently, atmospheric corrosion testing has been conducted at thirty-six sites across New Zealand. The corrosion rates of mild steel and hot dip galvanised zinc coated steel coupons were measured after one year of exposure starting in late 2011. These were compared with results obtained in past BRANZ surveying from the 1980s. At a limited number of sites, obvious changes in mass loss rates were found for mild steel or for zinc. The mechanisms behind these changes have been examined, taking account of any changes in the New Zealand climate over the past three decades. No strong correlation has been found between the changes in individual climatic factors and the latest metal corrosion rate. Analysis of current data implies that the atmospheric corrosivity map shown in the key New Zealand Standard, NZS3604, might have limitations in some regions. Current research is looking further into zone boundary adjustments and to develop a reliable, updated atmospheric corrosivity map for cost-effective building and construction across New Zealand.

1 INTRODUCTION

Atmospheric corrosion accounts for the highest metal loss among all forms of corrosion and therefore is a major contributor (>50%) to overall corrosion induced costs for most developed countries [1]. In New Zealand, corrosion related costs are estimated to be of the order of 2.5% of GDP [2].

Metallic claddings, fasteners and decorative components are widely used in the building and construction sector which is a key component of the New Zealand economy and contributes 4-5% of GDP. The nation’s housing stock is valued at approximately $600 billion [3]. Building components must meet or exceed the performance requirements of the New Zealand Building Code (NZBC) for use within any specified geographic environments whose corrosivity is defined by atmospheric corrosivity maps.

The atmospheric corrosivity map currently used in the New Zealand Standard, NZS3604: Timber-framed buildings [4], was established using experimentally collected metal corrosion rates by BRANZ in the 1980s [5-6]. Due to the fundamental role of this map in building design and materials specification, the on-going credibility of the data set must be maintained.
In addition, it was felt that sufficient time might have passed for the atmospheric corrosion behaviour of the metal, to reflect potential changes in climate.

2 EXPERIMENTAL

Mild steel and hot dip galvanised zinc coated steel coupons were used in this research (Table 1). The mild steel coupons were grit blasted to SA3 grade while the zinc coated steel samples were chemically cleaned prior to exposure. The dimensions of each coupon were measured. They were then cleaned, dried, weighed and sealed in plastic bags.

Table 1: Chemical composition of metals used in the present study (wt.%)  

<table>
<thead>
<tr>
<th>Metal</th>
<th>Element</th>
<th>C</th>
<th>N</th>
<th>Al</th>
<th>Si</th>
<th>P</th>
<th>S</th>
<th>Ti</th>
<th>V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mild steel (~150×100×3 mm)</td>
<td></td>
<td>0.18</td>
<td>0.003</td>
<td>0.007</td>
<td>0.04</td>
<td>0.027</td>
<td>0.014</td>
<td>0.005</td>
<td>0.002</td>
</tr>
<tr>
<td>Cr</td>
<td>0.03</td>
<td>0.69</td>
<td>0.02</td>
<td>0.03</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>Bal.</td>
<td></td>
</tr>
<tr>
<td>Hot dip galvanised zinc coating (~150×100×0.5 mm)</td>
<td>Mg</td>
<td>&lt;0.01</td>
<td>0.32</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>0.10</td>
<td>Bal.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Steel samples were mounted onto exposure racks located mainly within Auckland, Wellington and Dunedin regions during September and October 2011. The racks were placed approximately 1.8 m above the ground facing north at a 45° angle. After one year, these samples were retrieved and morphologically characterised. Corrosion products were cleaned by chemical etching recommended by ASTM G1 [7] as follows:

- Mild steel: 0.5 L/L hydrochloric acid (HCl, specific gravity = 1.19) + 3.5 g/L hexamethylenetetramine (C$_6$H$_{12}$N$_4$) at 20-25°C; and

- Hot dip galvanised zinc coating: 100 g/L ammonium chloride (NH$_4$Cl) at 70°C.

Before immersion in the cleaning solution, the loosely attached corrosion products on the mild steel samples were removed using a wooden spatula under flowing water. The solutions were agitated magnetically to make the cleaning period as short as possible. The cleaned and dried samples were then weighed again to obtain mass losses which were corrected by chemical etching of unexposed, reference samples of mild steel and hot dip galvanised steel.

Climate data, including ambient temperature, rainfall, wet days (defined as daily precipitation > 1 mm), relative humidity (RH) and wind speed, were retrieved from the National Climate Database (NIWA). Their changing trends in the period of 1980-2010 were analysed using linear regression fitting. Comparisons between 1987-1988 and 2011-2012 were also made.

3 RESULTS

3.1 Corrosion rate

The first-year metal corrosion rates, derived from current research, were compared, where possible, with those obtained in the 1980s by BRANZ.
At Auckland Airport, the first-year corrosion rate was significantly increased for mild steel from 298 to 491 g/m²/year (65% increase) and for zinc from 13 to 33.3 g/m²/year (156% increase). At Greymouth, the first-year mild steel corrosion rate decreased 33% from 511 to 342 g/m²/year while the corrosion rate of zinc appeared to be unchanged. At Tiwai Point (southern end of the south island), the mild steel corrosion rate slightly decreased while the zinc corrosion rate decreased markedly from 15.4 to 4.7 g/m²/year. At other exposure sites, changes in corrosion rate, both decreases and increases, were observed, but the extent was not as significant as that of the above sites. It was also commonly found that the changing trends of mild steel and zinc were not always the same at the same site and this almost certainly could be explained by their different corrosion mechanisms.

3.2 Change in climatic factor

From a global perspective, the most direct consequence of climate change is an increase in ambient temperature. Twelve of the fourteen exposure sites, where reasonably complete data can be obtained, showed an increase in annual air temperature of ~0.01-0.03°C per year in the past three decades. RH trends were very complicated, but it is quite clear that significant changes have not occurred. Most sites had a decreasing trend of annual rainfall and wet days, but a difference could be found at some exposure sites for these two factors. In other words, a higher rainfall does not always mean a larger number of wet days. Annual mean wind speed was generally decreasing during the past 30 years at most exposure sites.

4 DISCUSSION

4.1 Correlation between atmospheric corrosivity and climate

Atmospheric corrosion is governed by the presence and chemistry of a thin aqueous layer on the metal surface. Several climatic factors could affect this layer, directly or indirectly.

4.1.1 Ambient temperature

The ambient temperature can affect the metal surface temperature, which directly influences time of wetness (ToW), drying rate of surface electrolyte layer, and hence the duration of chemical and/or electrochemical reactions on the metal surface [8]. In New Zealand, the ambient temperature increase in the last 30 years was slight and no large increase occurred at most exposure sites between the periods of 1987-1988 and 2011-2012. A comparison of previous and present metal corrosion rates revealed no simple changing trend, indicating that the influences of ambient temperature cannot be considered alone. An ambient temperature increase would be expected to increase the metal surface temperature, leading to accelerated corrosive attack on the metal if RH of the surrounding environment was kept constant. However, an increase of temperature will generally lead to a decrease of RH and also ToW. Some researchers thus believe that the climate change induced warming is unlikely to have a significant effect on corrosion of most materials, even though they might have a positive temperature coefficient of corrosion rate. An increase of 2°C was estimated to increase corrosion rate by only ~0.6% [9-10].

4.1.2 Relative humidity

Analysis of present results showed that the changing trend of RH, to some extent, had a correlation with the mild steel corrosion rate change. At Ardmore and Auckland airports, the
Changes in RH and corrosion were similar. At Dunedin Airport, Levin and Wallaceville, RH exhibited a small change while the mild steel corrosion rate appeared to be unchanged. That said, at Greymouth and Paraparaumu, the mild steel corrosion rate obviously decreased while the RH only showed a small change.

A lower RH could lower the fraction of the metal surface wetted and hence a smaller area of metal surface would be attacked. This would tend to decrease the overall corrosion rate. However it should be realised that various gaseous pollutants and/or solid particles would participate in atmospheric corrosion processes. Most sites used in the present study were, more or less, influenced by marine atmospheres. Consequently, concentration and pH of salts dissolved in the surface moisture layer were playing a more important role. A decrease of RH together with a decreased fraction of surface wetted would probably favour a higher local corrosion rate, albeit over a relatively short time scale. Further, changes in environmental RH may induce changes in the size and surface deposition of airborne salt particles. Atmospheric corrosion may not initiate on steel surfaces covered with deliquescent NaCl particles having diameters less than ~45 µm since small droplets may contain oxygen above the critical level necessary to passivate the steel surface [11]. Therefore, RH changes will affect the nature, morphology and severity of corrosion attack with the involvement of chloride-containing particles though the exact mechanism was not clearly understood.

4.1.3 Rainfall

A clear correlation between long/short term trend of rainfall and metal corrosion rate change could not be derived with confidence in the present study (Figure 1). At Auckland Airport, an increase was observed for both rainfall and corrosion rate. At Dunedin Airport, Invercargill, Levin and Wallaceville, a reasonably stable trend for both rainfall and corrosion rate was found. However, at Ardmore, Greymouth and Paraparaumu, the first-year mild steel corrosion rate decreased obviously in the period of 2011-2012 while the rainfall did not show any obvious changes when compared with the period of 1987-1988.
The annual rainfall appeared to have a correlation with the atmospheric corrosion rate of mild steel at some sites. For example, annual rainfalls for Dunedin Airport, Ardmore and Greymouth were around 700, 1000 and 2000 mm, while the corrosion rates were measured to be 127, 209 and 511 g/m²/year for the period 1987-1988 and 130, 166 and 342 g/m²/year for the period 2011-2012, respectively. Nevertheless, at any one exposure site, an increase of the annual rainfall did not always correlate to a higher corrosion rate.

Rain will maintain the environmental humidity above the critical humidity for atmospheric corrosion. If rainwater was retained in pockets or crevices of the corrosion product layer, it may promote corrosion by supplying continued wetness in these localised areas [12-13]. Regular washing of the sample surface could also partially remove corrosion products, exposing fresh metal to further corrosive attack. On the other hand, rain may benefit corrosion resistance. Atmospheric corrosion has been observed to be more severe on surfaces that were not periodically washed by rain [14]. This effect is particularly noticeable in marine environments [15]. However, if the amount of rainfall is extremely limited, pollutants deposited may only redistribute on the metal surface, but their total load will remain the same. Experimental and theoretical work indicates that the amount of rainfall required for surface cleaning to begin is between 0.8 and 1.2 mm, while 1.3-3.7 mm of rain were required to totally clean a surface. Pollutants were reduced to 10% of their original concentrations after a rainfall of 1.5 mm [13]. However, this does not necessarily explain why the skyward surface of the hot dip galvanised zinc coating had more severe corrosion than the groundward surface when exposed at Auckland Airport.

The quantity of rainfall during the initial stage of atmospheric exposure was found to affect metal corrosion significantly. Several sample sets were exposed at BRANZ’s Judgeford campus during the periods of 19 March – 19 April, 19 June – 19 July and 19 September – 19 October 2013. The first-month mild steel corrosion rates were measured to be 206, 960 and 596 g/m²/year, respectively. The rainfall records in these three periods were 65.9, 314.9 and 176.6 mm, respectively. Higher rainfall corresponded to a higher corrosion rate and this could be explained by: (1) higher humidity and longer ToW, and (2) higher salt concentration in rain, particularly under storm conditions (20-23 June; rainfall ≈ 186 mm). However, it is not clear how high rainfall and fast corrosion in the initial stage will affect corrosion over longer periods. Studies are on-going at BRANZ to achieve a better understanding of this.

4.1.4 Wind speed

Wind is another climatic factor that should be considered in atmospheric corrosion [16-17]. Models incorporating wind speed are known to better predict experimental results [18].

In the present study, when a decrease in wind speed was observed, a decrease in the first-year mild steel corrosion rate could be expected. This is particularly true for Invercargill, Paraparaumu and Tiwai Point. At Levin, a slight increase of wind speed was observed together with an almost unchanged corrosion rate. These results suggest that wind speed might be related to mild steel corrosion rate in some New Zealand atmospheric environments. At Auckland Airport, Invercargill, Paraparaumu and Tiwai Point, the averaged wind speed normally ranged from 4 to 6 m/s and the wind speed decrease was around 1 m/s in the short period comparisons (1987-1988 versus 2011-2012). Relatively obvious changes in mild steel corrosion rate were observed at these sites. At Dunedin Airport, the wind speed was normally less than 4 m/s and had decreased ~1 m/s in the short period comparisons. No obvious change in metal corrosion rate was found. A critical wind speed, around 5 m/s, appeared to exist and
influence atmospheric corrosion. In other studies, a critical velocity, close to 3 m/s (or larger than 3-5 m/s), seemed to exist for saline winds. Above this, coastal atmospheric salinity notably increased and a considerable effect on atmospheric corrosion could be observed [19-20]. In order for corrosion to develop, salt aerosol must impinge onto a metal surface and this can only happen when the wind speed is above a certain minimum [21].

4.2 Potential Impacts on the Current Atmospheric Corrosivity Map

Based on the mild steel corrosion rates obtained during 2011-2012, the atmospheric corrosivity zone boundaries defined by the NZS3604 map were checked. It should be realised that this is supported by atmospheric corrosion tests that are separated by ~25 years.

![Diagram](image)

Figure 2: Zone boundary definition in NZS 3604 atmospheric corrosivity map with first-year mild steel corrosion rates obtained in current research

[Zone B (ISO 9223 C2) ■, Zone C (ISO 9223 C3) □, Zone D (ISO 9223 C4) □]

4.2.1 Auckland

In the NZS3604 atmospheric corrosivity map, the northern area was classified into Zone C (ISO 9223 C3) and the western coastal areas were classified into Zone D (Figure 2a, ISO 9223 C4). The current testing in the central area supported this zone definition. In the southern region, corrosion rates obtained at Ardmore, Hunua and Tuakau ranged from 139 to 166 g/m²/year. Geographically, the Hunua Ranges at the east of Hunua and Ardmore is covered by forest and vegetation. This may partly block the salt-laden wind from the east coast, leading to a recession of the boundary of Zone C to the east. Similarly, when the prevailing wind from the south-west direction is considered, the south end of Zone C could be moved north slightly. Testing is being carried out in Drury, Karaka, Paerata and Pukekohe to confirm these hypotheses.

4.2.2 Wellington

Wellington city is completely classified as Zone C by the NZS3604 atmospheric corrosivity map (Figure 2b). Current measurements indicated that the atmospheric corrosivity in the city region might be slightly higher than expected, possibly due to the strong prevailing wind from the northwest direction and slightly weaker prevailing wind from the north and south directions. Previous studies at Oteranga Bay (southwest coast) found mild steel corrosion rates of ~1600 g/m²/year [22-23] that could be comfortably classified into corrosion category E (ISO 9223 C5) or higher. Salt from the rough ocean might be brought a long way inland by the strong winds in Wellington region.
4.2.3 Otago

The present study recruited several sites located within areas classified as Zone C by the current map (Figure 2c). Mild steel corrosion rates ranged from 120 to 151 g/m$^2$/year and are within the range of Zone B (ISO 9223 C2: 10-200 g/m$^2$/year). Further, corrosion rates of zinc coatings at these sites were measured to be from 0.6 to 3.6 g/m$^2$/year and are also within the range of Zone B (0.7-5 g/m$^2$/year). The mild steel coupons, after one year of exposure, showed a mass loss of 250 and 301 g/m$^2$ at Highcliff and Taiaroa Head of the Otago Peninsula, respectively. These numbers are within the range of Zone C. The corrosion rates of zinc at these two sites, 5.7 and 5.0 g/m$^2$/year, can be classified into Zone C as well. These results imply that the corrosivity of the atmosphere over the Otago Peninsula was higher than that of the urban area. However, classification into Zone D might not be appropriate.

5 CONCLUSION

Based on experimental metal atmospheric corrosion rate measurements and climatic data analysis, the following conclusions can be drawn:

- At most exposure sites, the annual ambient temperature increased slightly, meanwhile relative humidity and wind speed decreased slightly. The variations in rainfall and wet days were complex and no uniform trend was found.
- Obvious changes in atmospheric corrosion rates were noticed at several sites, however the new corrosion rates were still comparable to the previous ones at most sites.
- No strong relationship between the changes in an individual climatic factor and metal atmospheric corrosion rate was revealed. In consequence, the potential effects of the variations in New Zealand climate over the past thirty years on atmospheric corrosion remain unclear. However, significant changes and strong effects have been absent over the last 30 years.
- Based mainly on the first-year mild steel corrosion rates, the current map in NZS3604 was found to have some areas where the atmospheric corrosivity might not be accurately defined.

Research is continuing to: (1) complete the only experimentally derived atmospheric corrosion rate data set available in New Zealand, (2) produce reliable data for areas where new building activities will be concentrated in the short and medium terms, and (3) establish a map with accurately defined corrosion zone boundaries for New Zealand. These outputs will contribute to the identification of options for adaptation through cross-disciplinary assessments of New Zealand buildings’ vulnerability to the environment.

6 ACKNOWLEDGEMENTS

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7 REFERENCES


COMPARING THE INFLUENCE OF FLY ASH ON THE DURABILITY PROPERTIES OF PORTLAND CEMENT-BASED AND ALKALI ACTIVATED SLAG-BASED CONCRETES

John L. Provis(1,2), Susan A. Bernal(1,2), Idawati Ismail(2,3), Rackel San Nicolas(2), David G. Brice(2,4), and Jannie S.J. van Deventer(2,4)

(1) Department of Materials Science and Engineering, University of Sheffield, Sheffield S1 3JD, United Kingdom – j.provis@sheffield.ac.uk
(2) Department of Chemical & Biomolecular Engineering, University of Melbourne, Victoria 3010, Australia
(3) Faculty of Engineering, Universiti Malaysia Sarawak, Kota Samarahan 93400 Sarawak, Malaysia
(4) Zeobond Pty Ltd, P.O. Box 23450, Docklands, Victoria 8012, Australia

Abstract
Low calcium fly ash is widely used as a pozzolan in the production of Portland-blended cements and concretes, and also in production of alkali-activated materials, either as sole precursor or blended into slag-based concretes. Significant chemical and structural changes are induced through the addition of fly ash, as a low-calcium aluminosilicate source, to more calcium-rich binders. However, there has not previously been a detailed comparison of the role of fly ash in blending with each of these two types of calcium-rich cementing systems. Although appearing superficially similar in chemistry and gel nanostructure, the very different alkali levels present in the two types of binders have important implications for the reactivity of fly ash in each instance. This study reports the mechanical strength development, water absorption properties, and chloride permeability of Portland cement-based and alkali-activated slag-based concretes with 25 wt.% fly ash inclusion in the binders, with mix designs tailored to give comparable 90-day strengths of 60±3 MPa. The incorporation of fly ash into Portland cement-based concrete is seen in general to enhance later-age properties, while the effects of its inclusion in alkali-activated slag concretes seem in several cases to be reversed. The reasons for this are related to the fact that there is not sufficient excess calcium in an alkali-activated slag to induce a pozzolanic-type reaction, and so the ongoing alkali-induced reaction of fly ash in an alkali-activated slag binder instead results in an alkali aluminosilicate gel which does not show the same influence on transport properties.

1 INTRODUCTION
In the last decades there has been a growth in awareness of the need to develop construction materials that can meet not just the mechanical strength and workability requirements of civil infrastructure construction, but which can also stand the test of time over an extended service life. Durability has thus become a key criterion for the selection and design of construction materials, as design to optimise full service life cost and environmental footprint - including construction, repair, rehabilitation and retrofitting - is being implemented in modern civil engineering practice.
Blending of cements or concretes with supplementary cementitious materials has become increasingly common in engineering practice in many parts of the world, including almost ubiquitous use of blended cements in some parts of the world such as the European Union. The influence of fly ash blending on the properties of Portland cement concretes has been studied for more than 75 years [1], and its role in the physical and chemical evolution of the Portland-cement based binder is now in general well understood [2-4].

Alkali-activated binders are now being commercialised and deployed as a lower-CO$_2$ alternative to ordinary Portland cement (OPC) in general applications in many parts of the world [5]. Several of the systems in commercial production or in scale-up around the world are based on blast furnace slag to achieve higher early strength at lower activator doses, but the incorporation of fly ash into these binders also brings advantages in terms of rheology and workability, as well as cost in areas such as Australia where slag can be much more expensive than fly ash. For this reason, it is of interest to determine whether the existing body of knowledge regarding incorporation of fly ash into Portland cement-based binders and concretes is also applicable in the analysis of fly ash blending into alkali-activated slag-rich systems, or whether the differences in chemistry and chemical reaction mechanisms between these types of materials lead to different effects upon blending with fly ash.

Therefore, this paper presents the results of physical and transport characterisation of concretes based on OPC and alkali-activated slag (AAS), each blended with the same quantity of the same fly ash. The materials are analysed for porosity and chloride permeability, and conclusions are drawn regarding the different effects of fly ash in each system.

2 EXPERIMENTAL METHODS

2.1 Materials

A fly ash classified as Class F according to ASTM C618-12 [6], from Bayswater Power Station, New South Wales, Australia, and a granulated blast furnace slag (GBFS) supplied by Zeobond Pty Ltd, Australia, were used as solid precursors for the preparation of the mortars and concretes. The GBFS had a specific gravity of 2800 kg/m$^3$ and a d$_{50}$ of 15 μm, compared to 2200 kg/m$^3$ and 12 μm respectively for the fly ash. Reference samples were produced using an ordinary Portland cement (OPC), type GP according to AS 3972-2010 [7] (Eureka Cement, Australia). The chemical compositions of these precursors are shown in Table 1.

<table>
<thead>
<tr>
<th>Precursor</th>
<th>Component (mass % as oxide)</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SiO$_2$</td>
<td>Al$_2$O$_3$</td>
<td>Fe$_2$O$_3$</td>
<td>CaO</td>
<td>MgO</td>
<td>SO$_3$</td>
<td>Na$_2$O</td>
<td>K$_2$O</td>
<td>LOI</td>
</tr>
<tr>
<td>Fly ash</td>
<td>63.9</td>
<td>25.3</td>
<td>5.3</td>
<td>&lt;0.1</td>
<td>1.0</td>
<td>0.2</td>
<td>0.2</td>
<td>1.3</td>
<td>2.7</td>
</tr>
<tr>
<td>Slag</td>
<td>34.2</td>
<td>13.8</td>
<td>0.4</td>
<td>43.1</td>
<td>5.4</td>
<td>0.8</td>
<td>0.1</td>
<td>0.4</td>
<td>1.8</td>
</tr>
<tr>
<td>OPC</td>
<td>20.3</td>
<td>4.5</td>
<td>4.6</td>
<td>62.9</td>
<td>1.2</td>
<td>2.6</td>
<td>0.3</td>
<td>0.3</td>
<td>3.3</td>
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</tbody>
</table>

Commercial sodium metasilicate granules with a chemical composition of 50.5 wt% Na$_2$O and 46.0 wt% SiO$_2$ (balance H$_2$O) were dissolved in tap water and cooled for two hours before use as the activating solution.
2.2 Concrete Preparation

The matrix formulations used for producing the concretes are given in Table 2. All the concretes were dosed with water to binder ratios (w/b) selected to meet target strengths of 60 ± 3 MPa after 90 days of curing. It is important to note that, even though the most usual age for strength and durability-based design is 28 days, we have selected 90 days instead, considering that the most significant effects of SCMs in the mechanical and durability properties of blended cements are identified at ages beyond 28 days. Therefore, 90 days seemed a more suitable age for making a fair comparison of well-developed concretes with different chemistries.

Concrete samples were mixed in an electric pan mixer following the Australian Standard AS 1012 [8], according to the mix designs in Table 3. Crushed gravel and sand were used as coarse and fine aggregates in the manufacture of concretes. The coarse aggregate was of 20 mm maximum size, with a specific gravity of 2830 kg/m$^3$ and water absorption of 1.10%. The specific gravity and water absorption of the sand are 2550 kg/m$^3$ and 0.90% respectively.

Table 2: Concrete binder types assessed

<table>
<thead>
<tr>
<th>Type of concrete</th>
<th>Binder proportion (wt. %)</th>
<th>Activator concentration (wt. % binder)</th>
<th>Water/binder</th>
<th>Paste volume fraction (m$^3$/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Slag</td>
<td>Fly ash</td>
<td>OPC</td>
<td></td>
</tr>
<tr>
<td>Alkali-activated</td>
<td>100</td>
<td>0</td>
<td>0</td>
<td>8</td>
</tr>
<tr>
<td>Portland cement</td>
<td>75</td>
<td>25</td>
<td>0</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>0</td>
<td>0</td>
<td>100</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>0</td>
<td>25</td>
<td>75</td>
<td>-</td>
</tr>
</tbody>
</table>

*0.5% PCE superplasticiser was added to this mix to enable the target 90-day strength to be reached with acceptable workability

Table 3: Concrete mix designs. All quantities specified in kg/m$^3$.

<table>
<thead>
<tr>
<th>Binder proportion (wt. %)</th>
<th>Total binder</th>
<th>Coarse aggregate</th>
<th>Fine aggregate</th>
<th>Activator</th>
<th>Water content</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Slag</td>
<td>Fly ash</td>
<td>OPC</td>
<td></td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>400</td>
<td>1150</td>
</tr>
<tr>
<td>75</td>
<td>25</td>
<td>0</td>
<td>0</td>
<td>400</td>
<td>1150</td>
</tr>
<tr>
<td>0</td>
<td>0</td>
<td>100</td>
<td>0</td>
<td>360</td>
<td>1150</td>
</tr>
<tr>
<td>0</td>
<td>25</td>
<td>75</td>
<td>360</td>
<td>1150</td>
<td>640</td>
</tr>
</tbody>
</table>

The concretes were cast in 100 mm diameter × 200 mm height cylinder moulds, and vibrated until suitably consolidated. The concretes were then cured in a water bath at ambient temperature (20-23°C) until testing. Additional data characterising some of these concretes, and also related paste and mortar specimens for the alkali-activated systems studied here, are available in references [9, 10].

2.3 Tests conducted

2.3.1 Compressive strength and water absorption properties

An ELE ADR Auto 1500 compression testing instrument was used to determine the compressive strengths of concrete cylinders after 28 and 90 days of curing, following the
standard procedure ASTM C39-10 [11]. Concrete specimens were loaded at a rate of 2.4 kN/sec until failure. The water absorption properties, specifically the volume of permeable voids (VPV) and capillary sorptivity, were determined for specimens with 28 and 90 days of curing. VPV was assessed according to the standard test ASTM C642-06 [12]. All concrete specimens were pre-conditioned by drying at 100°C until reaching constant weight, as specified by the standard. Capillary sorptivity was conducted following the procedure described by Fagerlund [13]. It is worth noting that the pre-conditioning used is severe, and can induce microstructural changes and microcracking, but the method has been adopted in this study to facilitate the direct comparison of Portland and alkali-activated concretes. Two replicate sets of measurements (4 specimens) were used for each test and each time of curing.

2.3.2 Chloride permeability

Penetration of chlorides into the specimens was assessed according the NordTest method NT Build 492 [14] using a 3.5 wt.% NaCl solution, 50 mm thick concrete slices after 28 and 90 days of curing, with colorimetric analysis applied at the end of the test and results analysed according to the equations specified in the test protocol. Silver nitrate (AgNO₃) was applied to freshly split samples to determine the chloride penetration depth. Silver nitrate reacts with both chloride ions and hydroxyl ions to form white AgCl and dark brown Ag₂O respectively. Chloride penetration depth is measured as the visible boundary between white precipitation of silver chloride (AgCl) where the chloride ion is present in sufficient quantities, and precipitation of brown silver oxide (Ag₂O) with OH⁻ in pore solution otherwise [15].

3 RESULTS AND DISCUSSION

3.1 Compressive strength development

Figure 1 shows the compressive strength development of the different mixes tested. All mixes reached the target strength of 60 ± 3 MPa at 90 days; the OPC-only mix was actually the slowest to reach this target strength, and showed the lowest early strength development, due to the much higher water/binder ratio required for this mix to match the 90-day strengths of the other concretes. The incorporation of 25% fly ash in the AAS concrete had very little effect on the strength at 28 days and at longer ages, at similar water/binder ratios.

This is a striking contrast to the performance of the OPC systems, where the incorporation of fly ash necessitated the use of a much lower water content, and the addition of a superplasticiser to achieve the required workability at this water content, for a comparable 90-day strength. The difference in reactivity between OPC and fly ash is much greater than the difference in reactivity between slag and fly ash, and the more aggressive chemical conditions in the alkali-activated binder systems triggered the dissolution of fly ash much earlier than is the case in OPC-based blended binders, where the induction period of the fly ash reaction is related to the extended time taken to raise the pH to a sufficient level through Portland cement hydration [3].
3.2 Water absorption properties

Figure 2 shows the differences in water absorption as measured by the ASTM C642 boiling test for each mix design at different ages. Although the differences between samples fall within the analytical uncertainty, it is interesting to note that the addition of fly ash to the OPC-based blends seems to increase the measured volume of permeable voids at all ages, even with the much lower water content of this mix. For the alkali-activated binder systems, the slag-only concrete has a lower VPV at early age, but this is reversed at later age, as the fly ash is evidently reacting during this time period, as also seen from the fact that the strength development profiles of these two concretes are very similar in Figure 1. From microtomography data for paste specimens [16], the pore structure evolution and degree of space-filling of the gels which develop in alkali-activated binders based on 100% slag or 25% fly ash/75% slag are very similar, and the elemental distributions within the gel systems are also largely similar between these mixes [10]. A similar situation is expected in OPC concretes, where a larger content of binding phases is expected to form with the progress of the hydration reaction. There is not a consistent trend as a function of time in the VPV data of the specimens studied, which may be attributed to the harsh pre-drying conditions of the test causing damage to the samples [9, 17].

The capillary sorptivity data for the samples tested are presented in Figure 3. Both fly ash-containing systems, and the alkali-activated slag-only concrete, show higher total capillary water uptake at 90 days than at 28, indicating that the increased total gel volume in these materials which show ongoing reaction of the aluminosilicate components is a key factor influencing these measurements. This is most dramatic in the AAS system, where the gel volume (as evidenced by total porosity, because the gel is the main porous component in the concretes) increases from 28 to 90 days as a result of ongoing chemical reaction processes. Conversely, the OPC binder shows a reduced, and slower, water uptake in the more mature binder system, indicating that the main effect of the increased maturity here is gel densification rather than an increase in total gel content. The reduced water/binder ratio of concretes containing fly ash (see Table 2), can also be a factor reducing the sorptivity of these materials.
Figure 2. Volume of permeable voids of fly ash blended OPC and AAS concretes. Error bars represent one standard deviation among 4 replicate samples.

Figure 3. Capillary sorptivity curves of plain and fly ash-blended OPC and AAS concretes. Error bars correspond to one standard deviation of four measurements.

It is also potentially of interest that both fly ash-containing systems show a faster initial water uptake at 28 days than at 90, while the converse is true for the two systems without fly ash. The initial rate of water uptake is related to pore radius, and so the indication is that the
presence of fly ash causes pore refinement in both systems, in parallel with production of a higher gel volume produced in the OPC/FA system at 90 days compared to 28 days.

3.3 Chloride permeability

Table 3 shows the differences in chloride migration coefficients as a function of mix design in the concretes studied. The difference in these coefficients between systems dominated purely by the hydration products of OPC (OPC-only at 28 and 90 days, and OPC/FA at 28 days), and those in which aluminosilicate components have played a significant role in the reaction (OPC/FA at 90 days, and all of the alkali-activated systems) is particularly striking, with a factor of 10 lower chloride diffusion in the systems in which Al supplied by slag or fly ash is playing a significant role in the gel chemistry. It is well known that the inclusion of SCMs in concrete increases its electrical resistivity, via changes in the composition of the pore solution and pore structure [18]. Therefore the contribution of the aluminosilicate component to chloride penetration resistance, and the resistivity changes induced in the material, require further attention and analysis, but this trend is consistent across a much wider range of slag/fly ash ratios in addition to the ratio studied here [9].

### Table 3: NordTest chloride diffusion coefficient of fly ash blended OPC and AAS concretes

<table>
<thead>
<tr>
<th>Type of concrete</th>
<th>Chloride diffusion coefficient (×10⁻¹² m²/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>28 days</td>
</tr>
<tr>
<td>OPC</td>
<td>18.6</td>
</tr>
<tr>
<td>OPC/FA</td>
<td>10.2</td>
</tr>
<tr>
<td>AAS</td>
<td>0.4</td>
</tr>
<tr>
<td>AAS/FA</td>
<td>1.0</td>
</tr>
</tbody>
</table>

4 IMPLICATIONS FOR THE USE OF FLY ASH IN BLENDED BINDERS

The difference between the reaction of fly ash in OPC-based binders and in AAS-based binders is fundamentally a result of the differences in chemical conditions prevailing within the binders at early age. At the start of the reaction of a slag-fly ash alkali-activated binder, the slag reacts with the silica from the activating solution, precipitating it as C-A-S-H and leaving the fly ash to react with a very alkali hydroxide-rich pore solution in the partially reacted paste. This results in a binder in which the extent of reaction of the fly ash is relatively high, and because it has started to react quite early, the fly ash-derived reaction products are able to be integrated into the developing C-A-S-H gel. Conversely, the reaction of fly ash with OPC starts much later in the reaction process, when the cement has eventually generated a sufficiently high pH, as seen from the chloride diffusion coefficient data in Table 3 where the fly ash has barely started to have an effect after 28 days. This will leave more isolated regions of fly ash-derived gel which will not be fully integrated with the bulk C-S-H binder.

5 CONCLUSIONS

When considering the blending of fly ash with more calcium-rich binder systems to form composite cements or concretes, the differences in the pH evolution of the liquid phase within the binder must be considered when predicting when, and how, the fly ash will react. This alters the properties of the material, for example the addition of fly ash to OPC concretes reduces the chloride diffusion coefficient, whereas the converse is true for fly ash addition.
into an alkali-activated slag concrete. Capillary sorptivity is mainly a function of gel volume, in both types of concrete. There are subtle but important differences in the reaction mechanism of fly ash in OPC-based and AAS-based binder systems, and these must be taken into consideration in the design of durable high-quality concretes based on these materials.

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REFERENCES
ASSESSMENT OF THE HYDRATION CONSTANTS OF POST BLENDED BLAST FURNACE SLAG CEMENTS

Jeanette Visser (1), Quirijn van Zon (1) Hans Beijersbergen (1), Anke Hacquebord (1) and Ron van Zwet (1)

(1) TNO, P.O. Box 49, 2600 AA Delft, jeanette.visser@tno.nl; Quirijn.vanzon@tno.nl; Hans.Beijersbergen@tno.nl; anke.haquebord@tno.nl; ron.vanzwet.tno.nl

Abstract

The amount of bound water in OPC / slag blends have been measured for three different temperature regimes: from 105 to 950 °C, 400 to 600 °C and 600 to 950 °C for three different OPCs and one type of slag, at 5 different replacement factor and at four ages. Based on the measured linear relationship between the amount of bound water with slag replacement, the effect of the replacement could be eliminated and the effect of the OPC and slag effect on the water contents could be separated. Time effects were next eliminated by modelling the results with help of an hydration model on the basis of the observed exponential increase in water contents with time. The resulting hydration constants were obtained for the total nonevaporable water content showing different values from 0.19 to 0.22 for the three different OPCs correlating well with other data from literature. A constant value of 0.06 was found for the slag. For the bound water in the calcium hydroxide, constants were approximately 0.07 for the OPC cements and 0.02 for the slag. For the Type I CSH or meta-jennite the constant was 0.06 to 0.1 for the OCPs and zero for the slag.

1 INTRODUCTION

1.1 Background

Slag is a waste product that can be blended with Portland cement to serve as binder for concrete. Blending reduces the use of raw materials and energy and leads to a strong reduction in CO$_2$. These so-called post blended slag cements allow for a more flexible use of slag than manufactured blast furnace slag cement (in short called preblended). However, post blending is only allowed as long as the performance of the concrete is maintained. The amount of replacement that can be accommodated while maintaining the performance depends on the composition of the slag and the Portland cement, but the underlying mechanisms that control the performance in relation to replacement and composition are unknown. To be able to optimize the slag content, these mechanisms need to be quantified.

The quantification has become all the more urgent since the boundaries in the EU are open for any Portland cements with CE-marking. This has resulted in a much wider variation in OPC-compositions than previously encountered, due to a high variation in raw materials and fuel. When these Portland cements are blended with the same slag, concrete was found to have sometimes an unforeseen, much lower performance. This results in applications with
less added value (i.e. lower strength classes or lower clinker replacement) and in a reduction of the market share for blast furnace slag as clinker replacement.

The aim of this research is to develop the fundamental knowledge of the reactivity of the ground granulated blast furnace slag in presence of Portland cement, required to develop a model to predict and influence the performance of the blend in concrete.

1.2 Approach

The most important performance demand for concrete is usually related to strength. In general, a minimum compressive strength is required. The strength depends on, among other, the way stresses are distributed in a material. Since empty spaces cannot transfer stress, local stresses thus are higher in materials with more empty spaces at a certain applied force. This results in a lower strength. With this, the strength depends on two factors: the initial porosity, depending among others on the initial water-to-cement ratio but also strongly on the particle packing in the material. Secondly, it depends on the way the pore space is filled during hydration of the OPC clinker and the slag. To separate the problems of pore space filling due to cement and slag hydration from geometrical problems such as particle packing, the gel/space ratio theorem of Powers and Brownyard [1] can be employed. Powers and Brownyard found that the compressive strength increases with the formation of hydrate products, and attributed this to the pore space filling. The idea behind the strength increase with pore space filling is, simply put, that with the clinker hydration, part of the space will be occupied by the reaction products. These have a larger volume than the original material, mostly as a consequence of the water-uptake in their structure. They fill up the pore space and thus increase the strength.

The relation between pore space filling and degree of hydration is well established for OPC. The amount of nonevaporable water content (defined here as the water that disappears between 105 and 950 °C) can be used as a measure of the amount of hydrates. In addition, the volume increase of the hydrates of OPC are also quite well established. A simple, unique relation therefor exists between the degree of hydration of OPC and the amount of nonevaporable water as measured between 950 and 105 °C:

\[ w_t = w_{950-105} = n_c \alpha_c \]  

In which \( w_t \) is the total amount of evaporable water content (on binder) which is defined as the difference in weight \( w \) at 950 °C and 105 °C and \( n_c \) is the hydration constant, that expressed the amount of bound water (or rather mass increase) in the hydration products while \( \alpha_c \) is the degree of hydration. Often, an average value of 0.23 for OPC is used, more specific values (for different OPC cements) can be found among others in Powers & Brownyard [1].

The hydrate products for blended cements of OPC and slag are less well known (see e.g. [2], [3], [4]). In addition, it is not clear if and how much water is chemically and physically bounded by the slag (hydrates) or blended hydrate products [4], although some number s are given by [5]. Wang et al [4] state for instance an amount of chemically bound water of 0.3 g / g reacted slag and CH amount of 0.22 g / g reacted slag. These average number however are insufficient to determine the effect of e.g. different OPCs on the compressive strength of various blends. In addition, these numbers seem a priori quite high for the materials as known in the Netherlands: blends with slags are known generate hardly any calcium hydroxide nor...
do they bind as much water as pure OPCs. Therefore, as a first step, it has been investigated if a similar relationship as in eq. (1) exists for the blended cements.

Since the total amount of evaporable water for blended materials depends on two types of hydrations, namely that of the clinkers and the slag. Therefore, an initial model is defined for the total non-evaporable water content as:

\[ w_T \equiv w_{950-105} = n_c(1 - RP)\alpha_c + n_sRP\alpha_s \]

(2)

in which RP is the Replacement Factor of OPC by slag and subscripts c and s denotes cement and slag respectively. However, there are now three unknowns in the equations: both degrees of hydrations and the nonevaporable water constant \( n_s \). In addition, it cannot be assumed a priori that nonevaporable water constant \( n_c \) will remain the same upon blending, and thus also has to be treated as an unknown. To reduce the amount of unknown, a more general expression will be used:

\[ w_T \equiv w_{950-105} = (1 - RP)w_c + RPw_s \]

(3)

which \( w_c \) now is defined as the amount of nonevaporable water due of the OPC alone and \( w_s \) at the amount of nonevaporables water due to the effect of the slag. Eq. (3) is solvable for both unknowns \( w_s \) and \( w_c \), provided the results show a linear relationships (otherwise the model is not correct). In addition to the total amount of nonevaporable water, other thermographic data can be collected, on account of the different thermographic ranges as established for cement-based materials:

- \(< 105\): decomposition of ettringite and the weakest bonded hydrate water from some of the CSHs, Al- ettringite
- \(105 - 400\): decomposition of many of the CSH phases, Fe-ettringite and gypsum
- \(400 - 600\): decomposition of Portlandite, including that formed from the decomposition of hydrogarnet phases
- \(800 – 950\): decomposition of the CSH-types (meta-)jennite and CSH I

This interpretation is based on the research of Pane and Hansen [6], Nijland and Larbi [7] and the CRC handbook [8]. Different interpretations however exists (see e.g. [9],[10],[11]). In this paper, two more results will be presented, namely those of the 400-600 and 600 – 950 °C range. In terms of slag replacement the weight losses van be expressed similarly as in eq. (3) but for different temperature ranges and thus expressing different results. Although there is some discussion about the interpretation of the thermographic data, the validity of the relationships do not depend on these interpretation. Nevertheless, for simplification of the discussion in the remainder of this paper, they will be defined as such.

In this paper, the experimental programme, results and models will be discussed for the thermographic data only. The hydration constants derived for the OPC and blended slag/OPC will be presented and discussed. They can be used in numerical hydration models which so far hampered from lack of reliable constants to describe the hydration of the slag. This paper hopefully contributes to a much better prediction and gains insight in this respect.

2 EXPERIMENTS

2.1 Materials

In order to investigate the strength development and formation of hydration products upon post blending slag with various types of (Ordinary Portland) cement (OPC), three types of
cement (OPC) of various fineness and composition have been used in the research: CEM I 52.5 R, CEM I 52.5 N, CEM I 42.5 R from different producers. These have been tested pure and at various replacement percentages: 35 %, 50 %, 72% and 85%. These represent post blended CEM II/S-B, CEM III/A, CEM III/B and CEM III/C. For these 15 types of cement / slag blends, mortar bars were prepared according to the NEN-EN 197-1. All specimens were made by first dry blending the slag and the cement. Further mixing was performed according to the standards. Mortars were all made in the standard reference mortar composition: ratio of sand, binder (in total) and water was 3 : 1 : 0.5 by mass.

2.2 Experiments

The development of the strength is determined by means of the uniaxial compression tests according to NEN-EN 196-1. For each mortar composition, specimens (40 x 40 x 160 mm in size) have been cast according to this standard. After one day, they have been demoulded and stored in a climate room at 20 °C and 96% RH. At an age of 1, 3, 7 and 28 days, one specimen has been subjected to a three point bending tests, after which either half has been subjected to uniaxial compression. After the compression tests, one half of the prisms is placed in the 40 °C oven for drying prior to thin section fabrication for the SEM/ PFM-analysis (not reported in this paper), the other half is placed in the 105 °C oven under (nitrogen) conditions, to prevent carbonation. The specimens have been oven dried at 105 °C until equilibrium and weighted. Further drying until equilibrium have next been performed at 400, 600 and 950 °C. In order to assess the reliability of the measurements, one set of samples was directly dried after equilibrium in 105 °C at 950 °C. The non-evaporable water content is calculated by means of:

\[
\omega_t = \frac{\omega_n}{w_b} = 4 \cdot \frac{M_{105} - M_{950}^c}{M_{950}^c}
\]

in which \(M_{950}\) and \(M_{105}\) are the weight of the sample at 105 °C and 950 °C and in which the weights at 950 °C are corrected for the loss of ignition (LOI) of the cement and slag at this temperature. All weights are expressed relative to the total binder weight \(w_b\), which is exactly equal to one fourth of the mortar dried at 950 °C and corrected for the LOI, on account of the large volumes used in the thermic measurements and the original mortar compositions (1 binder : 3 sand, on mass). All results in the paper are further reported as amount of mass change in its corresponding temperature range by:

\[
\omega_{(T1-T2)} = \frac{M_{T1} - M_{T2}}{w_b}
\]

The weight losses are presented relative to the binder mass (cement and slag).

3 RESULTS

As an example of the results, the results of the gravimetric experiments are shown in Figure 1 for the 400 – 600 °C range and the 600-950 °C range for one OPC, pure or blended with slag, at four different ages. As can be seen, to first order, the relationships are linear. For each slag/binder ratio versus nonevaporable weight loss, a least square analysis has been made according to eq.(3). The resulting \(w_s\) and \(w_c\) are shown in Figure 2.
Figure 1: Mass loss at 600 °C as compared to 400 °C (left) and 950 °C as compared to 600 °C (right) for CEM I 42.5 R at various slag/binder ratios and at 1, 3, 7 and 28 days.

Figure 2 Calculated \( w_c \) and \( w_s \) at 600 °C - 400 °C range (left) and 950 – 600 °C range (right) as a function of age for CEM I 42.5 R

4 INTERPRETATION AND DISCUSSION

Figure 2 shows that the variables \( w_c \) and \( w_s \) are time dependent. This will be due to the fact that they are dependent on the degree of hydration (viz. eq. (2) to eq. (3)). The time dependency of the degree of hydration can be modelled well by means of its exponential behavior: \( \exp \), as the hydration is fast at the start and slows down with age to such an extent that it slowly goes to a limit (normally if there is enough space to full hydration).

Including the appropriate limits, the hydration for the OPC can be modelled as:

\[
\omega_c(t) = w_{c,inf} (1 - \frac{w_{c,inf} - w_{c,0}}{w_{c,inf}} \exp(-\alpha_c t)) \equiv w_{c,inf}(\alpha_c)
\]

Upon blending the OPC with the slag, however, additional hydration may occur for OPC in excess to dilution. The additional hydration is among other due to so-called heterogeneous nucleation: the OPC is known to hydrate faster in the presence of more ‘foreign’ particles (in this case the slag) [12]. Because this heterogeneous effect is likely to be proportional with the slag replacement, its effect will not be incorporated in the above expression.

It is postulated that a similar expression as for the hydration of the OPC can be written for the ‘slag’ effect in which the latter contains all effects of the presence of the slag, including...
the slag hydration and such effects as heterogeneous nucleation, and any other effect that may be present:
\[ w_s(t) = w_{s,inf} \left( 1 - \frac{w_{s,inf} - w_{s,0}}{w_{s,inf}} \exp(\frac{-\alpha_s t}{\tau_s}) \right) \equiv w_{s,inf} \alpha_s(t) \] (7)

If it is an invalid approach, this will follows from the least square analysis. Substitution of the results in eq. (1) gives:
\[ w_n(t) = (1 - RP)w_{c,inf} \alpha_c(t) + RPw_{s,inf} \alpha_s(t) \] (8)

Least square analysis of CEM I 42.5 R are shown in Figure 3 as an example for the temperature range of 950 – 105 °C only. Similar analysis have been made for the 400-600 and 600-950 °C ranges. The most salient feature of the LSQR results is with respect to \( w_{inf} \), which are shown in Table 1.

![Figure 3 Data (symbols) and best fitted model for the nonevaporable water variables \( w_c \) and \( w_s \) for CEM I 42.5 R/slag (replacement factor independent) at the temperature range of 400-600 °C (left) and 600 – 950 °C (right)](image)

Table 1: Results of the least square modelling of the degree of hydration, results for \( w_{c,inf} \) and \( w_{s,inf} \) with \( w_{inf} \) coding: \( n = \) total non-evaporable water constant for \( T=950-105 \) °C, \( c = \) the nonevaporable water constant for \( T=600-400 \) °C and \( p = \) nonevaporable water constant for \( T=950-600 \) °C; results applicable up to a replacement of 85%.

<table>
<thead>
<tr>
<th>( w_{inf} ) code</th>
<th>( T ) (°C)</th>
<th>cement c</th>
<th>slak (and additional effects) s</th>
</tr>
</thead>
<tbody>
<tr>
<td>( n )</td>
<td>950-105</td>
<td>0.22 - 0.19 - 0.20</td>
<td>0.06</td>
</tr>
<tr>
<td>( c )</td>
<td>400-600</td>
<td>0.07 - 0.08 - 0.06</td>
<td>0.02</td>
</tr>
<tr>
<td>( p )</td>
<td>600-950</td>
<td>0.10 - 0.06 - 0.10</td>
<td>0.00</td>
</tr>
</tbody>
</table>

The results in Table 1 show that the nonevaporable water constant \( n \) differs slightly for the different OPC cements but is constant for the slag. All constants of the cement are denoting exactly the amount of bound water at a certain temperature range in the OPC as if no slag is added (non-dilution values). For instance: for the full temperature range 105 -950 °C, the best fit values \( w_{c,inf} \) denoted as \( n_c \), is thus equal to the (average) amount of water that is bound in CEM I 52.5 R, CEM I 52.5 N and CEM I 42.5 R. For the three different cements they are 0.22, 0.19 and 0.20 respectively. This is well within the range of reported values of Powers and
Brownyard [1] who found among other in the wcr range of 0.4 to 0.5 for mortar, values between 0.2 and 0.22 for three different OPCs.

The additional effect due to the presence of the slag, \(n_s\) (\(w_{s,inf}\) at \(t=950-1050^\circ C\)) is fairly constant at about 0.06. As said, this effect probably includes both the effect of heterogeneous nucleation (high hydration of the Portland cement in the presence of more foreign particles) and the hydration of the slag itself. This effect then can be seen to also increase proportionally with the slag content, i.e. the replacement factor. Unfortunately, no distinction can be made between the heterogeneous nucleation effect and the slag hydration effect from these data but surprisingly, these are independent of the OPC cement used.

In addition, the table shows for the investigate OPC and slag combination, an average calcium hydroxide constant \(c_c\) of about 0.07 for the OPC and \(c_s\) of 0.02 for the effect of the slag. Literature on the calcium hydroxide content in cement/slag-based systems is confusing. Calculating the expected amount on the basis of the major clinker minerals (see e.g. [2],[4]), results for the pure OPC at a total water binding of 0.23 in a calcium hydroxide constant of about 0.1. The result depend much on the exact fractions of the clinkers as the amount of calcium hydroxide is largest for the clinker mineral \(C_3S\) (1.5), followed by \(C_4S\) (0.5) while it decrease for the pozzolan clinker minerals \(C_3A\) and \(C_4AF\). The results of the modeling, being close to 0.07, seem therefor reasonable.

For the slag, it would have been expected that for truly latent-hydraulic behaviour, the calcium hydroxide constant would be zero. This is, as already mentioned, not the case: the calcium carbonation constant varies is about 0.02. Some calcium hydroxide might be formed since the slag has a CaO-content of 35%. On the other hand, this may not be enough in relation to the consumption of Ca in the CSH. In the latter case, the water loss might be due to the constant decomposition of CSH at various temperature. Hydrotalcite-like slag hydration products cannot account for the non-evaporable water in the 400-600 °C range, as their dehydroxylation occurs at 250-300 °C, and loss of interstitial water already at lower temperatures ([13], [14]).

An unexpected results is found in the temperature range from 600 to 950 °C. The infinite values for the slag \(w_{s,inf}\) are approximately zero, whereas for the OPC they are not. Also the CEM I 52.5N has a lower value (0.060) compared to the two rapid OPC-cements (0.100). The results might be correlated to the decomposition of the CSH-types (meta-)jennite and CSH I between 800 – 950 °C occurs, which are usually only reported for pure OPCs ([7]).

If the above explanation is true, the following relationships can be postulated to hold true for the results in this research: Using the following code for \(w_{c,inf}\) and \(w_{s,inf}\): \(n = \) total non-evaporable water constant for \(T=950-105^\circ C\), \(c = \) the nonevaporable water constant for \(T=600-400^\circ C\) and \(p = \) nonevaporable water constant for \(T=950-600^\circ C\);

\[
w_t \equiv w_{950-105} = n_c(1 - RP)\alpha_c + n_sRP\alpha_s
\]

\[
w_{Ca(OH)2} \equiv w_{600-400} = c_c(1 - RP)\alpha_c + c_sRP\alpha_s
\]

\[
w_{CSH(I)} \equiv w_{950-600} = p_c(1 - RP)\alpha_c
\]

with the constants \(n, c\) and \(p\) as given in Table 1 and results applicable for a replacement factor \(RP \leq 0.85\).
5 CONCLUSIONS

Although no distinction could be made between the addition effects of the OPC upon dilution (such as heterogeneous nucleation) and the hydration of the slag, the hydration of the OPC/slag blend proved to be linearly related to the slag replacement. Eliminating first the effect of the slag replacement on the non-evaporable water content and then the age (time effect), the hydration constant for both the cement and slag could be obtained for the total amount of hydrates (between 950 – 105 °C), for the calcium hydroxide content (between 600 - 400 °C) and meta-jennite (CSH I, between 950-600 °C).

The hydration of the slag and OPC could not be fully separated since additional effect of the slag on the cement increases with slag replacement as well. In addition, interpretation of the hydration constants is based on the broad temperature ranges that are not completely defined by just one type of hydrate, e.g. Ca(OH)$_2$. Nevertheless, the hydration constants, together with the models derived, will make it possible to predict the degree of hydration with replacement and age (time). In the net paper, these results will be coupled with the compressive strength, in the next step towards prediction of the compressive strength of OPC /slag blends.

6 REFERENCES

HYGROTHERMAL CONSEQUENCES OF APPLYING EXTERIOR AIR BARRIER SYSTEMS IN HIGHLY INSULATED WOOD-FRAME WALLS: A PARAMETER ANALYSIS.

J. Langmans, Evy Vereecken, Staf Roels

Building Physics section, KU Leuven, Heverelee – jelle.langmans@bwk.kuleuven.be

Abstract

This paper studies the durability of timber frame wall configurations with exterior air barrier systems in various European climates. First, the hygrothermal impact of different insulation materials will be discussed for Belgium climate conditions. The simulation results demonstrate that the application of mineral wool insulated timber frame walls in combination with exterior air barriers results in increased moisture loads. Moreover, unavoidable (small) air channels between the mineral wool and the adjacent exterior air barrier significantly increase natural convection and add up to harmful moisture levels. Yet the use of cellulose insulation can avoid these issues. The second part of the paper studies the performance of these wall elements for 12 European climates. The simulation results indicate that the technique of exterior air barrier is more suitable for continental climates rather than for cold and moderate sea climates in Europe.

1 INTRODUCTION

The execution of exterior air barrier systems in timber frame construction has gained increasing interest in the aim to meet the severe airtightness requirements of Passive houses ($n_{50} < 0.6$ ACH). In practice, fewer joints and perforations of exterior air barriers are an advantage compared to the traditional interior air barrier systems [1]. Also in Norway the practical advantages of exterior air barriers are mentioned [2,3]. Yet for cold and moderate climates, such as the Northern European countries, the hygrothermal impact of exterior air barriers remains unstudied. Consequently, the execution of wall systems with an exterior air barrier only is currently not recommended for these climate types.

The present paper performs numerical heat, air and moisture (HAM)-simulations on lightweight walls with exterior air barrier systems to explore the risks involved. Yearly simulations under realistic climate conditions have been conducted with an adjusted version of DELPHIN 5, capable to model forced and natural convection in interaction with detailed heat and moisture transport [4,5]. The model applies a two-domain approach; (1) porous building materials with (2) adjacent air channels assuming fully-developed laminar flow. The applied model is extensively documented and evaluated with experimental results in [5].
The current article performs numerical simulations to investigate the hygric response of exterior air barrier systems in timber frame construction in a European climate. In a first step, interest lies in the impact of different insulation materials. Thereafter, simulations are conducted for various European climate conditions. The transient simulations were conducted for yearly climate conditions and the mould index (M) based on the VTT-model [6] and the accumulated interstitial condensation (AC) were applied to assess the durability of these timber frame elements. The mould index M (-) expresses the presence of surface mould growth. The mould index corresponds to a discrete number from 0 (no mould growth) to 6 (fully covered). The threshold mould index (M_max) applied in the present study is 3. The maximum accumulated condensation level (AC_max) allowed in the current analysis was assumed to be 0.1 kg/m². The following section will discuss the used wall configurations and climate conditions.

2 WALL CONFIGURATION AND CLIMATE CONDITIONS

2.1 Wall configuration

The simulations throughout this paper are restricted to wall elements only. Figure 1 shows the general configuration which is in accordance with the current building practice of highly insulated timber frame construction in Belgium. Herein, the ventilated cavity of 25 mm contains top and bottom ventilation openings. The material properties of the cladding system are based on [7]; the wooden cladding layer is modelled according to [8] and its exterior paint layer has a sd-value of 0.1 m. The wooden top and bottom plates have a thickness of 40 mm and extend the full width of the insulation layer (30 cm). A service cavity of 40 mm is applied at the inside of the wall element, which is covered with a gypsum board finishing layer. It is important to note that both the interior vapour retarding layer (OSB) and the gypsum board layer are not continuously airtight, so, they will not act as an interior air barrier. Slots of 1 cm are left at both 20 cm from the top and bottom of the wall. The air barrier, however, is positioned at the outer side of the insulation layer by sealing the joints in the exterior sheathing layer.

Figure 1.: Applied wall configuration and simulation grid.
The material properties of the applied materials are summarised in Table 1. Herein, a bituminous impregnated wood fibreboard (BIFB), a spunbonded foil (FOIL), oriented strand board (OSB), mineral wool of 20 kg/m³ (MW20) and cellulose insulation of 60 kg/m³ (CL60) are included. In addition, the simulations apply non-standard mineral wool with a density of 30 kg/m³ and cellulose insulation of 40 and 50 kg/m³. Yet these materials are excluded from Table 1 for brevity but can be found in [5]. The properties of wood are adopted from [8].

<table>
<thead>
<tr>
<th></th>
<th>BIFB</th>
<th>FOIL</th>
<th>OSB</th>
<th>MW20</th>
<th>CL60</th>
</tr>
</thead>
<tbody>
<tr>
<td>d (mm)</td>
<td>18</td>
<td>0.2</td>
<td>15</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td>ρ (kg/m³)</td>
<td>285</td>
<td>-</td>
<td>630</td>
<td>21.3</td>
<td>60</td>
</tr>
<tr>
<td>c_p (J/(kgK))</td>
<td>2068</td>
<td>-</td>
<td>1880</td>
<td>840</td>
<td>2544</td>
</tr>
<tr>
<td>λ (W/m/K)</td>
<td>0.045</td>
<td>-</td>
<td>0.1</td>
<td>0.031</td>
<td>0.034</td>
</tr>
<tr>
<td>K⊥ (m²)</td>
<td>4E-14</td>
<td>airtight</td>
<td>8E-15</td>
<td>1.30E-09</td>
<td>8.8E-10</td>
</tr>
<tr>
<td>K∥ (m²)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>3.80E-09</td>
<td>8.8E-10</td>
</tr>
<tr>
<td>sd₉₀ (m)</td>
<td>0.1</td>
<td>0.05</td>
<td>0.3</td>
<td>0.3</td>
<td>0.6</td>
</tr>
<tr>
<td>sd₅₀ (m)</td>
<td>0.23</td>
<td>0.1</td>
<td>4.2</td>
<td>0.3</td>
<td>0.6</td>
</tr>
</tbody>
</table>

Table 1: limited overview of applied material properties (d (thickness), ρ (density), c_p (heat capacity), λ (thermal conductivity), K (air permeability) and s_d (equivalent vapour diffusion).

2.2Climate conditions

The walls are oriented to the North and the applied climatic data is retrieved from the software package Meteonorm (TRY). The air pressure difference across the wall is a combination of (1) stack pressure, (2) wind pressure and (3) mechanical ventilation. For the simulations at hand, the stack pressure is calculated for a two-storey building, assuming that the main leakages are located near the foundation, wind pressures are calculated based on surface-averaged pressure in which the wind pressure coefficients are adopted from [9] and a 5 Pa overpressure is assigned for mechanical ventilation. The inner temperature is assumed constant (20°C) throughout the year. Yet the inner humidity conditions are determined by a single zone model:

\[
\frac{V}{T_iRV} \frac{\partial P_{vi}}{\partial t} = (P_{ve} - P_{vi}) \frac{nV}{3600R_pT_i} + G_{vp}
\]  

in which V (m³) corresponds to the volume of the room and HIR* (kg/m³%RV) to its hygric inertia, n (1/h) is the ventilation rate and G_{vp}(kg/s) is the vapour production in the room. The used parameters for the single zone model are chosen rather conservative: a small volume of 50 m³ with a high moisture load of two active persons (120 gram/h) between 8 h-22 h. The nominal ventilation rate in living spaces is 3.6 m³/m²/h according to the Belgium standard, corresponding to a ventilation rate of 1.5 l/h for this room. Several studies, however, indicated that the actual ventilation rate is often much lower in reality. Yet to include the effect of a realistic ventilation system in operation, the nominal ventilation rate was reduced to 0.5 l/h. Finally, a value of 1.5 gram/m³%RV was chosen for the hygric inertia.
3 SIMULATION RESULTS

3.1 Insulation material

First, the impact of the insulation material is investigated. Two commonly applied groups of insulation materials have been selected for the numerical investigation: (1) mineral wool and (2) cellulose insulation. For mineral wool two standard densities (20-30 kg/m$^3$) have been included in the investigation. For inflated loose-fill cellulose insulation, however, densities of around 60 kg/m$^3$, commonly applied in practice, have been adopted here. In addition, cellulose insulations with lower densities (40 kg/m$^3$ and 50 kg/m$^3$) have been examined as well. Table 2 presents the maximum mould index and accumulated condensate on the exterior air barrier (BIFB) for the reference configuration (Figure 1) with the discussed insulation materials. The predicted maximum mould index never exceeds the proposed limit of 3 and no condensation occurred for this type of insulation for the wall elements insulated with cellulose. For the mineral wool insulation, however, mould growth problems are predicted for the standard density of 20 kg/m$^3$. A mould index of 4.3 was found for this density, corresponding to a visual mould covering percentage between 10-50%. Mineral wool with increased density (30 kg/m$^3$), in contrast, meets the proposed moisture limit state. Finally, this table shows that no condensation on the BIFB exterior air barrier occurred for both the mineral wool and cellulose.

<table>
<thead>
<tr>
<th></th>
<th>Cellulose</th>
<th>Mineral wool</th>
</tr>
</thead>
<tbody>
<tr>
<td>ρ (kg/m$^3$)</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>$M_{\text{max}}$ (-)/ $AC_{\text{max}}$ (kg/m²)</td>
<td>2.3/-</td>
<td>1.8/-</td>
</tr>
</tbody>
</table>

Table 2: Maximum mould index ($M_{\text{max}}$(-)) and accumulated condensate ($AC_{\text{max}}$ (kg/m²)).

The results of Table 2 correspond to an ‘ideal’ installation of the insulation layer, in that, perfect contact between the insulation and the adjacent layers is assumed. On the contrary, several experimental studies proved the importance of small discontinuities along the interface of mineral wool insulation layers [10]. Such small air cavities are responsible for increased levels of natural convection and consequently higher moisture loads. As a result the presence of small air cavities around mineral wool insulation should be considered. Therefore, the simulations with mineral wool insulated elements were repeated, introducing imperfections along the interface for the insulation and the adjacent layers. Herein, five configurations have been considered, varying the position of these air cavities: an air channel along the (a) cold side of the insulation, (b) on top of the insulation layer (c) both cold and warm sides, (d) both the cold and warm side and at the top of the insulation, and (e) at all four sides of the insulation. For these configurations the impact of channels of 1 mm, 3 mm and 5 mm have been studied. All combinations have been simulated for both mineral wool with a density of 20 kg/m$^3$ and 30 kg/m$^3$ for which the corresponding simulation results are summarised in Table 3. This table confirms that the risk for moisture problems is highly influenced by the presence of small air channels along the mineral wool layer. Even a small air channel of 1 mm at both sides of the insulation increases the mould index from 2.3 to 3.7
for MW30. As a consequence, the above-mentioned conclusion that higher mineral wool densities decrease the risk for moisture problems only holds for perfect contact conditions. From the moment imperfections around the mineral wool insulation layer (which are unavoidable in practice) are considered, the density of the mineral wool becomes of minor importance. In addition, Table 3 shows that most critical moisture limit state again corresponds to mould growth.

<table>
<thead>
<tr>
<th>Density of mineral wool (kg/m³)</th>
<th>Position of air channel</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td></td>
</tr>
</tbody>
</table>

Table 3: Impact of air cavities (1, 3 or 5 mm) around insulation layer on the maximum mould index (-) and maximum accumulated condensation (kg/m²) for mineral wool.

Figure 2 depicts the hourly condensation along the height of the exterior air barrier for MW20 with air channels of 5 mm at both sides. This graph demonstrates that condensation clearly occurs during winter periods and is concentrated at the upper height of this layer, indicating the importance of natural convection within the component.

<table>
<thead>
<tr>
<th>Max mould index (-) / Max accumulated condensation (kg/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1 mm)</td>
</tr>
<tr>
<td>(3 mm)</td>
</tr>
<tr>
<td>(5 mm)</td>
</tr>
</tbody>
</table>

3.2 Different climates

Finally, the hygrothermal response of wall elements with an exterior air barrier have been studied for various European climate conditions. The selected locations cover a wide range of different climates from South to North Europe. Main focus was on the latter and particularly on Scandinavian regions. Recent building practice in Norway tends to focus on increasing the airtightness of the exterior layer, as stated by e.g. [2,3]. Therefore, special
attention was given to this country by including Stavanger, Oslo, Bergen and Trondheim in the analysis. Figure 3 shows an overview of all locations studied. In addition, this figure compares the selected climates by means of averaged climate indicators.

First, the averaged outdoor winter temperatures ($\theta_{\text{winter}}$), provides a direct indication of the potential for buoyancy driven air flow within the component, and thus, the increased moisture loads. Second, the $\Pi_y$ -factor expresses the severity of the exterior climate. This factor (kg/m³), proposed by [11] for the selection of reference years, calculates the yearly averaged difference between the vapour concentration in the outdoor air and the maximum moisture concentration on the building envelope surface on hourly data:

$$
\Pi_y = \frac{v_{out,sat}(\theta_s)}{v_{out}} \bigg|_{year} 
$$

in which $v_{out,sat}(\theta_s)$ represents the saturation vapour concentration at the buildings surface for a North oriented wall and $v_{out}$ corresponds to the moisture concentration of the outdoor air. As this value is calculated on a yearly basis, it represents the drying potential of the climate, and is thus, a relevant indicator for the severity of the climate for the purpose of this study. Both the averaged outdoor winter temperatures ($\theta_{\text{winter}}$) and the $\Pi_y$-factor of the climates investigated are depicted in Figure 3. This graph illustrates how the Southern European climate, Lisbon, combines a high averaged winter temperature with a high $\Pi_y$-factor. Furthermore, this figure clearly shows higher $\Pi_y$-factors for Eastern European climates (Berlin, Vienna) compared to the Western European locations (Brussels, Nantes, Dublin). In contrast the Western European climates tested correspond to milder winter temperatures. Most critical locations expected are Helsinki, Stockholm and Trondheim, in that they combine low winter temperatures with low $\Pi_y$-factors. Furthermore, this graph illustrates great differences between the four Norwegian climates included.

The simulations have been performed for the wall configuration discussed in section 2.1 for which either MW30 with two vertical channels of 3mm on both sides or CL60 is applied. For the exterior air barrier material, on the other hand, two variants are included: (a) BIFB and (b) FOIL. Figure 4 depicts the simulation results for the wall elements insulated with mineral wool. Here, the maximum mould growth index on the walls using BIFB exterior air barriers is

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Figure 3: Overview of the European locations for which simulations are performed.
indicated in black and the maximum accumulated condensation amounts for the elements with a FOIL are given in red. These two graphs contain the same information, however, the left hand side is given as a function of $\theta_{\text{winter}}$ and the right hand side is presented as a function of $\Pi_y$. Out of the 12 climates tested only the Eastern European (Berlin, Vienna), the Southern European (Lisbon) and two of the Northern European locations (Oslo, Bergen) result in mould growth levels lower than 3. From this figure it follows that $\Pi_y$ is the most dominating climate parameter in the prediction of mould growth. For these limited number of climates it appears that no problematic mould growth is observed for $\Pi_y$-factors higher than 2 kg/m³. When it comes to condensation amounts on the non-hygroscopic foil, however, it appears that the temperature is the dominating climate parameter. Here only the coldest climates result in excessive condensation levels (Stockholm, Helsinki, Oslo and Trondheim).

In contrast to the mineral wool insulated walls, no moisture problems occured for the wall elements simulated with loose-fill cellulose insulation. Because no condensation was found for these configurations, the highest moisture contents of the cellulose insulation have been given here (in red). The highest moisture content of the cellulose insulation correspond to Stockholm and Helsinki.

In summary, this section explored the hygrothermal response of highly insulated wall elements with an exterior air barrier in various European climates. In total 12 climates have been verified. From this data it followed that Northern and Western Europe were most vulnerable to moisture problems when such wall systems were insulated with mineral wool. When these walls were insulated with loose-fill cellulose insulation, however, no moisture problems were obtained for the European climates tested.

Figure 4: Top: maximum mould index on BIFB and accumulated condensation on foil as a function of $\theta_{\text{winter}}$ (left) and $\Pi_y$ (right) for MW30 insulated elements, bottom: maximum mould index on BIFB and maximum moisture content of cellulose insulation as a function of $\theta_{\text{winter}}$ (left) and $\Pi_y$ (right) for CL60 insulated elements.

In contrast to the mineral wool insulated walls, no moisture problems occured for the wall elements simulated with loose-fill cellulose insulation. Because no condensation was found for these configurations, the highest moisture contents of the cellulose insulation have been given here (in red). The highest moisture content of the cellulose insulation correspond to Stockholm and Helsinki.

In summary, this section explored the hygrothermal response of highly insulated wall elements with an exterior air barrier in various European climates. In total 12 climates have been verified. From this data it followed that Northern and Western Europe were most vulnerable to moisture problems when such wall systems were insulated with mineral wool. When these walls were insulated with loose-fill cellulose insulation, however, no moisture problems were obtained for the European climates tested.
4 CONCLUSIONS

This paper investigated the hygric response of a timber frame walls with an exterior air barrier in a Belgian climate. The simulation results identified a large impact of the applied insulation material on the hygrothermal performance of this component. No moisture problems were found for the walls insulated with loose-fill cellulose insulation. For mineral wool (glass wool) insulated walls, however, excessive mould growth levels were obtained on the upper parts of the exterior air barrier as a result of natural convection within the insulation layer. Furthermore, the simulations emphasised the importance of small air channels along the mineral wool and the adjacent layers. Such limited deficiencies increased the risk for mould growth and harmful condensation amounts against the exterior air barrier. The second part of the paper verified the performance of highly insulated walls with an exterior air barrier in 12 European climates. From the limited amount of locations tested, it followed that Northern and Western Europe regions were most vulnerable to mould problems. No mould problems were found for the (drier) continental climates, such as Berlin and Vienna. For interstitial condensation on spunbonded foils, however, the decisive climate parameter appeared to be the winter temperatures. Excessive amounts of interstitial condensation were found for the coldest climates, such as Helsinki, Oslo, Trondheim and Stockholm.

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REFERENCES

THE DURABILITY OF BASALT FIBRES REINFORCED POLYMER (BFRP)

Giuseppe Alaimo(1), Antonino Valenza(2), Daniele Enea(3), Vincenzo Fiore(4), Alberto Cracolici(5), Miriam Spatafora(6)

(1) Department of Architecture, University of Palermo, Palermo – giuseppe.alaimo@unipa.it
(2) Dep. of Civil, Environmental, Aerospatial, of Materials Engineering – antonino.valenza@unipa.it
(3) Department of Architecture, University of Palermo, Palermo – daniele.enea@unipa.it
(4) Dep. of Civil, Environmental, Aerospatial, of Materials Engineering – vincenzo.fiore@unipa.it
(5) Department of Architecture, University of Palermo, Palermo – albertoflare@gmail.com
(6) Department of Architecture, University of Palermo, Palermo – miriamspatafora@gmail.com

Abstract
The technology of dry assembly for the production of multi-layer panels has evolved over the past 30 years, becoming an efficient alternative to traditional systems. The direction taken by the market has been increasingly drawn to eco-sustainable systems and in this way, the interest in the use of laminated composite panels for cladding, basalt fibres based. Basalt is a natural rock, silica based, which is produced through a variety of industrial processes into filaments, wool, chopped strands and fabrics. It is characterized by high resistance in chemically aggressive environments, high fire resistance, high mechanical resistance to compression and bending. The study focuses on two types of composite laminates produced by means of the technique of vacuum bagging, using layers of unidirectional and random basalt fabrics, with different areal weights, using an epoxy resin as matrix. The laminate samples were subjected to cycles of artificial aging in climatic chamber and outdoor exposure for a year and the parameters considered most significant: flexural modulus, tensile strength and mass, were investigated. The experimental investigations were conducted according to the methodology for the evaluation of the durability of the ISO 15686. Today, this is a requirement as timely as ever after the entry into force of the new EU Regulation 305/2011, which requires, for all construction works, the declaration of sustainability of the product and the requirement of durability, too. The results show the effectiveness over time of produced composite laminates and initial increase of measured performances after accelerated aging.

Keywords
Durability, laminated composite panels, basalt fibres, mechanical performance
1 INTRODUCTION

Composite materials based on epoxy resin matrix and reinforcements in carbon fibres, glass fibres and aramid fibres are increasingly used in the field of constructions. Due to the environmental requirements and energy saving objectives toward which nations are moving, the tendency is to use as much as possible eco-friendly fibres like lignocellulosic ones (hemp, flax, curauá, etc.) and mineral ones, such as basalt, not only as structural reinforcements [1], but also as insulating materials. The basalt fiber has a chemical composition very similar to the traditional glass fiber but, differently from the latter, it is obtained by a simple process of spinning from basalt rock, volcanic effusive nature, containing from 45% to 52% SiO₂. It is used as an insulation material in constructions due to its remarkable attitude for thermal and acoustic insulation, having an average thermal conductivity equal to 0.035 W m⁻¹K⁻¹, and as vibration damper. It is also not a flammable material, chemically inert, resistant to corrosion and biologically stable. Basalt fibres are marketed in the form of filaments, wool, roving, chopped strand, fabric, nonwoven (i.e. mat), characterized by mechanical properties comparable, even higher than glass fibres and greater resistance in acidic environment [2]. The main area investigated by the scientific community in recent years concerns with the evaluation of the mechanical properties of basalt fibres. Interest was particularly directed toward the identification of possible advantages in the use of basalt in place of traditional structural reinforcement of reinforced concrete structures. An equally interesting use consists in laminates for the manufacture of external covering panels of buildings. In this case it is fundamental the knowledge of the performance behavior over time of the composite laminates, by means of the monitoring of the main parameters, when subjected to aging. As regards the thermal stability of glass and basalt fibres, calculating the changes generated by heat treatments to mechanical properties, it was demonstrated that if subjected to heat treatments up to 600°C for 2 hours, basalt fibres maintained their properties in a temperature range significantly higher compared to glass fibres. For temperatures higher than 400-500°C, basalt becomes less resistant than glass, maintaining higher stiffness [3]. Sim et al. [4] showed that exposure to 1200°C for 2 hours (condition representative of a fire) produced on glass and carbon fibres the loss of volumetric stability on the contrary of basalt fibres still maintaining residual stiffness.

2 RESEARCH METHODOLOGY, MATERIALS USED AND PARAMETERS ANALYZED

2.1 Objectives and procedures

The aim of the research consisted in testing the durability of two types of composite panels basalt fibres based, made with a different sequence of basalt fibres layers, in terms of mechanical performance and physical-chemical parameters. The experimental work is the first step to evaluate the possibility of using basalt fibres for the production of composite panels for high energy performance building envelopes. The tests were carried out on the technical solution of the perimeter wall, following the procedure laid down in the ISO 15686-2:2012 (Buildings and constructed assets - Service life planning. Part 2: Service life prediction procedures) and the UNI 11156-3:2006 (Evaluation of the durability of building components. Part 3: Evaluation method for endurance or service life). These two standards require an approach based on the following points:
Definition: the identification of user requirements, technological requirements connoting the technical element, stressing agents, performance requirements and characterization of materials.


Preliminary tests: these are needed to assess whether the test methodology adopted and the techniques of measurement of functional characteristics.

Exposure and measurement: this phase includes the tests of accelerated aging and the monitoring of natural degradation due to the stressing agents on building components.

Analysis and interpretation of the results: the results obtained with the testing are analyzed and the service life of the building component under certain stress conditions is calculated.

Basalt fibres, from literature, show a remarkable resistance to harsh environments, especially acidic ones, but there is a lack of information about the maintenance of their performance when used for composite panels, especially regarding with the resistance to UV radiation and to rapid changes in temperature and relative humidity. Therefore, in defining the framework of stressing agents, the conditions under which a cladding panel is subject in the reference context of the city of Palermo (Mediterranean) were identified, being temperatures between 2°C and 60°C, relative humidity between 10% and 80%, and exposure to radiation by means of UV lamp [5]. The exposure to stressing agents was organized according to two different modes: the exposure to laboratory accelerated aging cycles and the exposure to external environmental conditions, in order to obtain useful data for comparison to the definition of the service life of the building component. The experimental program of exposure to aging was thus scheduled. As for the outdoor exposure, the effects of one year of weather conditions on the samples were monitored, orienting the samples to South, being inclined at 45° to the horizontal. The laboratory accelerated exposure of the samples was achieved by artificial aging cycles reproducing natural degradation mechanisms by means of accelerated kinetics, considering all the degradation agents considered significant and effective, identified in the preliminary analysis. The procedure for the comparison of performance obtained by the exposure of the technical component to short laboratory aging and outdoor long-term aging, is well-known as time rescaling; it allowed to predict the performance evolution over time of the technical component analysed.

2.2 Parameters monitored
The parameters considered most significant for composite panels basalt fibres, were:
- Flexural modulus;
- Flexural strength;
- Mass.

2.3 Manufacturing of samples
2.3.1 Samples of resin
Samples were made in epoxy resin (SP 106 by Gurit) dumbbell shaped. The size and shape of the samples derived from the ASTM D 638 03 (Tensile Properties of Plastics). The samples of resin were made by preparing a mixture of curing agent and resin according to the stoichiometric ratios in the data sheet of the resin, mixing the two components manually for 5 minutes at room temperature, in order to homogenize the mixture. Subsequently the resin was poured into silicone rubber molds (Fig. 1).
Fig. 1. (L) Shape of the samples of resin and (R) their manufacturing

Particular attention was paid to avoid the formation of bubbles or small defects in the samples, which could impair their performances, distorting the results of tests carried out subsequently. The samples were left to cure for 24 hours at room temperature and subsequently subjected to a process of post-curing at a temperature of 40°C for 4 hours.

2.3.2 Samples of laminate

Two types of composite laminate panels were made by overlapping 6 layers of basalt fiber fabric, according to the lamination sequences shown in Fig 2.

Three basalt fabrics were used in this work, having different weight, orientation and length of the fibres (Fig. 3): Unidirectional 300; Random 100 and Random 200. The final number denotes the areal weight of the fabric [g m⁻²]. The difference in the orientation and length of the fibres affects the mechanical characteristics of the single layer and, consequently, also the laminate composite. The unidirectional fabrics are characterized by long fibres oriented along one direction. In this way mechanical characteristics are higher in the longitudinal direction, but lower in the transverse direction. The random fabrics consist of short fibres randomly oriented, giving the fabric isotropic properties.

Fig. 2. (L) Hybrid laminate composition and (R) unidirectional laminate composition

Fig. 3 (L) Unidirectional fabric, (Centre) random 100 fabric and (R) Random 200 fabric
All samples were produced at room temperature with single lamination by vacuum bagging technology. This method involves an initial hand lay-up phase and the polymerization of matrix in a flexible bag in which negative pressure (i.e. depression) is reached by a vacuum pump. Vacuum technology brings some advantages to the final characteristics of composite laminate compared to hand lay up technology:

- greater compactness of the fiber layers to obtain both an efficient transmission of stresses and to prevent displacement of the fibres during the curing of the resin;
- moisture reduction, eliminating the presence of water negatively affecting the properties of the composite;
- optimization of the fiber/resin ratio in the composite structure, as it makes possible the elimination of resin excess;
- homogenization in the distribution of resin, avoiding the presence of dry zones or excessively impregnated ones;
- reduction of weight due to the elimination of resin excess.

The manual lamination was carried out by a repeated sequence of the following stages:
1) Placing an absorbent layer and a release sheet, named peel ply;
2) Mixing of the resin/curing agent (i.e. polymer matrix);
3) Placing the first layer and manual impregnation procedure with the matrix via roller;
4) 6 times repetition of the procedure equal to the number of layers of the composite panel;
5) Placing of a second layer of release and absorbent layers in contact with the last upper layer of the composite panel;
6) Closing of the laminate composite panel within the flexible bag. The depression inside the bag was maintained equal to -0.6 atm for 5 hours after which the panel was extracted from the bag and placed inside an oven for post-curing treatment at a temperature of 40°C for 24 hours. The laminate composites produced were cut in order to obtain samples having dimensions according to the ASTM D790 (standard test methods for flexural properties of unreinforced and reinforced plastics and electrical insulating materials). Due to the thickness of the laminate composite, equal to 2 mm, the length of the sample was set equal to 150 mm, being the distance between supports equal to 120 mm. The remaining 30 mm, 15 mm for each side, were necessary since, according to the standard, the external margin to each support, had to be at least 10% of the distance between the supports and in no case less than 6.4 mm. The water jet cutting of the laminates was performed by numerical control machine. The water jet cutting technique did not cause physical alterations or mechanical deformation to every sample. The width of the cutting groove is very low allowing containing waste to minimum. Regarding to the unidirectional laminate composite samples to expose to natural aging, these were cut in such a way the fibres were oriented perpendicularly to the direction of the length of the samples. This as to lead to a further study and quantify the reduction of resistance in the case of efforts orthogonal to the fibres. The time rescaling was not affected by this choice as, having to evaluate changes, a relative and not absolute parameter, the comparison was possible between unidirectional laminate composites cut in parallel to the fibres and those cut orthogonally. The samples of laminate composite were labelled in the following way:

- A: non-aged samples for bending flexural test;
- B, C and D: artificially aged samples for bending flexural test at the end of each cycle of accelerated aging;
- L and N: naturally aged samples for bending flexural test.
2.4 Artificial aging
To apply artificial aging in a climatic chamber, it was developed an aging cycle alternating hygrothermal stress and UV radiation. 3 steps of accelerated aging were set lasting 28 days each (Table 1). Within each step, each phase A and phase B were each repeated 24 times, for a duration of 7 days and repeated this alternation for 4 times, for 28 days overall. Phase A consisted in the hygrothermal stress and phase B in the UV irradiation.

Table 1. Values of the parameters of the accelerated aging

<table>
<thead>
<tr>
<th>PHASE A</th>
<th>Theoretical cycle (Min)</th>
<th>T (°C)</th>
<th>RH (%)</th>
<th>Real cycle (Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Transition</td>
<td></td>
<td></td>
<td></td>
<td>36</td>
</tr>
<tr>
<td>Dry climate</td>
<td>120</td>
<td>60</td>
<td>10</td>
<td>84</td>
</tr>
<tr>
<td>Transition</td>
<td></td>
<td></td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>Humid climate</td>
<td>120</td>
<td>2</td>
<td>80</td>
<td>102</td>
</tr>
<tr>
<td>TOTAL</td>
<td>240</td>
<td></td>
<td></td>
<td>240</td>
</tr>
<tr>
<td>PHASE B</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Humid climate + UV irradiation</td>
<td>180</td>
<td>35</td>
<td>87</td>
<td>180</td>
</tr>
<tr>
<td>TOTAL A+B</td>
<td>420</td>
<td></td>
<td></td>
<td>420</td>
</tr>
</tbody>
</table>

The whole experimental work consisted in 3 steps of accelerated aging, corresponding to 2016 hours (84 days) of overall stressing action in climatic chamber. For phase A, the samples were placed in the lower part of the chamber on 3 lines (2 central and 1 lateral) slightly sloped. For phase B, the samples were placed on a grid that is approximately 25 cm from a UV lamp and with guidance that follows an ideal sphere around the bulb of the lamp, to maintain constant irradiance equal to 5 Wm$^{-2}$, as illustrated in Fig 4.

3 ANALYSIS OF THE RESULTS
3.1 The investigation of the samples subjected to accelerated aging
From the data obtained from the three-point bending tests, summarized in Tables 2 and in Fig. 5 and 6, at the end of the experimentation, it was found that after the 1st step of aging, the average value of the ultimate strength was higher than the non-aged samples. In particular, it was found an 11% increase in ultimate strength and a 4.4% increase in flexural modulus. The tensile tests performed on the samples of resin showed a decrease of the ultimate strength and a not appreciable variation of the elastic modulus. These results led to the exclusion of
possible residual cross-linking of the resin due to UV radiation as a cause of the increase in mechanical performance of the laminate composites.

Table 2. Mean values of flexural properties over time for Uni, hybrid and resin samples

<table>
<thead>
<tr>
<th></th>
<th>Unidirectional</th>
<th>Hybrid</th>
<th>Resin</th>
</tr>
</thead>
<tbody>
<tr>
<td>T₀</td>
<td>238.54</td>
<td>1.28</td>
<td>23.65</td>
</tr>
<tr>
<td>T₁</td>
<td>317.4</td>
<td>1.64</td>
<td>25.51</td>
</tr>
<tr>
<td>T₂</td>
<td>273</td>
<td>1.7</td>
<td>23.4</td>
</tr>
<tr>
<td>T₃</td>
<td>146.5</td>
<td>1.8</td>
<td>11</td>
</tr>
</tbody>
</table>

Fig. 5. Average values of flexural properties over time for Uni (L) and hybrid (R) samples

Fig. 6. Mean values of the most significant parameters over time for resin samples

By measuring the percentage loss of mass, there were no significant changes (0.53% for unidirectional laminate composites, 1.32% for hybrid ones, 0.23% for the resin samples). This result was confirmed by the absence of chemical nature alterations within the composite during the aging and thus no post-crosslinking processes took place. For further confirmation DSC (Differential Scanning Calorimetry) analysis was performed to identify any thermal transitions. The analysis was carried out in inert atmosphere (nitrogen flow with a flow rate of 50 ml min⁻¹) in the dynamic mode, in the range of temperatures 40°C÷140°C, by setting a
constant heating rate of 10°C min\(^{-1}\). The analysis was conducted to assess the material residual heat of cross-linking. The test gave similar results both in the case of samples of resin at \(T_0\) and \(T_1\), thus confirming that the material did not undergo an increase in the cross-linking processes. As expected, an exothermic reaction was found at around 70°C (Fig. 7).

![Fig. 7. (L) DSC of the resin sample at \(T_0\) and (L) at \(T_1\)](image)

At the end of the 2\(^{nd}\) step, the results showed, instead, a decay of the mechanical properties of both laminate composites. For unidirectional laminate composites it was registered, in fact, a negative variation equal to 16% in the ultimate strength and a negative variation of 8% in the elastic modulus while, for the hybrid laminate composites, there was a reduction of 37.5% in the ultimate strength and of 6% in the elastic modulus. Similar results were obtained by the tensile test of the resin (percentage variation in the ultimate strength: -26%; percentage variation in the elastic modulus: -8%). The bending flexural test at the 3\(^{rd}\) step showed in fact that the unidirectional laminate composites a significant reduction in mechanical values (-52% for the elastic modulus and -87% for the ultimate strength) while the hybrid laminate composites almost stabilized on the values of the previous sampling (-4% for the elastic modulus and 5% for the ultimate resistance). Even the resin showed considerable reduction of the ultimate resistance (-33%), but no appreciable variation of the elastic modulus was found. As regards the monitoring of the mass, no significant changes were found in the 3 steps.

### 3.2 The investigation of the samples subjected to natural aging

The tests conducted on samples externally placed allowed conducting the time rescaling by comparing the mechanical changes measured with the results obtained from the tests conducted on samples subjected to accelerated aging. Both as regards the elastic modulus and the ultimate resistance (Table 3), it can be stated with good approximation that two steps of artificial aging, equal to 56 days, are equivalent to one year of natural aging.

<table>
<thead>
<tr>
<th>Hybrid A at (T_0)</th>
<th>(\sigma) [MPa]</th>
<th>Strain [%]</th>
<th>(E) [GPa]</th>
<th>(\sigma) [MPa]</th>
<th>Strain [%]</th>
<th>(E) [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hybrid N</td>
<td>92,7</td>
<td>3,36</td>
<td>4,30</td>
<td>Uni L(90°) at (T_0)</td>
<td>34,04</td>
<td>2,3</td>
</tr>
<tr>
<td></td>
<td>78</td>
<td>2,3</td>
<td>4,8</td>
<td>Uni N(90°)</td>
<td>23,9</td>
<td>1,6</td>
</tr>
</tbody>
</table>

Regarding to the unidirectional laminate composites, the values obtained refer to the samples obtained per orthogonal cutting of the fibers, thus resulting significantly lower values.
4. CONCLUSIONS

Both types of laminate composites underwent an increase in mechanical performance after the 1st step of accelerated aging, but not the resin. It was proved that UV radiation triggered post-curing process to the resin or interacting with the fibres. It is realistic that instead there was an interaction at the interface between matrix and reinforcement and then the composite material had a synergistic behaviour, due to UV irradiation. As regards the subsequent steps, for the hybrid laminate composites occurred the maintenance of the performance parameters, unlike the unidirectional ones whose mechanical characteristics showed considerably decay starting from the 3rd step, both for the elastic modulus and the ultimate strength (Fig. 8).

![Graph](image)

Fig. 8. Change in the ultimate strength (L) and the elastic modulus (R) of samples over time

This suggests the causes of degradation of the composites derive from the action of the environment on the interface fiber/matrix and not in an eventual degradation of resin or fiber. Such interfacial degradation is showed almost exclusively on unidirectional laminate composites depending on the hybrid fabrics of laminate composites protected the inner part. This is due to increased adhesion (for the arrangement of short fibres) providing kind protective action to the external environment. In terms of durability, the hybrid laminate composites showed higher performance as, even if their initial mechanical characteristics are lower compared to unidirectional ones, still maintained almost unchanged these properties. The unidirectional laminate composites should, therefore, more suitable for applications in which higher mechanical strength is requested rather than the durability, while those hybrid should be more suitable for the realization of technical elements more lasting over time.

REFERENCES

THE INFLUENCE OF CARBONATION ON DEICER SCALING RESISTANCE OF BLAST FURNACE SLAG CONCRETE (BFSC)

Hugo E. Egüez (1)(2), Nele De Belie (1) and Geert De Schutter (1)

(1) Magnel Laboratory for Concrete Research, Faculty of Engineering and Architecture, Department of Structural Engineering, Ghent University-Nele De Belie@UGent.be; Geert De Schutter@UGent.be
(2) Faculty of Earth Sciences, ESPOL University, Guayaquil–Ecuador-HugoEguezAlava@UGent.be

Abstract
Blends of BFS/OPC concretes with slag binder ratios (s/b = 0, 0.5, 0.7) were initially wet cured and stored for 7 days at 20 °C and RH > 95%, followed by 21 days dry storage at 20 °C and 65 % relative humidity. Afterwards, several samples were exposed to accelerated carbonation under 10 % CO₂ and 60 % R.H. while others were kept in laboratory controlled condition and natural CO₂ exposure. Carbonated concretes were exposed to freeze and thaw cycles under deicing salt according to CEN/TS 12390-9 standard. Samples were analysed utilizing optical and scanning electron microscopy to determine the structure left after carbonation and associate its influence in concrete’s de-icing resistance. The carbonation front was determined by spraying phenolphthalein on a freshly split surface. Open porosity of samples were determined after drying at 40 °C and 105 °C according to NBN B05-201.

Optical microscopy shows an irregular arrangement of tiny bright calcium carbonate minerals spread in the concrete paste for the BFS concrete. This pattern was also observed by SEM, a technique which shows more details of what is believed to be a porous leached paste zone co-existing next to a calcium salt enriched deposition sector. OPC concrete remains insensitive to carbonation maintaining the same scaling resistance with time, while carbonation in the blended concretes increases and scaling resistance decreases with higher BFS content.
1 INTRODUCTION

There are many advantages in the usage of blended cement concrete where high amounts of blast furnace slag (BFS) replace ordinary Portland cement (OPC). Among these benefits are improvements on concrete’s workability, reduced heat production, and if cured properly they develop good chemical and mechanical resistance. The inconvenience found in using high amounts BFS is that this last property is a condition difficult to assure specially if construction practice requires that structures are put to work in very short time and are deprived of the required curing time for concrete. Gruyaert et al. determined by image analysis of BSE-images [1] that (BFC) blast furnace cement pastes with slag/binder ratio of 0, 0.5 and 0.85 cured for 28 months reached a degree of hydration of 74%, 94% and 91% respectively for the Portland cement fraction while hydration of the slag fraction was 72% for the lower replacement and 39% for the higher. Utton [2] who studied a cement paste with relatively more slag, BFS:OPC (9:1), found only 45% for the overall degree of hydration after two years at 20 °C.

It is recognized that BFS concretes perform worse compared to OPC concretes when exposed to freeze and thaw cycles combined with de-icing salts, a deleterious process supposedly principally of physical nature due to the freezing of water in the capillary pore system of concrete. From these findings one should assume an important contribution of the hydration behaviour of the BFS on concrete’s de-icing salt scaling resistance, regarded that paste microstructure is shaped by this process. However, in addition a cautious analysis has to be done of the influence of carbonation on the initial pore structure since it might be altered by CO$_2$ attack. Several tests have been performed in this research in order to elucidate in what manner carbonation modifies the microstructure of concretes and how this affects the de-icer scaling resistance of BFS concrete.

2 EXPERIMENTAL PROCEDURE

Three concrete mixtures were proposed for this research, as presented in table 1. The aggregates were siliceous gravel and a natural sand. Portland cement was used, type CEM I 52.5 N according to European Standards, with no special durability property. A (ground granulated) blast furnace slag addition was also used to replace 50 and 70% by weight of the Portland cement. In order to improve the fresh concrete properties (slump between 160-210 mm) a super plasticizer (polycarboxilate based) was used in amounts between 0.2 – 0.4% by weight of total binder.

Table 1: Mix design, kg/m$^3$, w/b = 0.45, Slump S4 (160-210 mm)

<table>
<thead>
<tr>
<th>Material type</th>
<th>$S_0$</th>
<th>$S_{50}$</th>
<th>$S_{70}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cem I 52.5 N</td>
<td>350</td>
<td>174</td>
<td>102</td>
</tr>
<tr>
<td>BFS</td>
<td>0</td>
<td>174</td>
<td>238</td>
</tr>
<tr>
<td>Water</td>
<td>158</td>
<td>157</td>
<td>154</td>
</tr>
<tr>
<td>Sand 0/4</td>
<td>781</td>
<td>777</td>
<td>759</td>
</tr>
<tr>
<td>Gravel 2/8</td>
<td>619</td>
<td>616</td>
<td>601</td>
</tr>
<tr>
<td>Gravel 8/16</td>
<td>480</td>
<td>477</td>
<td>466</td>
</tr>
</tbody>
</table>
The chemical composition of OPC and BFS are presented in Table 2.

Table 2: Cement and slag chemical composition (LOI = loss on ignition)

<table>
<thead>
<tr>
<th></th>
<th>CaO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>SO₃</th>
<th>MgO</th>
<th>K₂O</th>
<th>CO₂</th>
<th>Na₂O</th>
<th>CT</th>
<th>Sulphide</th>
<th>Ins.</th>
<th>Resid.</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC (%)</td>
<td>63.37</td>
<td>18.90</td>
<td>5.74</td>
<td>4.31</td>
<td>3.34</td>
<td>0.89</td>
<td>0.73</td>
<td>0.50</td>
<td>0.47</td>
<td>-</td>
<td>-</td>
<td>0.41</td>
<td>1.51</td>
<td></td>
</tr>
<tr>
<td>BFS (%)</td>
<td>41.24</td>
<td>36.37</td>
<td>9.83</td>
<td>0.26</td>
<td>1.62</td>
<td>7.41</td>
<td>0.41</td>
<td>0.90</td>
<td>0.28</td>
<td>0.02</td>
<td>0.79</td>
<td>0.43</td>
<td>1.30</td>
<td></td>
</tr>
</tbody>
</table>

After mixing, concrete was cast in cylindrical molds (Ø 100 mm, h 50 mm). All cylinders were compacted on a vibrating table. Subsequently, they were stored for 7 days at 20 °C and RH > 95%. Afterwards, the cylinders were removed from the molds and maintained for an additional 21 days at 20 ± 2 °C and 60 ± 5 % RH. This condition of curing simulates more closely the real curing situation at field site.

After the initial curing, the external cylinders surfaces were covered with adhesive aluminum foil, leaving only 2 faces (top and bottom faces) uncovered, this is to permit CO₂ ingress without restriction for these two opened faces.

As shown in fig. 1, half of the samples were exposed to a 10 % CO₂ concentration in a carbonation chamber, while the other half were exposed to natural carbonation, at CO₂ concentration of 0.03 % in a climate controlled room. For both exposure conditions, the samples were maintained at 20 ± 2°C and 60 ± 5 % RH until the time for testing.

![Fig. 1: Accelerated and natural CO₂ exposures.](image)

The cylinders were tested at the age of 0, 2, 4, 8, 11, 15, and 19 weeks. For each testing age, cylinders were taken out from the curing place (accelerated and normal carbonation), sawn into 2 half cylinders (Ø 100 mm, h 50 mm) and the aluminum foil was removed. Then 3 half cylinders representing each type of mix, were tested on the trowelled carbonated surface side (top face) for deicing scaling resistance according to the temperature cycle prescribed in CEN/TS 12390-9 Standard “Testing hardened concrete-Part 9: Freeze-thaw resistance-Scaling”, Fig. 2.
The other carbonated samples were utilized to measure the concrete’s open porosity after drying at 40 °C and 105 °C according to NBN B05-201 and also to determine concrete’s carbonation depth by using 1% phenolphthalein in 70% ethyl alcohol on a freshly split surface. Several exposed concrete samples were chosen to prepare thin sections for optical microscopy analysis and Scanning electron microscopy techniques.

3 OBTAINED RESULTS AND INTERPRETATION

3.1 Carbonation depth

The carbonation was measured on the sample’s freshly split surface, spraying a solution made of 1% phenolphthalein in 70% ethyl alcohol. The carbonated surface is colourless while the non-carbonated zone turns purple due to the presence of free calcium hydroxide (Portlandite) which yields a pH ≥ 8.3. The obtained results (median of ten measurements) for accelerated 10% CO\textsubscript{2} exposure taken from 3 specimens representing 1 sample are shown in Fig. 3.

Fig. 3: Carbonation depth of concrete exposed to accelerated 10% CO\textsubscript{2} vs. the square root of time.
In the graph the carbonation depth “y” (mm) is plotted against the square root of the CO$_2$ exposure time “d” (days), in order to obtain the carbonation coefficient “A” which is the slope of the straight line and $y_0$ is the initial carbonation depth at the beginning of the carbonation exposure employing equation (1), according to Borges et al. [3].

$$y = y_0 + A \sqrt{d}$$ \hspace{1cm} (1)

The accelerated tests were run using a CO$_2$ concentration (10 %) which is about 33 times higher than the one found in Urban Cities (0.3 %) and 333 times higher than the one found in rural areas (0.03 %), Tam et al. [4].

To establish a correlation between depths ‘$y_{ac}$’ and ‘$y_n$’ (accelerated and normal), we shall consider the corresponding CO$_2$ concentration according to equation (2).

$$\frac{y_{ac}}{y_n} = \left( \frac{\text{CO}_2_{ac}}{\text{CO}_2_n} \right)^{0.5}$$ \hspace{1cm} (2)

The results shown that concrete’s sensibility to carbonation increases with increasing slag replacement content, while the reference concrete (with slag/binder ratio $s/b = 0$) during the test period has zero carbonation coefficient which means that its carbonation rate is very slow. Concretes with $s/b = 0.7$ ($S_{70}$) have a carbonation coefficient 2.2 higher than concrete with $s/b = 0.5$ ($S_{50}$).

To compare the two types of concretes with slag replacement, and have an indication of what is expected regarding the natural carbonation progress (0.03% and 0.3% CO$_2$) with time, some calculations were made using the found results utilizing equation (2), and synthetized in table 3.

Table 3. Expected carbonation depth (mm) of BFS concretes ($s/b = 0.5, 0.7$) for CO$_2$ concentration 0.03% for rural areas, 0.3% for urban Cities.

<table>
<thead>
<tr>
<th>Mix</th>
<th>Carbonation depth mm (CO$_2$ = 0.03 %)</th>
<th>Carbonation depth mm (CO$_2$ = 0.3 %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.1 4.8 6.7 8.2 9.4</td>
<td>0.3 15.1 21.2 25.9 29.9</td>
</tr>
<tr>
<td></td>
<td>0.1 10.6 14.9 18.3 21.1</td>
<td>0.3 33.5 47.3 57.8 66.7</td>
</tr>
<tr>
<td>t (years)</td>
<td>0 25 50 75 100</td>
<td>0 25 50 75 100</td>
</tr>
</tbody>
</table>

From the previous table it can be inferred that for the same environmental condition, the carbonation depth of concrete with 70% BFS replacement, progresses 2.2 times deeper than that achieved by concrete with 50% BFS.

### 3.2 Open porosity

Carbonated samples, half cylinders ($\varnothing$ 100 mm, h 50 mm) were oven dried at 40 °C and 100 °C until constant mass (< 0.1 % @ 24h) before vacuum saturation according to NBN B05-201 paragraph 6.1 (1976), the corresponding porosity was then calculated by hydrostatic weighting (air & water), the obtained results are shown in Fig. 4.
The open porosity at 40 °C gives a rough estimate of what is happening with capillarity after the samples are exposed to accelerated carbonation, thus capillary porosity tends to increase after CO₂ exposures for samples S₅₀ and S₇₀, while average porosity for S₀ concrete has remained almost unchanged. On the other hand, overall concrete porosity at 105 °C, seems to remain unaltered in all the samples.

3.3 Scanning electron (SEM) and thin section microscopy

With the purpose of exploring the carbonated microstructure of OPC and BFS concretes, several thin sections were studied under petrographic microscope, and under scanning electron microscope.

Fig. 5 shows a photograph of carbonated BFS concretes (S₅₀ & S₇₀) under parallel light.

An incompletely carbonated paste composed of tiny dull and bright crystals was observed between aggregates. It was also noticed that the calcium carbonate minerals on S₅₀ concrete seem to develop a more dense structure than that found in S₇₀ pastes.

With the intention of having a closer look at the carbonated microstructure, the same thin sections were investigated using SEM. Fig.6 shows the pore structure of carbonated BFS S₇₀.
Fig. 6: S<sub>70</sub> 19 weeks (10% CO<sub>2</sub>) carbonated paste showing a leached porous paste coexisting with a deposition zone of sponge-like carbonate salts white deposit (left), pore size shown (right).

Although the porosity of the concrete paste was not quantified, an increase of the void system was noticed as the slag replacement rises in the concrete. This can be expected if it is considered that concretes have been exposed to short wet curing times (7 days), followed by 21 days at relatively dry environment (60 % R.H.), before the carbonation exposure, which has contributed to poor OPC/BFS hydration specially at the surface, leaving a free way to CO<sub>2</sub> ingress that also continues with porosity creation by leaching action.

3.4 Freeze and thaw with deicing salt

Fig. 7. shows the results of freeze and thaw cycles (56) of carbonated half cylinders (Ø 100 mm, h 50 mm).

Fig. 7: Scaling resistance of BFS concretes (s/b = 0, 0.5, 0.7) with 3% NaCl solution (CEN/TS 12390-9), after 10% CO<sub>2</sub> exposure and environmental CO<sub>2</sub> exposure (S<sub>70N</sub>, S<sub>50N</sub>).

From these results it is concluded that carbonation does not have any significant influence on the de-icing resistance of pure OPC concrete. For the concretes made with BFS/OPC blends and cured as in this investigation, carbonation has a deleterious influence. The harmful effect increases with carbonation depth until a maximum is reached. Beyond that point there is not further increase of the deleterious action of carbonation. This behaviour could be
explained by the decrease in porosity with increasing depth into the surface found by Battaglia et al. [5] in the carbonated zone of BFS concretes, while inversely, porosity of OPC concretes increased continuously with distance from surface.

Summarizing our results it can be said that $S_{50}$ reaches its maximum rate of scaling after 6 mm of carbonation depth while $S_{70}$ needs about 12 mm to reach it.

Compared to that of OPC concrete, the average rate of scaling for $S_{70}$ and $S_{50}$ are 11 and 5 times higher respectively, while $S_{70}$ has a rate that is about twice the one found in $S_{50}$.

4 CONCLUSIONS

– While the freeze and thaw performance of BFS concretes is considered good, its resistance in the presence of de-icing salts can be qualified as poor, especially when concretes are carbonated.
– Carbonation is a complex process which depends not only on the nature of materials used to prepare the concrete, but also on the amount and proportions utilized, curing conditions and the amount of CO$_2$ the concrete is exposed to.
– The purpose of this research is to acquire information from BFS concrete de-icer scaling resistance when subjected to unfavourable conditions for hydration and the best situations for carbonation, such as a short period of wet curing, followed by a long and steady period of intermediate relative humidity. In such circumstances BFS consumes part of the produced Ca(OH)$_2$ for its hydration and competes with CO$_2$ in its depletion. In this scenario and when CH is not available, carbonation of CSH is started [5] giving as a result a leached microstructure, the progress of hydration is reduced. Porosity and permeability are increased enabling the ingress of more CO$_2$ to the system continuing the damage.
– The augmented BFS concrete’s porosity and permeability driven by carbonation in the concrete microstructure, provides the most unfavourable condition for its deicer scaling resistance.

ACKNOWLEDGEMENTS

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DISSOLUTION KINETICS EXPERIMENTS AS A METHOD TO ASSESS THE REACTIVITY OF SUPPLEMENTARY CEMENTITIOUS MATERIALS (SCMs): THE EFFECT OF SOLUTION COMPOSITION AND SCM STRUCTURE

R. Snellings (1), K. Scrivener (1)

(1) Laboratory of Construction Materials, Ecole Polytechnique Fédérale de Lausanne (EPFL), Lausanne, Switzerland – ruben.snellings@epfl.ch

Abstract
One of the most effective ways to improve the sustainability of cement production is the blending of Portland clinker with SCMs. However SCMs are less reactive than clinker so leading to slower development of strength and other properties. This limits the amount which can be substituted. Better understanding of the intrinsic reactivity of SCMs would facilitate the formulation of better and more sustainable blended cements.

Direct measurements of the reactivity or the degree of reaction of a SCM in a hydrating cement paste are a complicated task. Separating the contribution of the SCM reaction from the complex interplay of cement hydration reactions is not straightforward and the direct quantification of the amount of unreacted SCM may be hindered by issues such as SCM fineness or crystallinity. Therefore a better approach could be to breakdown the hydration of blended cements by analysing separate reactions in simplified model systems. This approach presents the added advantage of enabling the variation of individual parameters such as SCM or pore solution composition without affecting the hydration of the clinker phases.

This contribution presents a comprehensive study of the reactivity of a range of synthetic SCMs by dissolution experiments at high dilution and varying pH and links the obtained results to the hydration of the same SCMs in blended cements. The results show a strong dependence of the SCM reactivity and surface chemistry on pore solution composition and deliver insights that pave the way to actively enhancing the reactivity of SCMs in cement.

1 INTRODUCTION

The cement and concrete of the future will be blended. Blended cements containing one or more supplementary cementitious materials (SCMs) already make up the large majority of produced cementitious binders. In 2010, the clinker factor, the clinker fraction in the final cement, was estimated at 0.77 as a global average. Considering the global 2012 cement production of 3700 Mt, at least 500 Mt of SCMs were used in cement and concrete on a yearly basis. To reduce CO₂ emissions and the exploitation of primary resources, cement producers are looking to ways to reduce clinker factors further without compromising the performance and durability of their product. The supplies of conventional SCMs such as blastfurnace slags (215 Mt/y) are limited and future reductions will need to depend on an
increased use of other industrial by-products such as coal fly ashes (670 Mt/y), or primary resources such as natural pozzolans and thermally activated clays. Ternary blends of clinker with SCMs and limestone have received recent attention and show potential to driving down clinker factors beneath 0.55 [1], far below the industries 2050 goal of 0.71.

One of the major limiting factors in clinker substitution is the low reactivity of alternative SCMs. Identifying the parameters that control the SCM reaction kinetics in blended cements is not a straightforward task due to the complex interplay of reactions. The impact of parameters such as the SCM fineness and composition, but also the cement pore solution composition on the SCM reaction kinetics needs to be further explored. The analysis of the overall hydration reaction into separate parts has been proven to be a successful in further elucidating the mechanistic controls.

The cement hydration reaction can be divided into two balanced parts occurring simultaneously: the dissolution of the anhydrous reactants (clinker, SCMs) and the precipitation of the hydrate products. Models describing cement hydration have considered mainly the nucleation and growth of hydration products, dissolution was largely ignored. Recently however, based on concepts developed in geochemistry, Juillard et al. [2] and Nicoleau et al. [3] demonstrated that the dissolution behaviour of C$_3$S and C$_2$S enabled to describe cement hydration kinetics over the first hours of hydration, both in dilute and concentrated solutions.

A central concept in the description of the dissolution behaviour is the dependence of dissolution rates on the undersaturation degree - the ratio of the ion activity product and the solubility product - of the solution. When during the initial dissolution period solution concentrations rise, C$_3$S and C$_2$S dissolution rates strongly drop and result in the induction period of slow reaction. The subsequent precipitation of hydration products triggers the removal of ions from solution and thus enables dissolution and overall hydration rates to increase again during the acceleration and main reaction stage. Dissolution and precipitation are thus strongly coupled during cement hydration and the dependence of dissolution rates on the solution composition should not be ignored and can be used to steer hydration kinetics.

Initial dissolution rates of glasses of typical SCM composition in pH 13 solutions were recently demonstrated to be 2-4 orders of magnitude lower than for C$_3$S and C$_2$S in far-from equilibrium solutions (Table 1; [4]), correlating with a general lower reactivity of SCMs. Optimizing the reactivity of SCMs and further decreasing clinker factors in blended cements will depend on the development of generic quantitative models describing the dependence of dissolution rates on the SCM glass structure and the cement pore solution composition. This contribution mainly describes the impact of both SCM and solution composition on dissolution mechanisms and kinetics. In addition, the link between dissolution rates in dilute solutions and the reactivity of SCMs in the concentrated cement pore solution is elaborated.
Table 1: Chemical composition and initial dissolution rates \( (r_{0,\text{BET}}) \) at pH 13 of the synthetic glasses, the blastfurnace slag and the metakaolin used in the present study.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Chemical composition (wt%)</th>
<th>log ( r_{0,\text{BET}} ) (mol/m(^2)/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CaO MgO Al(_2)O _ SiO(_2) Fe(_2)O _ TiO(_2) K(_2)O Na(_2)O MnO SO(_3) P(_2)O(_5)</td>
<td></td>
</tr>
<tr>
<td>G1</td>
<td>BFS 43 - 19 38</td>
<td>-6.87</td>
</tr>
<tr>
<td>G2</td>
<td>BFS 36 - 16 48</td>
<td>-7.33</td>
</tr>
<tr>
<td>G2M5</td>
<td>BFS 31.9 3.3 16.2 48.6</td>
<td>-7.41</td>
</tr>
<tr>
<td>G2M15</td>
<td>BFS 23.5 10 16.6 49.9</td>
<td>-7.29</td>
</tr>
<tr>
<td>G3</td>
<td>FA 20 - 37 43</td>
<td>-7.97</td>
</tr>
<tr>
<td>G4</td>
<td>FA 15 - 27 58</td>
<td>-8.11</td>
</tr>
<tr>
<td>G5</td>
<td>NP 6.5 - 11.5 82</td>
<td>-8.31</td>
</tr>
<tr>
<td>G6</td>
<td>SF 0 - 0 100</td>
<td>-8.45</td>
</tr>
<tr>
<td>Slag</td>
<td>BFS 40.4 8.6 11.4 38 0.38 1.23 0.5 0.27 0.45 0.1</td>
<td>-7.03</td>
</tr>
<tr>
<td>MK</td>
<td>MK 0.02 0.09 47.6 51.4 0.38 0.18 0.28 0.08</td>
<td>-7.61</td>
</tr>
</tbody>
</table>

2 MATERIALS AND METHODS

A range of glasses was synthesized to represent typical SCM compositions, ignoring minor elements. In a first series of calcium aluminosilicate (CAS) glasses, compositions varied between blastfurnace slag and silica fume. In a second series CaO was replaced by MgO (molar substitution) in the glass to investigate the effect of Mg on glass dissolution. A commercial blastfurnace slag and metakaolin were added for comparison. The chemical composition of the samples is given in Table 1. Synthesis and sample preparation procedures are described in detail in [4,5].

Initial dissolution rates of the glasses and SCMs were determined from the linear increase of elemental concentrations over time during glass dissolution in a pH 13 NaOH solution. The dissolution rates presented in Table 1 were obtained by normalization to the initial BET specific surface area. The glass surfaces after dissolution were visualized by SEM on carbon coated stubs.

Glass surface titration experiments were carried out to study the response of the glass surface to solutions of varying pH. Glasses were suspended in a 0.01 M NaCl solution for 4-6 h to create a stock solution. Aliquots of the stock solution were titrated with fixed amounts of HCl and NaOH solutions to effectuate a pH change. The resulting solutions were stirred for 30 min and filtered at 0.2 µm. The solution pH was measured immediately after filtration and elemental concentrations were analyzed by ICP-OES. The experiments were carried out at room temperature under a N\(_2\) atmosphere.

The reaction of the SCMs/glasses in blended cement pastes was followed by monitoring the heat release during hydration and by directly determining the SCM degree of reaction by image analysis of BSE-element mappings as detailed in [5].

3 RESULTS

A comparison of the pH 13 dissolution rates of the calcium aluminosilicate glasses reveals a clear dependence of the dissolution rate on the glass structure. The ratio of framework modifiers (Ca, Mg) to framework formers (Si, Al) is indicative for the depolymerisation of the
glass aluminosilicate framework; the higher Ca and Mg, the higher the dissolution rates. The replacement of Ca by Mg in the glass did not have a significant effect on the dissolution rates.

The glass surface features as visualized by SEM (Fig. 2) revealed angular glass particles with clear surfaces prior to the dissolution experiments. After 48 h of dissolution in a pH 13 solution, all glasses except the ones containing Mg showed smooth and clear surfaces. The Mg-containing glasses and the blastfurnace slags showed the precipitation of hydrotalcite on the glass surface. The precipitation occurred without changing the dissolution rates calculated from the release of Si to the solution. The dissolution experiments showed that dissolution was congruent at pH 13 for CAS glasses. Also the dissolution of the Mg-CAS glasses can be considered congruent if the precipitation of hydrotalcite is taken into account.

The pH titration experiments was used to evaluate glass dissolution for pHs below 13. The Si released by the glass can be considered as a proxy for the dependence of glass dissolution rate on pH (Fig. 3A). For all glasses the Si release was lowest near the immersion pH,
established in the stock solutions. The immersion pH was related to the glass basicity ((Ca+Mg)/(Si+Al)). Depolymerized glasses (G1, G2) were most stable around pH 10 and became increasingly soluble at both high and low pH. Polymerized glasses (G3-G6) were less soluble overall and showed a wider range of stability between pH 5 and 9. Comparing the release of the other glass components to the release of Si, the dissolution congruency can be verified. At pH below 12, the experiments showed that Ca was being preferentially released or leached from the glass surface as illustrated for glass G2 in Fig. 3B. Al concentrations in solution were lower than could be expected from the Si release, and can be explained by the low solubility and precipitation of Al(OH)₃ at near neutral pH.

For G2 glasses and slag with a similar particle size distribution, recent work [5] investigated if the differences in dissolution rates (Table 1) translated in similar variations in the SCM reaction kinetics in blended cements. Fig. 4 shows two independent methods to assess the degree of reaction of SCMs. Both electron microscopy image analysis and a comparison of the heat released by blended cements with inert quartz filler and the SCMs indicate that the reactivity of the G2 glasses is about half that of the slag. This suggests that relatively small differences in dissolution rates may translate in important differences in reactivity.

![Fig. 3: Degree of reaction of G2, G2M5 and G2M15 glasses and blastfurnace slag as determined by BSE image analysis. B) Cumulative heat of hydration (normalized to cement content) of a reference cement and blended cements containing slag, G2, G2M5 and G2M15 glasses as SCMs and quartz as inert filler (adapted after [5]).](image)

4 DISCUSSION

The high pH dissolution experiments on the SCM glasses fill an important knowledge gap. Dissolution studies in geochemistry that established pH-dissolution rate dependencies were mainly carried out in acidic to low-alkalinity conditions (pH 2-12). Even though falling outside this experimental range – and thus in need of experimental validation – the dissolution rates for common minerals were extrapolated to pH 13 (25°C) and compared to the experimental glass dissolution rates in Fig. 4. All minerals showed lower pH 13 dissolution rates than the CAS glasses. Least soluble were Mg-silicates such as forsterite and diopside, and kaolinite. Common basaltic rock, quartz and albite feldspar showed dissolution rates one
to three orders of magnitude less than the CAS glasses, rendering them essentially unreactive at comparable fineness.

The obtained results enable to discuss a model for SCM glass reaction during Portland cement hydration. The reaction environment in hydrating cements is mainly controlled by the hydration reactions of the major clinker minerals. Initial rapid dissolution of the clinker minerals and alkali sulfates combined with the precipitation of ettringite, portlandite and C-S-H result in a rapid rise in solution alkalinity and the establishment of a high pH pore solution. Glass dissolution is strongly enhanced by the rise in pH (Fig. 2A) and the low solubility of C-S-H will keep pore solution silica concentrations at far from equilibrium conditions for glass dissolution. The increasing glass dissolution rates with pH correlate with the dissolution process becoming congruent. At high pH the glass decomposition rate becomes similar to the rate of outward diffusion of cations that produces a leached layer at lower pH. In consequence, the recombination of silica and alumina in the leached layer to form a passivation layer is avoided and further dissolution is not hindered (Fig. 5).

Hydrated reaction products form readily in hydrating cementitious systems. The low solubility of cement hydrates (C-S-H, ettringite, AFm phases) results in an almost complete transfer of dissolved reactants into precipitated solids. Widespread precipitation and growth of hydrates is usually preceded by a nucleation stage. The number and size of nuclei that form depend on the degree of supersaturation of the solution. Once the nuclei have reached a critical size, they become stable and hydrate growth will start. At this moment the solution will have reached a critical supersaturation degree (towards the hydrate products), that rapidly
drops when hydrate growth accelerates. For C₃S hydration, dissolution has been demonstrated to be strongly decelerated during the C-S-H/CH nucleation period. The reduction in solution saturation during the subsequent hydrate growth stage is coupled with an increasing C₃S dissolution rate and a resumption of the overall hydration reaction [3]. The resumption of hydration due to precipitation in alkaline media has also become a concern in glass durability studies for nuclear waste disposal [10]. Especially in Ca-rich, alkaline environments such as cement pore solutions, nuclear glass containers may eventually be consumed over the long time-scales of storage [11,12], and cause remobilization of hazardous elements.

Hydrate precipitation also affects the reactivity of SCMs in cements. The formation of hydrotalcite reaction products during the initial stage of the Mg-glass dissolution allowed dissolution rates to remain high (Fig. 5). This contrasts with the very low dissolution rate of Mg-silicate minerals at high pH (Fig. 4). In the case of Mg-silicates this is explained by slowly dissolving Mg-O complexes/layers formed at the glass surface at pH above 12 [13]. In the case of slag the availability of Al enables the precipitation of hydrotalcite-like phases \( \text{Mg}_x\text{Al}_y(\text{CO}_3)(\text{OH})_{16}.4\text{H}_2\text{O} \) reducing solution saturation and increasing dissolution rates as demonstrated for nuclear glass in the case of smectite [14] and M-S-H [15] precipitation at pH below 11.

Fig. 5: Glass surface dissolution mechanisms depending on solution pH and time.

The cement pore solution is controlled by the hydration product assemblage. Changing the hydration product assemblage is an indirect but effective way of activating SCMs as illustrated by supersulfated slag cements. The addition of Ca-sulfate source results in the precipitation of ettringite as major product [16]. Because of the low solubility of ettringite, the Al activity in the pore solution is much reduced and the slag dissolution rate is enhanced. Altering the C-A-H phase assemblage by adding carbonates to blended cements is showing similar effects on the reactivity of Al-rich SCMs such as metakaolin, and can partially explain the improved reactivity of metakaolin in combination with limestone in ternary blended cements [1,4].

5 CONCLUSIONS AND PERSPECTIVES

This paper describes and discusses the reactivity of a range of synthetic and commercial SCMs. The dependence of dissolution rates and mechanisms on both glass and solution composition are explored, identifying the glass SiO₂ content and the solution pH as primary factors controlling the reactivity of SCMs in cement. In addition, the coupling and interplay
between glass dissolution and hydrate precipitation were demonstrated. Future work will need to further develop quantitative links between SCM dissolution rates, pore solution composition and hydrate precipitation.

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DURABILITY OF STRUCTURAL STEEL IN TRANSITIONAL FRAMED BUILDINGS

Sara C. Guerrero, P.E. (1)  
(1) Facility Engineering Associates, P.C., Fairfax, VA, USA  
bentz@feapc.com  Sara.Guerrero@feapc.com

Abstract  
The American Institute of Steel Construction (AISC) defines serviceability as “a state in which the function of a building, its appearance, maintainability, durability, and comfort of its occupants are preserved under normal usage.” AISC states that, “structural components shall be designed to tolerate corrosion or shall be protected against corrosion that may impair the strength or serviceability of the structure.” The parallel can be drawn that protection from corrosion would increase durability. In early 1900 United States, building façade design was making the transition from mass masonry to curtainwall systems. The introduction of steel framing into façades was a way to reduce the thickness of the masonry, but consideration for the serviceability of the steel was not typically made. As these transitional-framed buildings have aged, the resulting water absorption through the masonry in close contact with the structural steel has resulted in hidden corrosion issues and the phenomenon known as “rust-jacking” which can ultimately lead to section loss in the structural steel members. The paper will draw from the author’s experience in evaluating and repairing transitional buildings and develop an opinion on the expected useful life of steel when encased in masonry with examples of severely corroded steel.
1 INTRODUCTION

The evolution of building construction has followed a steep learning curve since the turn of the 20th Century. In many regards, this evolution has been driven by the cost of construction materials, the cost of labor, and the desire to gain as much square footage as possible by building higher buildings on reduced available land. Throughout this evolution, building designers have adjusted the materials they use to meet these demands and have often relied on new technologies to reach new structural and architectural heights. A significant change occurred in the early 1900’s where load-bearing masonry walls as the primary structural support began to be replaced by the idea of a curtainwall and a structural steel superstructure supporting the building. During this transitional period, many designers did not take full advantage of the steel superstructure, or possibly did not have available materials to do so. As such, wall thicknesses initially did not decrease, but rather were built around the steel. This transitional-frame approach resulted in minimally-protected (shop primer) steel in direct contact with masonry in many taller buildings. As these buildings have aged and the steel has been cyclically exposed to moisture absorbed through the masonry, the steel has oxidized and developed a condition known as “rust pack”, or “paging”. As the rust accumulates on the steel, the resulting increased thickness must be accommodated, and results in movement of the surrounding masonry. If sufficient space to accommodate the movement is not available, the masonry will shift and crack as the rust pack grows. This phenomenon of continued rust accumulation is referred to as oxide-jacking, or rust-jacking. A significant amount of the United States turn-of-the-century building inventory in large metropolitan areas such as New York, Chicago, Boston, and other cities is affected by this phenomenon. If left unchecked, structural failure can occur from the resulting loss of cross-section within the steel components. This paper will discuss different observed cases of rust-jacking and the resulting deterioration of the underlying steel. The observations will be used to develop an estimated service life, as related to durability, for structural steel in transitional-framed buildings.

2 MASONRY

For the purposes of this paper, masonry will be discussed in three categories; stone, terra cotta, and clay brick. All three construction methodologies employ unitized components, set in mortar, to make-up the final wall assembly. The systems require reinforcement in the form of steel lintels or iron rods in order to carry loads over window openings or large spans. The primary distinctions between the three masonry materials for the purposes of this paper are the overall moisture absorption rates of the components and the number of resulting joints, as summarized in Table 1 below.

<table>
<thead>
<tr>
<th>Masonry Material</th>
<th>Typical Unit Length</th>
<th>Typical Unit Finish</th>
<th>Water Absorption Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stone(^2)</td>
<td>2-4 feet</td>
<td>Various Chiselled or Polished Finishes</td>
<td>Granite – &lt; 1%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Limestone – 10-12%</td>
</tr>
<tr>
<td>Terra Cotta(^3)</td>
<td>18-24 inches</td>
<td>Baked-On Glaze</td>
<td>Glazed – 4.2-6.5%</td>
</tr>
<tr>
<td>Clay Brick(^4)</td>
<td>6-12 inches</td>
<td>Un-Glazed</td>
<td>Un-Glazed – 15-20%</td>
</tr>
</tbody>
</table>
In general, it can be seen from Table 1 that for a 20-foot length of wall, each system would have a dramatically different amount of mortar joints. Considering that mortar is used in each assembly to place the units, it follows that the larger the unit surface area, the less mortar in the wall. With mortar being significantly more absorptive than any of the masonry materials, the amount of water in the wall is primarily a function of the size and absorptive properties of the masonry units as well as the resultant quantity of absorptive mortar joints.

3 WALL ASSEMBLIES

For the purposes of this paper, three wall systems were considered as described in Table 2. Each wall assembly is found at the parapet, typically on the uppermost levels of a building.

Table 2: Wall Assemblies

<table>
<thead>
<tr>
<th>Assembly</th>
<th>Age</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Limestone</td>
<td>1929 Construction</td>
<td>9-12 inch thick stone over 3-wythes brick</td>
</tr>
<tr>
<td></td>
<td>2009 Repair</td>
<td></td>
</tr>
<tr>
<td>2. Granite/Terra Cotta</td>
<td>1913 Construction</td>
<td>15-inch thick stone or glazed terra cotta over clay brick back-up</td>
</tr>
<tr>
<td></td>
<td>2013 Repair</td>
<td></td>
</tr>
<tr>
<td>3. Clay Brick</td>
<td>1929 Construction</td>
<td>3 to 4-wythes of brick masonry</td>
</tr>
<tr>
<td></td>
<td>2009 Repair</td>
<td></td>
</tr>
</tbody>
</table>

The wall assemblies are graphically illustrated in Figures 1-3. The steel where corrosion due to masonry embedment and loss of cross-section occurred is shown by the dark lines.

4 OBSERVATIONS

The following observations were made once the steel was uncovered during construction:

4.1 Assembly 1

The three primary steel components observed at Assembly 1 were the spandrel, channel, and blocking. The blocking and channel combined create an outrigger assembly to support the thicker portions of the masonry wall. The spandrel supports the adjacent floor system and the back portion of the parapet. Due to the construction being clay brick masonry throughout the wall depth, there are a multitude of mortar joints and paths for water to travel to the steel. At this location, rust-pack in excess of 1-inch thickness was removed from the channel, and blocking. The channel and blocking were effectively reduced to nothing, with 100% cross-section loss noted in many locations. At this location, the masonry had essentially become self-supporting. The spandrel had approximately 1/2-inch of rust-pack removed from the web and nearly 1-inch removed from the flange. As can be seen in Figure 4, the upper flange had been reduced to a ragged “knife-edge” requiring the addition of welded plates to supplement the section loss.
Figure 1: Assembly 1

Figure 2: Assembly 2
Figure 3: Assembly 3

Figure 4: Spandrel and channel of Assembly 1.
4.2 Assembly 2

The three primary steel components observed at Assembly 2 were the stone anchor rod, channel, and angle. The stone anchor rod restrains the granite water table against rotation. The channel supports the edge of the roof slab and also the outer portion of the cornice below and the parapet above. The angle supports the granite water table against gravity loads. At this assembly, 100% section loss of the stone anchor rods was observed due to their embedment in the back-up masonry and nearly constant exposure to moisture. The remainder of the steel components had either granite or concrete somewhat protecting them from constant exposure, so rust was limited to surface only.

![Stone anchor rod at Assembly 2.](image)

**Figure 5: Stone anchor rod at Assembly 2.**

4.3 Assembly 3

The three primary steel components observed at Assembly 1 were the channel and connection angles and blocking. At this location, rust-pack in excess of 1-inch thickness was removed from the channel. The channel was effectively reduced to nothing, with 100% cross-section loss noted in many locations. At this location, the 8-inch thick limestone masonry had shear-cracked along a horizontal line at the channel level due to the 1-inch outward movement from the rust-pack. As can be seen in Figure 6, the web of the channel was corroded through, requiring complete replacement.
5 CONCLUSIONS

Each of the assemblies considered demonstrates a different depth of water penetration or resulting steel corrosion location. Not only did the assembly configuration affect the amount of rust, but it also played a role in where the rust occurred. While Assemblies 1 and 3 show corrosion of the web and flange of the structural steel channel and blocking, Assembly 3 showed only loss of cross-section at the steel rod and areas of excessive water penetration at the steel lintel.

Based on these observations, the authors have concluded that the expected useful service life of steel in embedded in masonry can be estimated at 50-years. Although the cases shown have exceeded the 50-year indicated life, they have also shown significant loss of load-carrying capacity in the members considered. Therefore, it is our conclusion that at the 50-year mark steel embedded in masonry has reached a critical point where corrosion has moved from surface exfoliation to detrimental cross-sectional loss. It is our opinion that structures with embedded masonry should be evaluated for rust-jacking conditions in the 40-50 year range and the resulting amount of section loss evaluated. If protective measures such as preparation, priming, painting, and through-wall flashings are implemented, it is possible to allow steel to function effectively beyond the 50-year mark. Additional consideration should
be given to new technologies including cathodic protection and impressed current to preserve the steel in aging structures.

Further study of this topic is on-going as the authors continue to expand their inventory of transitional buildings repaired. The authors are continuing to develop a monitoring program using 3D laser-scanning technology to observe small movements in the masonry façade over time, to then develop predictive models for cross-section loss with minimal disruption to the façade. This topic will be revisited in later publications.

REFERENCES
DEVELOPMENT OF EVALUATION METHOD BY SMALL CORE SPECIMEN FOR DETERIORATED FIBER CEMENT CORRUGATED SHEET AND SOME RESULTS OF ROOFS OVER STATION PLATFORMS

Saori Ishihara (1) and Kyoji Tanaka (2)

(1) Chiba Institute of Technology, Chiba – saori.ishihara@it-chiba.ac.jp
(2) Tokyo Institute of Technology, Tokyo – tanaka-kyoji@kme.biglobe.ne.jp

Abstract
This paper studies the availability of deterioration assessment for fiber cement corrugated sheet with small core specimen. Normally, mechanical properties of fiber cement corrugated sheet are measured by bending test with one fiber cement corrugated sheet. However it requires a huge amount of effort to remove presently-used sheet. Therefore deterioration assessment with core specimen 10mm in diameter which can remove and patch after remove specimen easily was developed.

Small core specimens that had been exposed to weathering from 10 to 50 years were collected. Mechanical properties were tested with split-compression test and microstructure was observed by optical microscope and EPMA analysis.

Two types of fracture modes were observed on split-compression test, one is delamination which observed all specimens, and the other is splitting failure which observed a few specimens. Following the increase in past years, maximum load per specimen unit thickness measured by split-compression test and bending strength measured by bending test are decreased. Also, there is good correlation between maximum load per specimen unit thickness and bending strength.

Exact thickness was measured by optical microscope and element distribution was measured by EPMA analysis. Ca leaching was observed with long-term aging specimen which contributes to the deterioration.

It is concluded that it can evaluate of deterioration for fiber cement corrugated sheet with 10mm in diameter core specimens.
1 INTRODUCTION
Fiber cement corrugated sheet is a composite material comprised of fibrous material solidified in cement. It is relatively light, but has superior fire and water resistance properties. Consequently, it is often used in Japan for roofs of factories, warehouses and station platforms. Fiber cement corrugated sheet has relatively high durability. Even so, deterioration, such as cracks and wear, has progressed in recent years[1]. Tackling cracks and damage to roofs is particularly important because of the risk of accidents that may lead to property damage and physical injury caused by falling broken pieces.

To prevent such accidents, it is necessary to identify in advance whether repairs or replacement sheets are necessary. However, in order to do so, several pieces of information, focussed on strength in particular, have to be obtained to determine to what degree the fiber cement corrugated sheet has deteriorated. In Japan, the bending test method is specified in JIS A5430 as the test for finding the strength of fiber cement corrugated sheet, but a minimum of two actual-scale fiber cement corrugated sheet are required for this. However, because the removal of fiber cement corrugated sheet is a large-scale operation, implementing this test at the investigation and diagnosis stage poses a number of problems. Thus, a simple strength determination method is desirable. In addition, information relating to reduced sheet thickness and the degraded state of the surface, etc., which are factors in fiber cement corrugated sheet deterioration, is also required.

With these factors in mind, in this study, we aim to develop a test method for a simple evaluation of fiber cement corrugated sheet deterioration using a small core test specimen and use this method to clarify the deterioration condition of fiber cement corrugated sheets following long-term use on station platforms.

2 DEVELOPING A METHOD FOR EVALUATING FIBER CEMENT CORRUGATED SHEET DETERIORATION USING A SMALL CORE TEST SPECIMEN

2.1 Fiber cement corrugated sheet used in the test
The test pieces for the deterioration evaluation were taken from the large fiber cement corrugated sheets used for roofs of station platforms. In total, 18 sheets were obtained from 16 stations, including stations in coastal and heavy snowfall regions.
Table 1 shows the corresponding number of locations from which fiber cement corrugated sheet was collected for each number of years in use. All of the fiber cement corrugated sheets had been in use for more than 10 years and the oldest sheets had been in use almost 50 years.

Table 1: The corresponding number of locations from which fibre cement corrugated sheet was collected for each number of years in use

<table>
<thead>
<tr>
<th>Age of service</th>
<th>11-15</th>
<th>16-20</th>
<th>21-25</th>
<th>26-30</th>
<th>31-35</th>
<th>36-40</th>
<th>41-45</th>
<th>46-50</th>
</tr>
</thead>
<tbody>
<tr>
<td>number of specimen</td>
<td>3</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>3</td>
<td>2</td>
<td>3</td>
</tr>
</tbody>
</table>
2.2 Taking test pieces from the fiber cement corrugated sheet
2.2.1 Small core test specimens
We used a small diameter core drill with a bit that has a major diameter of 18mmφ and a minor diameter of 10mmφ. Fiber cement corrugated sheet has a wavelike cross section and therefore we took test pieces from two locations; the valley part (the part with the curved surface) and the side part (the relatively flat part). Also, because the roofs are roofed so that parts overlap, there are some non-exposed parts. The progress of deterioration of these non-exposed parts is relatively slow because they are not directly exposed to sunlight and rainwater. Thus, we took test pieces from both exposed and non-exposed parts. Figure 1 is an example of one of the small core test specimens taken. We took 5 test specimens from each location.

2.2.2 Rectangular test specimens
We also took small rectangular test specimens from the side part of the fiber cement corrugated sheet to examine the bending strength. The test pieces acquired were rectangular with a length of 100mm and a width of 25mm and were taken from along the longitudinal direction of the sheets. In this case also, 5 test pieces were taken from both exposed parts of the sheets and overlapped, non-exposed parts.

2.3 Strength tests
2.3.1 Splitting compression test* on small core test specimens
We removed any burr, etc. that remained on the edges of the acquired test pieces using nippers and shaped them into cylindrical shapes using a file. The diameter of each test piece was 10mm, but the thickness varied depending on the product and the location from which the test piece was taken so we used callipers to measure the width of each test piece. As a mechanical test, we conducted a splitting compression test whereby load was applied vertically to the cross section of the small core test specimen. The test conditions are shown in Figure 2. The test was conducted at a temperature of 23 ± 2°C and at a loading rate of 0.5mm/minute. The load and displacement were measured.

2.3.2 Bending test on rectangular test specimens
The thickness of the test piece was measured before the test in 2 locations close to the loading point at the centre of the test piece and a bending test conducted according to JIS A 1408. The test conditions are shown in Figure 3. Pressure was applied from the upper surface of the roof. In the same way as the splitting compression test, the test was conducted at a temperature of 23 ± 2°C and at a loading rate of 0.5mm/min. The load and displacement were measured.
2.4 Cross-sectional observations of the small core test specimens
It is necessary to expose a smooth cross section to conduct cross-sectional observations. Because the diameter of the small core test specimen is microscopic at 10mm, to facilitate easy cutting we fixed the entire test piece using epoxy resin and once this had hardened we cut through the centre with a diamond cutter to expose the cross section and made the following observations and measurements. (1) Cross section state observation (2) Thickness measurement (3) Cross section EPMA observation.

*Splitting compression test
When conducting a splitting compression test on deteriorated corrugated slate sheets with the core removed, in some cases a splitting line is added in a longitudinal direction of the circular cross section and fractured to reduce proof stress and in other cases the sheets are delaminated in the thickness direction and crushed to reduce proof stress. The former can be regarded as pseudo tensile strength and the latter as pseudo peel strength. The results obtained here differ slightly in nature to the strength information obtained in a normal splitting test, therefore we used the name splitting compression test.

3 STRENGTH TEST RESULTS AND CONSIDERATIONS

3.1 Splitting compression test results
3.1.1 Fracture mode
Figure 4 shows the fracture modes of the test pieces following the splitting compression test. The test pieces fractured due to splitting failures as shown in the left-hand photograph and due to delamination as shown in the right-hand photograph. The majority of test pieces fractured due to delamination. However, several fractured due to splitting. Even in these cases, delamination occurred if we continued to apply pressure. This is attributed to the manufacturing method used when manufacturing fiber cement corrugated sheet. That is, fiber cement corrugated sheets are formed by making sheets from a mixture of cement and fibrous materials and laminating several layers of these sheets and applying pressure. It is thought that when the fiber cement corrugated sheet deteriorates over time, the strength between the layers decreases and these points become fragile sites and as a result the sheet slate separates into layers.

![Figure 4 Fracture modes of splitting compression test](image)

(left: splitting failure right: delamination)

3.1.2 Test results and strength evaluation items
An example of the load-deformation curve during the splitting compression test is shown in Figure 5. In the case of test pieces that have been used for fewer years, the load rises sharply.
after pressure is applied. A split then occurs vertically in the surface and the load drops. If loading is continued, delamination occurs between the layers as mentioned earlier and the load fluctuates. (Progressive splitting failure).

In the case of test pieces that have been used for many years, the load increases relatively gently as delamination occurs. The load repeatedly decreases at delamination and then increases again, but eventually decreases. Further, the load level is considerably low compared to the test pieces that have been in use for fewer years. (Progressive delamination)

The test pieces exhibited different behavior whereby the load produced a splitting failure first or delamination first, but after the maximum load was reached, there were several peaks as the load dropped. In all cases, the maximum load is important when studying the strength of fiber cement corrugated sheet. Therefore, in this study, we divided this by the minimum fiber cement corrugated sheet thickness, which we then defined as the splitting compression strength per core specimen unit thickness, and included it as a strength evaluation item.

Figure 6 shows the relationship between the age of service and the splitting compression strength per core specimen unit thickness. In the case of both the exposed and non-exposed parts, the greater the number of years in use, a tendency for splitting compression strength to lower is apparent. However, when both are compared, there is no apparent difference when the number of years in use is few, but when the number of years in use is greater, the reduction in the splitting compression strength of the exposed parts becomes greater. This is thought to be because the progress of deterioration of exposed parts is quicker than that of non-exposed parts, since exposed parts are exposed to wind and rain.

3.2 Bending test results

Figure 7 shows an example of a load-deformation curve during the bending test. The peak load value for the test pieces in use for few years is high, but this drops significantly for test pieces used for a greater number of years.

Figure 8 shows the relationship between the age of service and the bending strength. Generally, there was a tendency for the bending strength to deteriorate, the longer the fiber cement corrugated sheet had been in use and, as with the splitting compression strength, there was no apparent difference between exposed parts and non-exposed parts when the fiber cement corrugated sheet had been in use for fewer years, but when the fiber cement corrugated sheet had been in use for a greater number of years, the deterioration in bending strength tended to be greater in exposed parts.
3.3 Estimating the bending strength from the small core splitting compression test results

Evaluating the durability of fiber cement corrugated sheet presents the greatest risk of falling through the roof and of material damage and from this perspective, bending strength is important. Therefore, it is desirable if the bending strength can be estimated from the splitting compression test results using a small core test specimen. For this reason, we studied the relationship between the splitting compression strength and bending strength. Figure 9 shows the relationship between the splitting compression strength of the small core test specimen and the bending strength of the rectangular test specimen.

When splitting compression strength of both the exposed parts and non-exposed parts of the small core test specimens increase, the bending strength also increases and a relatively good correlation is apparent for both (correlation coefficient 0.801). In most cases, this relationship can be expressed using Formula (1).

\[
\sigma_{\text{bending strength N/mm}^2} = 0.14 \times \sigma_{\text{splitting compression strength N/mm}}
\]  

(1)

Figure 7 An example of the load-deformation curve during the bending test

Figure 8 Relationship between age of service and the bending strength

Figure 9 Relationship between the splitting compression strength and bending strength
4 TEST RESULTS AND CONSIDERATIONS RELATING TO CROSS SECTIONS

4.1 Cross section state observations
A cross-sectional photo of a small core test specimen used for 22 years is shown in Figure 10. The black part on the surface of the exposed part is pollution deposits. Pollution deposits could be seen on all test pieces in the case of exposed parts, but the thickness of these deposits varied depending on the number of years in use and the region from which the test pieces were taken. However, generally the layer of deposits was a lot thicker in the valley parts of the corrugated sheets compared to on the side parts. This is because pollutants accumulate easily in the valley parts. In contrast, because the non-exposed parts are protected by the fiber cement corrugated sheet they are covered with, there were no visible pollution deposits in the majority of cases and when there were any they were negligible. Further, a stripe pattern of several layers could be seen across the cross section of the fiber cement corrugated sheet, which is evidence of lamination of the cement and fibrous layers during manufacture of the commercial product.

![Figure 10 A cross-sectional photo of a small core test specimen used for 22 years](image)

4.2 Cross section EPMA observation
We used an electron probe microanalyser (EPMA) to examine the deterioration state of the corrugated slate sheet and analysed the elemental distribution of the cross sections of the small core test specimens. The EPMA measurements were conducted under the following conditions; illumination current at $5 \times 10^{-8} \text{A}$, accelerating voltage at $15.0 \text{kV}$ and with a beam diameter $10 \text{nm}$. The measurement range was adjusted to incorporate the entire thickness direction of the small core test specimen with measurements conducted within a range of $7 \text{mm}$ across and $7 \text{mm}$ long. The target elements were Ca and Si, which are the main component materials of slate sheets.

As an example from the results, the distribution condition of Ca and Si for 41 years of use is shown in Figure 11. Further, we conducted binarization processing of the measurement results to make it easier to discriminate the results. According to the measurement results for the amount of the cement constituent, Ca, there is a clear difference between the exposed parts and non-exposed parts for test pieces in use for a greater number of years and the Ca is distributed throughout the non-exposed part. However, in the case of the exposed parts, the majority of the Ca is distributed at the bottom, in other words, the underside of the roof. A significant difference in the amount of Si in exposed and non-exposed parts was not apparent to the same degree as Ca, and it is thought that much of the Si remained between the fiber cement corrugated sheets.
4.3 Fiber cement corrugated sheet deteriorating mechanism

When we look at the Ca distribution using EPMA, the Ca amount for exposed parts is clearly reduced compared to that of non-exposed parts. This is thought to be because the Ca that exists in fiber cement corrugated sheet is eluviated by rainwater, etc. Specifically, the amount of Ca lost is greater the closer to the vicinity of the surface layer. This is because deterioration occurs early in the surface layer due to the direct impact of weathering on the surface. Also, Ca eluviation leads to weakening of the slate. This weakened part becomes gradually thinner as the surface layer is eroded due to ablation caused by flow-down of rainwater and sediment, etc. At the same time, it becomes easier for rainwater to infiltrate into the fiber cement corrugated sheet and the strength between the layers of fiber cement corrugated sheet made from several laminated layers also deteriorates due to repeated water absorption. It is thought that the material weakens due to repeated weathering during the period of use.

<table>
<thead>
<tr>
<th></th>
<th>Ca</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exposed</td>
<td><img src="image1" alt="Exposed Ca" /></td>
<td><img src="image2" alt="Exposed Si" /></td>
</tr>
<tr>
<td>Non-exposed</td>
<td><img src="image3" alt="Non-exposed Ca" /></td>
<td><img src="image4" alt="Non-exposed Si" /></td>
</tr>
</tbody>
</table>

![Figure 11 An example of the distribution condition of Ca and Si for 41 years of use](image5)

5 CONCLUSION

We obtained the following conclusions from this study.

1) We conducted a simple deterioration evaluation of fiber cement corrugated sheet using small core test specimens.
2) We defined the maximum load value obtained from the splitting compression test on the small core test specimen divided by the thickness as the splitting compression strength and clarified the tendency for splitting compression strength to decrease, the greater the number of years in use. Further, we identified the good correlation with bending strength and provided the formula for estimating the bending strength.
3) Using EPMA observation, we clarified a deteriorating mechanism in fiber cement corrugated sheet in use for a greater number of years whereby the slate is weakened by Ca eluviation from the surface and abrasion of the surface due to ablation.

REFERENCES

SERVICE LIFE PREDICTION AND EMBODY CO2 OF WOODEN BUILDINGS

S. Nakajima (1), N. Yamaguchi (2), J. Koga (3), M. Nakajima (4), I. Momohara (5), W. Ohmura (6) and S. Tomura (7)

(1) Building Research Institute, Tsukuba City – nakajima@kenken.go.jp
(2) Building Research Institute, Tsukuba City – yamaguch@kenken.go.jp
(3) National Institute for Land and Infrastructure Management, Ministry of Land, Infrastructure, Transport and Tourism, Tsukuba City – koga-j2kn@nilim.go.jp
(4) Kanto-Gakuin University – mnaka@kanto-gakuin.ac.jp
(5) Forest and Forest Products Research Institute – momohara@ffpri.affrc.go.jp
(6) Forest and Forest Products Research Institute – murasan@ffpri.affrc.go.jp
(7) Forest and Forest Products Research Institute – tomura@ffpri.affrc.go.jp

Abstract

There are many useful tools that can calculate the LCCO2 of wooden buildings. In most of these tools the embodied CO2 of wooden buildings is not precisely calculated with regard to the service life of the building elements. Buildings designed for durability will have much more environmental benefit than normally designed buildings in terms of CO2 emission. Life cycle assessment tools with engines that can precisely evaluate the service life of buildings or building elements could encourage durable design and consequently promote low carbon society.

Methodology and computer assisted calculation tool were developed to calculate both the service life and embodied CO2 of wooden buildings. The service life prediction method was developed by applying the “Factor Method”. By giving the location of the building, the types of the materials and components, the weather protection design of the building and the quality of maintenance works the tool can calculate the service life and life cycle embodied CO2 of the assessed building. The outline of the tool and the results of the case studies are reported in the paper.

1 INTRODUCTION

In Japan the service life of wooden residential buildings is reported to be approximately thirty years. This service life is extremely short compared to that of the wooden residential
buildings in Europe and North America. The durability of wooden buildings is governed by the environment of the site where the buildings exist, the durability of the materials and components, the weather protection design of the buildings and the quality of the construction works and the quality of the maintenance works.

From 1980 to 1984 a national research project was run to develop a calculation method to evaluate the service life of wooden buildings (Gihodo Shuppan Co. Ltd., 1986). This method is well known as the “Factor Method”. The ISO Standard 15686-1: Buildings and constructed assets - Service life planning - Part 2, Service life prediction procedures (ISO, 2008) was standardized referring this Factor Method. And the evaluation standard for buildings’ durability in the Japan Housing Labelling System (Ministry of Land Infrastructure and Transport, 2001) was standardized referring the service life prediction method developed in that national research project.

Recently many useful tools are proposed to calculate the embodied CO2 of buildings. However, most of these tools do not have engines that can precisely determine the service life of the buildings and the building elements. Buildings with durability will have longer service life. In addition, building with longer service life can reduce embodied CO2 in comparison to the normally designed buildings.

Buildings designed for durability have much more environmental benefit than normally designed buildings in terms of embodied CO2. Life cycle assessment tools with engines that precisely evaluate the service life of buildings and building elements could encourage durable design and consequently promote low Carbon society.

2 SERVICE LIFE EVALUATION METHOD

2.1 Backgrounds

The service life evaluation method was developed almost 30 years ago in 1984. New technologies and new knowledge for durability have been developed and found in these 30 years. Some parts of the evaluation method became old-fashioned due to the change of the lifestyle in Japan and also the new technologies. The evaluation method was updated with consideration to the latest knowledge and the newly collected data.

2.2 Outline of the updated service life evaluation method

The structure of the updated service life evaluation method is almost same to that of the former one. The outline of the calculation for the updated evaluation method is given in Figure 1. The difference between the former method and the updated method can be summarized as follows:

1) Service life was classified into 4 classes as shown in Table 1. This classification follows the classification used in the Japan Housing Labelling System.

2) The climate classes of the construction site were given for almost 900 areas in Japan. The climate class of each area was determined according to the possible fungi and termite attack.

3) Detail classification has been proposed for the service classes of the building units. Service classes of the building units represent the possibility of the units to be exposed to humidity and water.

4) Classification for the durability of building materials and components has been updated.
5) Design specification against water protection was added as a new indicator.
6) The quality of maintenance was evaluated with regard to the maintenance operated by the builders and the maintenance operated by the owner.

![Figure 1: Outline of the updated service life evaluation method.](image)

Table 1: Service life classification.

<table>
<thead>
<tr>
<th>CLASS</th>
<th>SERVICE LIFE</th>
<th>REPRESENTATIVE SERVICE LIFE</th>
<th>FACTOR</th>
</tr>
</thead>
<tbody>
<tr>
<td>3 Generation</td>
<td>90 years and more</td>
<td>90 years</td>
<td>2.7=&lt;Coef.</td>
</tr>
<tr>
<td>2 Generation</td>
<td>60 years to 90 years</td>
<td>60 years</td>
<td>1.8=&lt;Coef.&lt;2.7</td>
</tr>
<tr>
<td>1 Generation</td>
<td>30 years to 60 years</td>
<td>30 years</td>
<td>0.9=&lt;Coef.&lt;1.8</td>
</tr>
<tr>
<td>0 generation</td>
<td>Less than 30 years</td>
<td>10 years</td>
<td>0=&lt;Coef.&lt;0.9</td>
</tr>
</tbody>
</table>

2.3 Climate classes of the construction site

The climate classes of the construction sites were given for almost 900 areas in Japan following the methodologies proposed in the former system. Climate classes for wood-rotting fungi attack and climate classes for termite attack were given respectively. The climate classes for wood-rotting fungi attack were classified into 5 classes according to the summation of the duration of time (hours) that the temperature is 15 Degree Celsius or higher. The 5 classes and the corresponding factors were defined as follows:

- Class 1: Areas where duration is equal/less than 3520Hrs: Factor 1.20
- Class 2: Areas where duration is more than 3520Hrs and equal or less than 4700Hrs: Factor 1.10
- Class 3: Areas where duration is more than 4700Hrs and equal or less than 5900Hrs: Factor 1.00
- Class 4: Areas where duration is more than 5900Hrs and equal or less than 7050Hrs: Factor 0.90
- Class 5: Areas where duration is more than 7050Hrs: Factor 0.80

Class 3 represents the average area in Japan and the factor 1.00 is given for that area. The factor for the areas that have higher risk for fungi attack is less than 1.00 and the factor for the areas that have lower risk for fungi attack is higher than 1.00.
The climate classes for termites were classified into 3 classes according to the possible attack of the two major termites inhabiting in Japan. The 3 classes and the corresponding factors were defined as follows:

Class 1: Area where no termites are inhabiting: Factor 1.20
Class 2: Area where one major species are inhabiting: Factor 1.00
Class 3: Area where two major species are inhabiting: Factor 0.80

Class 2 represent the typical areas for termites’ inhabitation in Japan and the factor 1.00 is given for these areas.

2.4 Service classes of the composing units

Service classes were defined for the followings composing units of the buildings: Exterior wall, Interior wall, Floor, Roof, Wall of balcony, Floor of balcony. The service classes for the composing units were classified according to the possible exposure of the structural members to water or humidity. For example, different service classes are given for the walls and floors of the bathroom and those of the bedrooms. The factors defined for the exterior wall are given in Table 2 as an example. The factors were decided by expert judgments.

Table 2: Service class and factors for exterior wall.

<table>
<thead>
<tr>
<th>EXTERIOR ENVIRONMENT</th>
<th>ROOM TYPE</th>
<th>DOOR / WINDOW / OPENNING</th>
<th>DRAIN</th>
<th>FACTOR for the 1st FLOOR</th>
<th>FACTOR for the 2nd FLOOR</th>
</tr>
</thead>
<tbody>
<tr>
<td>DRY</td>
<td>LIVING ROOM/ BED ROOM etc.</td>
<td>NO</td>
<td>NO</td>
<td>1.00</td>
<td>1.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>NO</td>
<td>0.90</td>
<td>1.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>YES</td>
<td>0.80</td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td>WASHROOM / KITCHEN etc.</td>
<td>NO</td>
<td>NO</td>
<td>0.90</td>
<td>1.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>NO</td>
<td>0.80</td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>YES</td>
<td>0.70</td>
<td>0.90</td>
</tr>
<tr>
<td></td>
<td>BATHROOM etc.</td>
<td>NO</td>
<td>NO</td>
<td>0.70</td>
<td>0.90</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>NO</td>
<td>0.60</td>
<td>0.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>YES</td>
<td>0.50</td>
<td>0.70</td>
</tr>
<tr>
<td>WET</td>
<td>LIVING ROOM/ BED ROOM etc.</td>
<td>NO</td>
<td>NO</td>
<td>0.80</td>
<td>1.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>NO</td>
<td>0.70</td>
<td>0.90</td>
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<td></td>
<td>YES</td>
<td>YES</td>
<td>0.60</td>
<td>0.80</td>
</tr>
<tr>
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<td>WASHROOM / KITCHEN etc.</td>
<td>NO</td>
<td>NO</td>
<td>0.70</td>
<td>0.90</td>
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<tr>
<td></td>
<td></td>
<td>YES</td>
<td>NO</td>
<td>0.60</td>
<td>0.80</td>
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<tr>
<td></td>
<td></td>
<td>YES</td>
<td>YES</td>
<td>0.50</td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td>BATHROOM etc.</td>
<td>NO</td>
<td>NO</td>
<td>0.50</td>
<td>0.70</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>NO</td>
<td>0.40</td>
<td>0.60</td>
</tr>
<tr>
<td></td>
<td></td>
<td>YES</td>
<td>YES</td>
<td>0.30</td>
<td>0.50</td>
</tr>
</tbody>
</table>
Exterior wall of the living rooms etc. on the 1st floor with dry exterior environment and no opening and no drain are defined as standard exterior wall and the factor 1.00 is given for these walls. The factor is reduced according to the possible exposure to humidity and water. And 0.20 is added to the factor for the walls on the 2nd floor as they have less possibility to be exposed to humidity or water.

### 2.5 Durability of the materials and components

Durability of the materials and components is classified according to the type of materials, the species of the wooden materials and the type of the adhesive used for glulam, LVL, plywood and other sheathing materials. They are also classified according to the type of the chemical processing applied for decay prevention. The factors were defined through expert judgements and also by referring the Japan Agricultural Standard (Ministry of Agriculture, Forestry and Fisheries, 2008). For example the factors for lumber were determined as follows: The factors of the lumber were determined according to the species, wood type (sapwood / heartwood), cross section size and the level of the chemical processing. Sapwood lumbers with cross section size less than 12.0mm x 12.0mm and on-field chemical treatment were determined as lumber with normal durability and the factor for these lumbers was determined as 1.00. And extra points were added or subtracted to the factor according to the specification of the lumbers. Table 3 gives values added or subtracted to the standard factor.

Table 3: Specification of the lumbers and the points added or subtracted.

<table>
<thead>
<tr>
<th>SPECIES &amp; WOOD TYPE</th>
<th>CROSS SECTION SIZE</th>
<th>CHEMICAL TREATMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>D1 &amp; D2 Species, Sapwood:</td>
<td>C.S.&lt;12x12mm: 0.0</td>
<td>None: -0.3</td>
</tr>
<tr>
<td></td>
<td>12x12mm=&lt;C.S.&lt;13.5mm: 0.0</td>
<td>Field Application: 0.0</td>
</tr>
<tr>
<td>D2 Species, Heartwood:</td>
<td>+0.2</td>
<td>Type K2</td>
</tr>
<tr>
<td></td>
<td>13.5mm=&lt;C.S.: +0.3</td>
<td>Type K3 +0.3</td>
</tr>
<tr>
<td>D1 Normal Species, Heartwood:</td>
<td>+0.4</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Type K3 +0.6</td>
<td></td>
</tr>
</tbody>
</table>

Note: D1 Species are the species that have normal durability and D2 Species are the species that have high durability.

C.S. represents “cross section size”.

Chemical treatment K3 has higher resistance against wood decay fungi than chemical treatment K2.

### 2.6 Design for durability

Design for durability was classified in accordance with the designs applied to protect wooden structural members from water and humidity. For example high ranks were given to the design such as long projected eaves, highly ventilated under floor and attic, uncovered and ventilated structural members. High factors were given for durable design and low factors were given for non-durable design. The judgement for durable and non-durable designs were made through referring the evaluation standard for buildings’ durability in the Japan Housing Labelling System (Ministry of Land Infrastructure and Transport, 2001) and the Design and Construction Standards for Housing Insurance (Organization for Housing Warrant, 2012).
2.7 Measurement against water tightness of the facade

Measurement against water tightness was evaluated and factors were given in accordance with the evaluated results. The evaluation was made by using the check list for the mandate measurements. The mandate measurements were chosen by referring the Standards for Housing Insurance (Organization for Housing Warrant, 2012). When all measurements in the check lists were taken the factor was set as 1.0. And the factor was reduced by multiplying the value obtained by dividing the amount of the neglected measurements by the amount of the mandate measurements.

2.8 Quality of maintenance (Builders and Owners)

The quality of the maintenance works operated by the home builders and the home owners was classified into 3 classes, Class A, Class B and Class C. Class A was defined as “Maintenance operated in an adequate interval”. Class B was defined as “Maintenance operated not so frequent as Class A but in a certain interval”. And Class C was defined as “Maintenance operated only when some leaking or other damages are observed”. The adequate interval was determined by referring the Guideline for Long Service Life Houses (Ministry of Land Infrastructure and Transport, 2012).

2.9 Calculation method

The formula to calculate the factor that represents the service life is given in equation 1 and equation 2.

\[ PSP_1 = \{P \times (F_{\text{FUN}} \times D_{\text{TER}}) + B \times W\} \times D \]  

(1)

Where,

- \(PSP_1\) is the coefficient that represents the initial service life of buildings (i.e. independent to the effect of the maintenance quality),
- \(F_{\text{FUN}}\) is the coefficient that represents the inhabitation of wood-rotting fungi,
- \(D_{\text{TER}}\) is the coefficient that represents the inhabitation of termites,
- \(D\) is the coefficient that represents the service classes of the composing units,
- \(P\) is the coefficient that represents the durability of the materials and components,
- \(B\) is the coefficient that represents the design specification for durability,
- \(W\) is the coefficient that represents the quality of the design of the facade,

\[ PSP_2 = PSP_1 \times (M_{\text{EXP}} \times M_{\text{USE}}) \]  

(2)

Where,

- \(PSP_2\) is the coefficient that represents the service life of building considering the effect of the maintenance quality,
- \(PSP_1\) is the coefficient that represents the initial service life of buildings (i.e. with no regard to the effect of the maintenance quality),
- \(M_{\text{EXP}}\) is the coefficient that represents the quality of maintenance works of the builder,
- \(M_{\text{USE}}\) is the coefficient that represents the quality of maintenance works of the owner.

The service life is classified into 4 classes in accordance with the values of \(PSP_1\) and \(PSP_2\) (see Table 1).
3 CALCULATION TOOL

3.1 Outline
A tool to calculate the service life and embodied CO2 of wooden houses was developed.
The tool can be run on the computer. The outline of the tool is summarized in the following section.

3.2 Climate classes
Climate classes for the construction site can be defined by choosing the location of the site.
The factors that represent the climate classes of the sites will be automatically given.

3.3 Grids setting
In the tool one unit of the wall or one unit of the floor are expressed as one grid. Figure 2(b) and 2(c) give examples of the grid setting. The grids shown in Figure 2(b) and 2(c) represent the grids for the floor plan given in Figure 2(a). The dark grey grids represent the exterior walls, the grey grids represent the interior walls and the light grey grids represent the floor.

3.4 Service classes of the composing units
The service classes can be set for all composing units respectively. By choosing the service class and clicking the corresponding units (grids) the service classes will be given.

3.5 Durability of materials and components
By choosing the type of the materials and components the factors that represent the durability of the materials and components will be given.

3.6 Design for durability, design for the façade and quality of maintenance works
Coefficients that represent the quality of the design for durability, the quality of design of the façade against water tightness and the quality of maintenance works are given by choosing the designs and measures adopted.

3.7 Calculation for embodied CO2
The embodied CO2 is calculated by summing up the embodied CO2 of all building materials consumed in constructing and maintaining the house. The tool has a window to input the type and amount of the composing materials for every unit. The embodied CO2 for the 100 years’ service life of the house is calculated by summing up the embodied CO2 caused by the initial construction works and the refurbishment works. The embodied CO2 caused by refurbishment is calculated by summing up the embodied CO2 of the units that have come to their service life.

4 CASE STUDY
A case study was operated to evaluate the service life and embodied CO2 of a single detached house. The floor plan of the 1st story of the evaluated house is shown in figure 2(a). The estimated service life of the units of the 1st floor is given in figure 2(b) and 2(c). The service life was calculated for two cases - Case 1: With no maintenance (see Figure 2(b)) and Case 2: With maintenance (see Figure 2(c)). The specification of most of the walls and floors
was set as to be one generation in average when no maintenance was operated. For Case 1 almost all units of the house except some units of the interior walls were ranked as one generation or two generations. For Case 2 the walls and floor of the bathroom were ranked as one or two generations and the floors of the sanitary room, toilet and kitchen were ranked as two generations. Other units such as the walls and floors of the living room, dining room and Japanese room were ranked as three generations. The total embodied CO2 was calculated as 6650 (kg-C) for Case 1 and 3806 (kg-C) for Case 2. More than 40% of the total embodied CO2 could be reduced by taking measures to extend the service life of the house.

(a) Floor plan of 1st floor  (b) Case 1 (With no maintenance)  (c) Case 2 (With maintenance). Figure 2: Floor plan and the estimated service life of the case studied house.

5 CONCLUSIONS

- Methodology and computer assisted calculation tool were developed to calculate both the service life and embodied CO2 of wooden houses.
- The service life prediction method developed almost 30 years ago was revised by adapting the current technologies and knowledge.
- The embodied CO2 was calculated by summing up the embodied CO2 of all building materials consumed in constructing and maintaining the house.
- A case study was operated. The service life and embodied CO2 of the assessed house was evaluated. The results of the case study showed that when measures are taken to extend the service life of the house the service life of the house will be evaluated longer and the embodied CO2 will be evaluated less.

REFERENCES

PERFORMANCE ANALYSIS AND DURABILITY OF ENVIRONMENTAL AND ENERGY SUSTAINABILITY INTERVENTIONS ON EXISTING BUILDINGS

T. Basiricò (1), A. Cottone (1)

(1) Faculty of Engineering and Architecture, "Kore" University of Enna

Abstract
The housing built in the '70s and '80s in Italy shows significant degradation on both energy consumption and environmental sustainability, particularly those realized with load-bearing structure made of r.c. walls and slabs casted together as the coffrage-tunnel technique provides.

This technology shows high thermal dispersions due to thermal bridges and low thermal insulation of r.c. walls and slabs, due to their low thickness and great extension of concrete walls exposed to external climatic conditions.

This paper presents a study on representative buildings of this technology in order to investigate the impact of the thermal bridges on the energy consumption.

The chosen case study concerns with the Sperone district realized in the ‘70s in Italy. For these buildings energy losses and thermal conductivity of the various technical elements of building envelope were evaluated both by analytical calculations and instrumental analysis (thermography) before and after retrofitting interventions, designed to increase energy performance of the buildings according to current regulations.

The paper shows the inapplicability for the calculation of the thermal bridges detected in the couffrage-tunnel technology, of the standardized methods and databases, proposing an addition to the current legislation regarding with the specific types of thermal bridges, in order to provide designers with practical tools for their calculation.

Keywords: durability, industrialized building, energy sustainability interventions, thermal bridge

1 INTRODUCTION
The increased requirements of thermal comfort and the need for adaptation of existing buildings to the limits set by the regulations on energy savings lead to make retrofitting. These interventions often cause a percentage increase of heat losses through thermal bridges no longer be ignored in the energy analysis of buildings. Thermal bridges provoke anything
but secondary effects, despite how often it is believed. Errors in the evaluation of thermal exchanges between inside and outside can even reach 50%, in the case of buildings realized by means of fair-faced reinforced concrete load-bearing structure, considering only the transmittance of the outer walls [1].

These recurring errors also affect the durability of the technical elements. In fact non homogeneous temperature distribution causes, in certain cases, condensation of steam, with substantial risk of surface degradation due to mold, algae, lichens and stains.

Therefore it is increasingly important to define simplified methods to determine the heat flows through the linear thermal bridges or extend the tabulated values in existing databases and the UNI 14683, relating to limited recurring case studies. There are not, in fact, available in the Italian standard and databases, thermal bridges data referred to buildings realized by means of industrialized techniques such as banches et table and couffrage-tunnel. Although these construction techniques have been widely used in the 70s to build a lot of districts in different Italian contexts.

The paper investigates heat losses due to thermal bridges in buildings realized by means of these construction techniques, focusing on a case study in order to calculate the specific values of linear thermal transmittance and implement the existing databases.

2 HEAT LOSSES IN R.C. WALLS BUILDINGS

The case study is a social housing district in Palermo of the early 70s realized by means of the couffrage-tunnel industrialized system. The buildings of the district, designed just before the entry into force of the Italian laws on energy saving, a consequence of the 1973 oil crisis, do not respond, as can be seen from the energy analysis of the present state (after reported), to the current regulations. These regulations arose for the increased energy demands of indoor environmental comfort and reduction of CO\textsubscript{2} emissions.

There is, therefore, nowadays the need to adapt these buildings to current regulatory standards by providing passive interventions on the building envelope.

2.1 Typological and technological analysis

In order to evaluate the thermal losses of the buildings, it was necessary to carry out typological analysis of buildings and technological analysis of the technical elements of the envelope. The district is made up of 26 multi-storey buildings obtained from the aggregation of 3 different types of apartments "B", "C" and "D", setting "B" and "D" always on the head and "C" in the central part of the buildings (Fig. 1).

![Fig. 1: Typical floor plan of a multi storey building in the Sperone district](image-url)
From the constructive point of view, these buildings were realized with load-bearing structure made of reinforced concrete walls arranged orthogonally to the body of the building and reinforced concrete slabs casted simultaneously by means of the technology of couffragetunnel. Inevitably, this technology shows high thermal losses due to problems related to thermal bridges and reduced hygro-thermal insulation of reinforced concrete walls and slabs, due to the low thickness and large extension of concrete walls exposed to outside weather conditions.

Particularly, the transverse head facades were realized with reinforced concrete walls, 14 cm thickness, with a 2 cm polystyrene panel glued, set in the inside face, and finished with plasterboard, 1 cm thickness (RCW). The predominantly longitudinal facades were made with fair faced hollow brick walls in the outer face (12 cm), hollow bricks in the inside face (8 cm) and a 10 cm air space, finished internally with gypsum plaster (HBW). The reinforced concrete slabs, 16 cm thickness, showed a 2 mm layer of sound insulating cork, 4 cm concrete screed, a 1 cm layer of mortar and 1.3 cm tile flooring. The fixtures, originally galvanized sheet monoblocks, were replaced, in a completely arbitrary and random way, with new ones of various types (anodized aluminum, with or without insulating glass, etc.) [2].

2.2 Energy analysis of an apartment type

As a case study, among other buildings in the district, it was chosen one of the buildings with seven floors, having in the head the apartment types B and D. The energy analysis carried out analytically showed, in addition to the parameters below the values of current regulatory standards, critical points especially with regard to the heat losses through thermal bridges, being very high for this construction technology.

The heat losses by conduction, ventilation and thermal bridges were calculated for the floor type of the apartment D. The analysis showed that heat losses through the exterior walls are two times higher than the regulatory value. In order to adapt the apartment to the required regulatory standards, retrofitting interventions were provided to the walls, consisting in the external application of 4 cm acoustic plaster for r.c. walls and blowing perlite loose in the cavity between the two layers of the wall and the substitution of existing metal fixture and single glazed with other ones made in aluminum and double glazed.

For the correct assessment of the energy performance of the building the analysis of thermal bridges both before and especially after intervention cannot be ignored.

Until 2012, the current practice for the calculation of heat exchange for transmission through thermal bridges of the existing buildings was the application of a percentage increase to the heat flux, as described in the UNI TS 11300-1, in case of unreliable or low precision information project data [3].

But the need to obtain more precise data of heat losses through thermal bridges led to exclude this possibility. The UNI TS 11300-1, in fact, as amended in 2012, provides among the possible ways of computing or numerical methods in accordance with the UNI EN ISO 10211, the adoption of standard values of linear thermal transmittance $\psi$ calculated for some recurring thermal bridges and reported in the UNI EN ISO 14683 or in databases of thermal bridges complying with this standard. But these values of $\psi$ certainly do not cover the totality of existing technologies, much less those with load-bearing structure made of reinforced concrete walls and slabs of the case study. Furthermore, the values of the coefficient of linear thermal transmission reported in the UNI EN ISO 14683 are calculated for predetermined
boundary conditions and for materials and thicknesses of the various technical elements also predetermined.

There are also a series of national and international databases of thermal bridges (the “National Atlas of thermal bridges”, the New Zealand “Catalog of thermal bridges in Commercial and Multi-Family Construction”, the Switzerland “Catalogue des ponts thermiques”) that are more flexible than the UNI 14683, because they report many more case study of geometric parameters and physical and technical characteristics of materials, always setting the boundary conditions, thickness and conductivity of the insulation.

In general, the standard values have an accuracy ranging from 0% to 50%, manual calculations and databases of thermal bridges show an accuracy of ±20%, while numerical methods have an accuracy of ±5%.

For the case study, it was chosen to determine the heat losses through thermal bridges by means of numerical calculation, ensuring greater accuracy and also due to that not all types of thermal bridge of the case study are reported in the UNI and databases. In addition, for thermal bridges within the types of databases, it was necessary applying numerical calculation, being parameters set in the databases (boundary conditions, geometric parameters and physical and technical characteristics) quite different from those of the case study.

For first on the building, the discontinuity in the envelope was identified and classified as “geometric”, consisting in the corners and in the inserts between homogeneous materials, in “material” or structural, in the case where elements of high conductivity (eg. r.c. structural walls or slabs) are inserted in the technical elements of lower conductivity as the wall plugs, and “mixed”, for the presence of different materials in discontinuous geometric solutions. These discontinuities cause deviations of the one-directional heat flow (orthogonal to the surfaces of the element technical) determining thermal bridges.

The linear thermal bridges identified, reported in Fig. 2, were, therefore, divided into “geometric”, “structural” and “mixed”, and also into vertically and horizontally linear. Each of them was given a code identifying in analogy to what is reported in the UNI 14683 and various databases.

In these buildings, existing thermal bridges are characteristic of the coffrage-tunnel technology, ie. those between head concrete walls and r.c. slabs (Fn), r.c. wall and balcony (Bn), r.c. wall and cavity wall (Cn), and the one between r.c. wall and internal partition (IWn), not provided in either the UNI 14683 or databases.

While the other thermal bridges correspond, except for the thickness and the thermal characteristics, to the types F2, B2 and C2 of the UNI EN 14683.

To calculate the surface thermal transmittance of the walls and fixtures and linear thermal transmittance ψ of the identified various thermal bridges, the software Therm 7.1 and Window 7.1 of the L.B.L. of the University of California was used by means of the finite element method in accordance with the UNI EN ISO 10211. To provide the calculation of dispersions per ventilation, two air changes per hour for toilets and 0.5 air changes per hour for other rooms, were considered, resulting for the case study 1065.785 W, equal to the 15% of total thermal dispersion for the actual state and 22% after retrofitting interventions.

The boundary conditions used for thermal analysis were those of the city of Palermo (Sicily, Italy) in winter, outside temperature +5 °C, indoor temperature +20 °C, indoor relative humidity 40%, liminal external surface conductivity equal to 25 Wm⁻²K⁻¹ and the internal one equal to 7.7 Wm⁻²K⁻¹.
thicknesses of the various technical elements and the conductivity and the thermal emissivity of various materials. Furthermore for the calculation the internal dimensions were always considered, as there are no variables even after intervention.

Table 1: Thermal dispersions for conduction and through thermal bridges

<table>
<thead>
<tr>
<th>Technical elements</th>
<th>Before retrofitting</th>
<th></th>
<th>After retrofitting</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Conduction</td>
<td>Thermal bridges</td>
<td>Conduction</td>
<td>Thermal bridges</td>
</tr>
<tr>
<td></td>
<td>Watt</td>
<td>%</td>
<td>Watt</td>
<td>%</td>
</tr>
<tr>
<td>HBW</td>
<td>918,637</td>
<td>12%</td>
<td>401,794</td>
<td>5%</td>
</tr>
<tr>
<td>External RCW</td>
<td>788,812</td>
<td>11%</td>
<td>180,504</td>
<td>2%</td>
</tr>
<tr>
<td>Internal RCW</td>
<td>263,503</td>
<td>4%</td>
<td>114,986</td>
<td>2%</td>
</tr>
<tr>
<td>Fixtures</td>
<td>2763,110</td>
<td>38%</td>
<td>812,547</td>
<td>11%</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>4734,062</strong></td>
<td><strong>64%</strong></td>
<td><strong>1509,831</strong></td>
<td><strong>21%</strong></td>
</tr>
</tbody>
</table>

The software reported the trend of surface temperature, the isothermal lines and the heat flows, some of which are described below. These calculations were carried out both before and after the intervention of energy retrofitting demonstrating an low increase in the heat transmission through thermal bridges after the intervention variable, except for the thermal bridges IW and Cn for which there is a decrease, due to the realization of the outer thermal insulation by means of 4 cm acoustic plaster. Particularly in the thermal bridge Cn such
plaster, covering the protruding wall from all sides, creates with the insulation blown into the cavity of the wall, a thermal coating almost continuous.

Fig. 3. Trend of the isotherms before and after retrofitting thermal bridge in Cn

Through the visualization of the isothermal lines, it is possible to have a clear view of the phenomenon and in particular how phenomena occur at times hardly guessed. Particularly, the trend of the isotherms before and after the intervention in the thermal bridge F_n, between the r.c. wall and slab, where the effect of the insulating plaster creates a deviation of the thermal flow downward, instead to bring, as it would be expected, just the disappear of the thermal bridge with symmetrical flow to the slab, as in the present state.

In fact, the present state of the r.c. wall, as in Fig. 3, shows a thermal bridge due to the diversity of the heat exchange internal and external surfaces (for the presence of the slab), with a symmetrical trend of the heat flow with respect to the slab.

The thermographic survey carried out, shown in Fig. 4, allowed to find the location and the amplitude of thermal bridges in the joint between the the r.c. wall and the slabs.

Fig. 4. Heat flows of F_n thermal bridge (left) and thermography of transverse wall (right)
3. CONCLUSIONS

The aim of the research, namely the demonstration of the inapplicability for the calculation of the thermal bridges detected in the *coffrage-tunnel* technology, of the Atlas of the UNI 14683 and other databases on the Italian market was reached.

Comparing, in fact, the data of the linear thermal transmittance resulting from the finite element calculation, according to the UNI EN ISO 10211, the UNI EN ISO 14683 and the schedules of the Edilclima atlas [4] for the types B2, F2, C2 and C6, the obtained values, by means of numerical calculation, were almost equally to those arising from the Edilclima atlas and values ever so slightly higher, in the interest of safety, for those of the UNI 14683.

As for the types of thermal bridge typical of the buildings with r.c. walls and slabs (Bn, Fn, Cn and IWn) it was found significant difference with the Ψ data of the Edilclima atlas and with those of the UNI 14683, ranging from 0.10 to 0.45. The estimated percentage error in the case of their application is, therefore, in the range of 30-45%.

![Fig. 5. Linear thermal transmittance of the opaque walls of apartment types with different methods of calculation](image)

It is therefore proposed an addition to the UNI 14683 with these four new types of thermal bridges, in order to provide designers with practical tools for their calculation. Below are reported the details of the thermal bridges analysed in the study, using the same graphics for materials and the same data in the standard UNI consisting of the linear bidimensional coefficient of thermal coupling $L^{2D}$ and the three values of Ψ, based on internal, total internal and external dimensions ($\Psi_i$, $\Psi_{io}$, $\Psi_e$), according to the data obtained with the numerical calculation (Fig .6). The research also assessed the impact of thermal bridges before and after retrofit intervention, demonstrating that the dispersions through thermal bridges in buildings realized with reinforced concrete walls and floors are very significant in the overall transfer of heat.

Below are the results of the incidence rate of heat losses for transmission (divided into individual items) and ventilation compared to the total dispersion. By these data, the effect of
thermal bridges on total dispersion is 21% before intervention and after intervention increases up to 32%.

Fig. 6. Graphic representation of analyzed thermal bridges

Fig. 7. Percentage heat loss before and after retrofitting

REFERENCES


FREEZE-THAW RESISTANCE OF ULTRA-HIGH PERFORMANCE CONCRETE IN NORMAL AND SEVERE CONDITIONS

Dils Jeroen (1), Veerle Boel (2) and Geert De Schutter (1)

(1) Magnel Laboratory for Concrete Research, Ghent University, Belgium
(2) Industrial Technology and Construction, Ghent University, Belgium

Abstract
Ultra-high performance concrete is a new type of cementitious material. The key factors behind its production, are an optimal particle packing, a compatible combination between superplasticizer and binder, and a suitable mixing process. This leads to outstanding mechanical properties compared with vibrated concrete. As a consequence, it can be used in specific applications were a high performance is necessary. However, current test methods to examine the durability of this concrete, are too mild to capture its real potential.

Therefore freeze-thaw cycles were performed according to the Belgian standard NBN B27-009 and also in more severe testing conditions. Here, the lower limits were changed from -15°C to -20°C or -35°C and the upper limits from 15°C to 30°C, 50°C or 90°C. In addition, it was checked whether an air content reduction during the mixing process had an impact on the resistance. The splitting tensile strength was used to monitor the internal failure due to freeze-thaw cycles. For the normal limits, the specimens did not show a decrease in tensile strength even after 100 cycles. In case of the adapted limits a drop in tensile strength was generally noticed after 32 cycles. Nevertheless, an important residual strength was retained. No difference was found between the performance of specimens made under a lowered air pressure of 50 mbar and those made under atmospheric pressure. In conclusion, ultra-high performance concrete can withstand the freeze-thaw cycles according to NBN B27-009, even if the limits are taken more detrimental.
1 INTRODUCTION

During the last years, the interest and applications for ultra-high performance concrete has increased. Currently, it is used as a material to repair bridge decks, to create slender garden furniture or as substitute of steel in specific elements of oil rigs. Besides this, it has been checked if this material is suitable for the protection of important facilities such as high rise buildings and military facilities against impact forces. The possibility to use this type of concrete as protection of nuclear plants and storage facilities of nuclear waste, has also been investigated.

The main reasons for these applications is the outstanding mechanical behaviour of ultra-high performance concrete. Furthermore, the main part of the porosity is found at the nanometer scale, within the hydrates nano-structure. This lead to a very low water and oxygen permeability. As a consequence a good resistance against chloride ion penetration, attacks of aggressive acid and salts and leaching of calcium is obtained. The dense structure gives the concrete a high resistance against freeze-thaw cycles with and without de-icing salts. Several authors have reported results on the resistance against freeze-thaw cycles. Lee et al. completed ASTM C 666 on a UHPC and found that the stream treated specimens retained 90% of their relative dynamic modulus (RDM) after 1000 cycles with a strength decrease from initially 181 MPa to 170 MPa. Also Bonneau et al. performed 300 cycles and 100% of the RDM was maintained. Furthermore Graybeal et al. exposed a similar UHPC to 690 freeze-thaw cycles, leading to a relative dynamic modulus of at least 96%. Similar conclusions can be made concerning the scaling resistance of UHPC. After 112 cycles, according to the technical specification CEN/TS 12390-9, Piérard et al. obtained a total amount of scaling residue of maximum 0.27 kg/m². Cwirzen et al. reported a surface scaling of 0.5 kg/m² after 150 cycles according to the CDF test procedure (capillary suction of de-icing solutions and freeze-thaw test). Graybeal et al. tested a reactive powder concrete according to ASTM C 672 and collected 0.008 kg/m² material after 145 cycles.

This work will verify previous results. The scaling resistance was tested according to EN 1339:2003 during 277 cycles on UHPC cylinders mixed under two different air pressures. The mass of the scaling material was measured during 9 months. Furthermore, UHPC cylinders were casted and underwent 100 freeze-thaw cycles. Their internal damaged was evaluated by their splitting tensile strength. Similar as the results in literature, little damage was recorded in this work. To simulate more severe conditions, the boundaries of the freeze-thaw cycles were adapted. The lower limit was changed from -15°C to -20°C or -30°C and the higher limit was increased from 15°C to 30°C, 50°C or 90°C. As a consequence microcracks were induced in the cylinders. This diffuse damage is similar as observed after several tens of years seasonal thermal and hydraulic variations. Consequently, the rapid heating and cooling had a bad influence on the freeze-thaw resistance of UHPC which was also reported by Setzer et al.

This work comprises four different UHPC batches. Two of them were heat treated for 48 h to accelerate the hydration and to induce microcracks before the start of the tests. Two of the batches were mixed under a reduced air pressure. It was checked if the low amount of air voids lead to a lower freeze-thaw resistance for the UHPC. In conclusion, a residual splitting tensile strength of at least 7 MPa was obtained after 32 extreme freeze-thaw cycles. This excellent performance promotes the use of UHPC in countries with cold environments, even the Arctic could be one of the possibilities.
2 EXPERIMENTAL WORK

2.1 Materials and mix proportion

The composition of the ultra-high performance concrete, used in this program can be found in Table 1. The chemical composition of the cement and silica fume is given in Table 2.

Table 1 - Mix proportion UHPC

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mass/volume ratio (kg/m³)</th>
<th>Material/cement ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52.5 N HSR/LA</td>
<td>632</td>
<td>1.000</td>
</tr>
<tr>
<td>Silica Fume (SF)</td>
<td>198</td>
<td>0.314</td>
</tr>
<tr>
<td>Sand 0/0.5</td>
<td>434</td>
<td>0.687</td>
</tr>
<tr>
<td>Filler</td>
<td>159</td>
<td>0.250</td>
</tr>
<tr>
<td>Basalt 2/4</td>
<td>870</td>
<td>1.376</td>
</tr>
<tr>
<td>Superplasticizer solids (SP)</td>
<td>/</td>
<td>0.0149</td>
</tr>
<tr>
<td>Water</td>
<td>151</td>
<td>0.238</td>
</tr>
<tr>
<td>W/B</td>
<td></td>
<td>0.18</td>
</tr>
</tbody>
</table>

Table 2 – Chemical composition of cement and silica fume [%]

<table>
<thead>
<tr>
<th></th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>Na₂O</th>
<th>K₂O</th>
<th>SO₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I 52.5 HSR/LA</td>
<td>20.90</td>
<td>3.64</td>
<td>5.19</td>
<td>63.68</td>
<td>0.77</td>
<td>0.17</td>
<td>0.62</td>
<td>3.03</td>
</tr>
<tr>
<td>Silica Fume</td>
<td>94.73</td>
<td>0.36</td>
<td>0.71</td>
<td>0.20</td>
<td>0.39</td>
<td>0.20</td>
<td>0.90</td>
<td>0.27</td>
</tr>
</tbody>
</table>

The quartz sand 0/0.5 had a $d_{50}$ of 342.0 µm. This value is determined with a dry laser diffractometer, Mastersizer 2000. The CEM I 52.5N HSR/LA had a $d_{50}$ of 11.88 µm, a Blaine fineness of 4322 cm²/g and a density of 3137 kg/m³. The quartz flour had a $d_{50}$ of 13.43 µm. The silica fume was undensified and had a BET specific surface of 15.51 m²/g, an unburned carbon content of 0.46%, a $d_{50}$ of 316 nm and a density of 2217 kg/m³. The $d_{50}$ of the powders were determined with the same laser diffractometer but with a wet unit. Isopropanol was used as dispersant. Before the measurement, the powders were submitted to a sonification bath during 5 minutes.

2.2 Mixing procedure

A 5 liter intensive vacuum mixer was used to mix the ultra-high performance concrete, Figure 1. First the dry powders were mixed during 15 s. In the next 20 s, the water immediately followed by the superplasticizer were manually added to the mixture at a mixing speed of 1.6 m/s. This is followed by an intensive mixing period. The duration was determined based on the power curve, for which the agitator speed was kept constant at 6 m/s. The stabilisation time was considered to be reached when the curve had a gradient of -0,0006. The authors chose a hybrid mixing procedure, consisting of an intensive phase for 150 s at a speed of 6 m/s until the maximal power is reached and a slow phase for 120 s at a...
speed of 1.6 m/s until stabilisation. In case of vacuum, a reduction from 1013 mbar to 50 mbar was established at the moment of the intensive phase until the end of the mixing procedure.

Figure 1: A 5 liter intensive vacuum mixer with inclined mixing pan. A: the pin-agitator; B: the vacuum pump; C: mixing pan and outer protection ring.

2.3 Methods and sample preparation
2.3.1 Preparation after mixing
After mixing the samples were casted and compacted on a vibrating table of 50 Hz during 30 s. Next, they were stored for two days in a climate room with a relative humidity of 90% ± 5% and 20°C ± 2°C. After removing the moulds, all the cylinders h50/Ø110 mm and half of the cylinders h55/Ø50 mm, were again stored in the climate room until 28 days. The other half of the cylinders was heat cured during 48 h at a temperature of 90°C. The specimens were shocked from ambient temperature to 90°C, no transition period was used. Then the specimens were again stored in the climate room until 28 days.

2.3.2 Preparation freeze-thaw test
On 28 days all the cylinders h55/Ø50 mm were saturated until constant mass. One half of the cylinders were exposed to a normal temperature variation according to NBN B 05-203. The other half of the cylinders were cooled to -30°C or -20°C in a standard freezer and heated in a semi-adiabatic container up to 30°C, 50°C or 90°C. A 12 day temperature variation of the latter is shown in Figure 2. The internal damage was monitored by the evolution of the splitting tensile strength conform NBN EN 12390-6.

2.3.3 Preparation salt scaling test
The resistance of UHPC against freeze-thaw cycles in the presence of deicer salts is investigated on cylinders with dimensions h50/Ø110 mm. A total scaling surface of 0.008 m² was tested. The test was performed according to EN1339:2003. A detailed summary of the setup can also be found in Piérard et al. A 3 mm deep layer of 3% sodium chloride (NaCl)
solution was used, to simulate the most severe conditions\textsuperscript{15}. Every month the mass of scaled material from the UHPC cylinders is weighed and the solution is refreshed.

Figure 2: Temperature course extreme freeze-thaw cycles

3 RESULTS AND DISCUSSION

3.1 Characterization UHPC

The outstanding durability of UHPC is largely attributed to the reduction of the pore size and volume\textsuperscript{5,7,8}. The porosity was characterized by mercury intrusion porosimetry (MIP), air void analyses (AVA) and computed tomography (CT). The fresh air content was also determined with the pressure gauge method as described in EN 12350-7. The compressive strength of the mixture was measured on cubes with side 100 mm at 28 days conform NBN EN 12390-2. The results can be found in Table 3.

Table 3: Porosimetry and compressive strength of the UHPC mixture

<table>
<thead>
<tr>
<th>mixing pressure</th>
<th>1013 mbar</th>
<th>50 mbar</th>
</tr>
</thead>
<tbody>
<tr>
<td>curing mode</td>
<td>20°C</td>
<td>90°C</td>
</tr>
<tr>
<td>fresh air content [%]</td>
<td>2.6</td>
<td>1.0</td>
</tr>
<tr>
<td><strong>MIP - range</strong></td>
<td>100 µm – 8 nm</td>
<td></td>
</tr>
<tr>
<td>total porosity [%]</td>
<td>3.02</td>
<td>2.37</td>
</tr>
<tr>
<td>50 µm – 8 nm [%]</td>
<td>2.95</td>
<td>2.31</td>
</tr>
<tr>
<td>100 µm – 50 µm [%]</td>
<td>0.07</td>
<td>0.06</td>
</tr>
<tr>
<td><strong>AVA - range</strong></td>
<td>4 mm – 10 µm</td>
<td></td>
</tr>
<tr>
<td>total porosity [%]</td>
<td>2.39</td>
<td>2.91</td>
</tr>
<tr>
<td>distance factor [mm]</td>
<td>0.58</td>
<td>0.60</td>
</tr>
<tr>
<td><strong>CT -range</strong></td>
<td>1.5 mm – 20 µm</td>
<td></td>
</tr>
<tr>
<td>total porosity [%]</td>
<td>1.69</td>
<td>/</td>
</tr>
<tr>
<td>( f_{c,28d} ) [MPa]</td>
<td>144</td>
<td>153</td>
</tr>
</tbody>
</table>

According to the results of the mercury intrusion porosimetry, both the heat treatment as vacuum mixing lead to a clear reduction of the total porosity. The influence is more pronounced for the capillary pores between 50 µm – 8 nm. Furthermore, a very low amount of air voids was detected with this technique, nevertheless the air void analyser and the computed tomograph registered air bubbles between 4 mm and 10 µm after curing at 20°C or
90°C. This discrepancy is well known and has been described inter alia by Diamond. For this paper, the authors will only use the total porosity to explain the results of the freeze-thaw tests with and without de-icing salts. The reduction in porosity by the heat treatment was not noticed by the air void analyser, mainly because this type of curing influences primarily the capillary pores. In contrary, a similar decrease in air content was measured when the UHPC was mixed under almost vacuum. This technique decreases the specific surface of the air bubbles and increases the distance factor. This implies, that mainly some larger air bubbles remain in the UHPC. Furthermore, all the mixtures could be nominated UHPC according to the Association Française de Génie Civil, except the mixture mixed under 1013 mbar and cured at 20°C were the compressive strength was below 150 MPa.

3.2 Salt scaling resistance of UHPC

The resistance against freeze-thaw cycles with de-icing salts was monitored on six specimens. Three of them were made under atmospheric pressure, the other three were mixed under a reduced pressure of 50 mbar. The results can be seen in Figure 3, an offset of 5 cycles was used for UHPC – 50 mbar in order to show clearly the spread on the results.

Figure 3: Evolution of the scaling mass for UHPC mixed under different air pressures

After 277 cycles the average scaling mass was 0.12 kg/m² for the UHPC made under 50 mbar and 0.15 kg/m² for UHPC made under 1013 mbar. These results correspond well with those of Cwirzen et al. and Piérard et al. The results indicate that the air content reduction leads to a slightly lower surface scaling. Despite the very high distance factor, the low porosity and high compressive strength (Table 3) give the surface excellent micro-mechanical properties. Consequently, the surface is better able to confront the penetration of the cracks generated in the salt-ice (glue-spall theory). Nevertheless, further testing has to confirm this trend.
3.3 Freeze-thaw resistance of UHPC

The results of the splitting tensile tests in function of the total freeze-thaw cycles and the temperature variation are given in Table 4. The evolution of the tensile strength gives an idea on the internal damage. The tensile strength is namely the mechanical property which is addressed when stresses are built up in the concrete due to the ice expansion during the cooling step.

Table 4: Splitting tensile strength in function of freeze-thaw cycles and temperature variation

<table>
<thead>
<tr>
<th>curing mode</th>
<th>limits</th>
<th>A (15°C)</th>
<th>B (30°C)</th>
<th>C (50°C)</th>
<th>D (50°C)</th>
<th>E (50°C)</th>
<th>F (90°C)</th>
<th>G (90°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1013 mbar + 20°C</td>
<td># cycles</td>
<td>0</td>
<td>100</td>
<td>0</td>
<td>32</td>
<td>0</td>
<td>32</td>
<td>0</td>
</tr>
<tr>
<td>50 mbar + 20°C</td>
<td>f’t, [MPa]</td>
<td>13.4</td>
<td>12.8</td>
<td>13.4</td>
<td>12.3</td>
<td>14.2</td>
<td>14.2</td>
<td>15.4</td>
</tr>
<tr>
<td>1013 mbar + 90°C</td>
<td>f’t, [MPa]</td>
<td>14.5</td>
<td>14.0</td>
<td>14.5</td>
<td>14.1</td>
<td>13.8</td>
<td>14.4</td>
<td>13.1</td>
</tr>
<tr>
<td>50 mbar + 90°C</td>
<td>f’t, [MPa]</td>
<td>15.8</td>
<td>12.8</td>
<td>15.8</td>
<td>13.6</td>
<td>15.0</td>
<td>13.9</td>
<td>13.1</td>
</tr>
<tr>
<td>1013 mbar + 90°C</td>
<td>f’t, [MPa]</td>
<td>15.3</td>
<td>13.0</td>
<td>15.3</td>
<td>14.6</td>
<td>13.2</td>
<td>13.2</td>
<td>12.2</td>
</tr>
</tbody>
</table>

Not enough samples: *fct, 14cycles = 8.5 MPa; ** fct, 14cycles = 6.8 MPa; fct, 14cycles = 9.2 MPa

In general, the results of Table 4 show that the heat treated specimens have a residual splitting tensile strength not lower than 12 MPa after 100 normal cycles or 32 extreme cycles. Almost no decrease in tensile strength was noticed for the specimens cured at 20°C for the limits A, B & C. When the upper limit was changed to 50°C or 90°C the residual tensile strength began to decrease. In contrary, a drop in residual tensile strength was seen for the heat treated specimens with the limits A, B & C. Moreover, an increase was measured when the upper limits were 50°C or 90°C. The drop in residual tensile strength for 20°C limits D, E, F & G and for 90°C limits A, B & C can possibly be explained by the formation of thermal microcracks, Figure 4. Hence, more moisture uptake was possible which is a necessity for freeze-thaw damage. Consequently, this leads to a decrease in tensile strength. In case of 90°C, the cracks were formed before the start of the freeze-thaw cycles. For 20°C, the microcracks are formed during the freeze-thaw cycles by the thermal shock at 50°C or 90°C (no ramp).
At this moment, no reason could be found, why the splitting tensile strength for 90°C limits D, E, F & G increased. In further research, this phenomenon will be closely monitored as the tests are still going on. The evolution of the microstructure will also be investigated. In conclusion, UHPC has an outstanding resistance against freeze-thaw cycles with and without de-icing salt. Even when the temperature limits were changed it withheld this characteristic, especially when heat treatment was applied. This makes UHPC ideal for cold environments and applications with large temperature differences.

CONCLUSIONS

A research was conducted towards the durability of UHPC and the influence of vacuum mixing on this property. The following conclusions can be made:

- A very low porosity was measured for UHPC by mercury intrusion porosimetry, air void analysis and computed tomography.
- The low porosity lead to a high resistance against freeze-thaw cycles with de-icing salts. After 277 cycles the damage was only 0.15 kg/m².
- In case of a heat treatment, a high resistance against freeze-thaw cycles was observed. This resistance was withheld even when the temperature limits became more critical.

REFERENCES


GYPSUM EFFLORESCENCE ON CLAY BRICK MASONRY: FIELD SURVEY AND LITERATURE STUDY

Jacek Chwast (1), Jelena Todorovic (2), Hans Janssen (2) and Jan Elsen (1)

(1) KU Leuven, Department of Earth and Environmental Sciences, Celestijnenlaan 200E, 3001 Leuven, Belgium - jacek.chwast@ees.kuleuven.be; jan.elsen@ees.kuleuven.be

(2) KU Leuven, Department of Civil Engineering, Kasteelpark Arenberg 40, 3001 Leuven, Belgium - jelena.todorovic@bwk.kuleuven.be; hans.janssen@bwk.kuleuven.be

Abstract
Persistent efflorescence on masonry facades is a growing problem, and as an initial stage in a larger research project, this paper presents a field survey and literature review on the topic. In the field survey, 28 cases of persistent efflorescence in Belgium were investigated. In most cases, the deposit was identified as gypsum, explaining the persistence of the efflorescence. It appears that the gypsum originates from within the masonry, and is transported to the surface via moisture transfer. In the literature review, plausible formation and transport mechanisms for gypsum in masonry are identified: gypsum can be derived from the brick and the mortar, and can be transported via pore water advection and diffusion. On the other hand, the crystalisation of gypsum on the surface and the only recent occurrence of gypsum efflorescence can, for now, not be soundly explained. One hypothesis is the recent application of mortar additives, which may affect the formation, transport and crystallisation behaviour of gypsum. The final question marks ultimately make up the research questions for the larger research project.

Keywords: efflorescence, gypsum, clay brick

1 INTRODUCTION

Salt efflorescence, the growth of salt crystals on a surface caused by evaporation of salt-laden water, is a typical surface phenomenon commonly observed on brick, mortar and concrete facades. Mostly, it concerns whitish deposits of water soluble salts, like alkali sulphates or chlorides, which generally appear soon after erection of the facade. Due to their high solubility, these salts are easily brought to the facade’s surface via moisture flow, and they are similarly easily washed away from the surface by natural weathering. Since the 1980’s however, a grey-white efflorescence has started appearing on a number of clay brick masonry facades, in the UK, in the Netherlands as well as in Belgium, with a particular efflorescence rich in gypsum [1, 2]. This specific efflorescence type usually appears a couple of years after construction. The main efflorescence constituent is lowly soluble gypsum, what explains its persistence against natural weathering. The field survey and literature review presented in this paper are motivated by the progressively growing number of gypsum efflorescence (GE) cases reported in Belgium.
From their investigations of ten GE cases in the UK, Bowler and Winter [1] deduced a number of crucial observations. Firstly, it is a recent phenomenon, occurring only since the 1980’s, solely affecting newly erected facades. Moreover, this staining is commonly perceived only after a number of years, contrary to the earlier efflorescence of more soluble salts. Gypsum efflorescence furthermore mainly affects facades with a high wind-driven rain load, and these facades often comprise cavity insulation. There is no clear relation with the brick type however, as GE is observed on bricks with both low and high sulphate contents.

Although calcium sulphate is one of the major salts present in building materials, it is commonly believed that, due to its very low solubility and inert nature with respect to changes in RH and temperature, its presence in bricks does not lead to any damage risk [3]. These are the primary reasons why calcium sulphate has remained under the radar. Resultantly, even though GE has been occurring for some decades, the physico-chemical processes behind this phenomenon are still unclear. This paper therefore has a double objective. The reported field survey establishes the occurrence of GE in Belgium and confirms and widens the earlier observations by Bowler and Winter [1] and Brocken and Nijland [2]. The presented literature review gathers the current knowledge in relation to GE, and formulates the main research questions to be tackled in order to come to further insight in this phenomenon.

2 FIELD SURVEY ON PERSISTENT EFFLORESCENCE

Persistent efflorescence is a growing problem in the construction industry. Besides the occurrence in the UK and the Netherlands [1, 2], the Belgian brick producers are receiving progressively more complaints. In order to contribute to the knowledge on this issue, 28 cases of Belgian buildings affected by persistent efflorescence are analysed. Some of these cases were identified from the complaints of building owners to brick producers, other cases were found during an exploration of the Leuven area in Belgium by the researchers.

2.1 General observations on persistent efflorescence

The investigated cases concern a type of permanent efflorescence, which does not wash off with natural weathering. In two cases the building facades had been cleaned with high-pressure water jets, but in both cases efflorescence has reappeared, indicating that the efflorescence source is not affected by the treatment. In contrast to early efflorescences of well soluble salts, often being soft and thick, these persistent efflorescences give the impression of being very compact and strongly adhering to the brick surface, while simultaneously generally being very thin. In most cases the efflorescence affects the bricks to a much greater extent than the mortar joints.

The building facades are all constructed with common Belgian ‘hand’ moulded clay bricks. In all cases, the most affected facades of the building are those oriented West to South, with efflorescence being most pronounced on the edges and the upper parts of the façade, façade orientations and locations that typically receive relatively much wind-driven rain. The year of construction is known for 15 of the 28 cases, and ranges from 1997 to 2007. The other cases have an equally modern appearance, and the problem thus appears to exclusively affect buildings erected during approximately the last two decades. Based on observations by building owners, the efflorescence is perceived only several years after construction. However, it is not clear whether these stains indeed develop with a delay, or it is just a very slow process that goes unnoticed in its early stages.
2.2 Composition analysis of persistent efflorescence

To determine the efflorescence composition, efflorescence samples are collected and analysed, revealing four primary groups in the studied persistent efflorescence cases.

The collected samples are initially gently ground and then sieved through a 63µm sieve. The efflorescence minerals are identified via the powder X-ray diffraction (XRD) method. The diffraction patterns are gathered with a Philips PW1830 diffractometer using CuKα radiation (45kV, 30mA). The standard 2θ scan range is 5-70° with a step size of 0.02° 2θ and a counting time of 1s.

Table 1 gathers the results of sample analyses and case evaluations. Out of the four identified minerals – gypsum, calcite, hematite and quartz – only the former two are potential persistent efflorescence components. In most cases where gypsum is identified in a sample, it is present in a substantial amount compared to the other identified minerals. As it is not a raw brick component, it is clear that its accumulation at the surface of masonry is due to the efflorescence formation. Unlike for gypsum, the origin of calcite is uncertain, since besides forming efflorescence it can also be a raw brick component. In most cases where calcite is identified in a sample, it is present in very low amounts, and its origin can therefore not be unambiguously determined. Based on the analysis of the persistent efflorescence composition and the field survey observations, the 28 analysed cases can be divided into four groups: gypsum (G, 13 cases), calcite (C, 1 case), mixed (G+C, 5 cases), and ambiguous (A, 9 cases).

Table 1: Evaluation of the analysed persistent efflorescence field study (FS) cases. Relative content of gypsum (G) and calcite (C) is approximated: +++ dominantly present, ++ present, + present in low amounts, ? possibly present, - not identified.

<table>
<thead>
<tr>
<th>Case n°</th>
<th>XRD group</th>
<th>Case n°</th>
<th>XRD group</th>
<th>Case n°</th>
<th>XRD group</th>
</tr>
</thead>
<tbody>
<tr>
<td>FS01</td>
<td>+++</td>
<td>G</td>
<td>FS17</td>
<td>-</td>
<td>A</td>
</tr>
<tr>
<td>FS02</td>
<td>-</td>
<td>A</td>
<td>FS18</td>
<td>+++</td>
<td>G</td>
</tr>
<tr>
<td>FS03</td>
<td>+</td>
<td>A</td>
<td>FS19</td>
<td>+++</td>
<td>G</td>
</tr>
<tr>
<td>FS07</td>
<td>-</td>
<td>A</td>
<td>FS20</td>
<td>+++</td>
<td>G</td>
</tr>
<tr>
<td>FS08</td>
<td>+++</td>
<td>G</td>
<td>FS21</td>
<td>+++</td>
<td>?</td>
</tr>
<tr>
<td>FS10</td>
<td>+++</td>
<td>?</td>
<td>FS22</td>
<td>-</td>
<td>+</td>
</tr>
<tr>
<td>FS11</td>
<td>+</td>
<td>A</td>
<td>FS23</td>
<td>+++</td>
<td>+</td>
</tr>
<tr>
<td>FS13</td>
<td>-</td>
<td>+</td>
<td>FS24</td>
<td>+++</td>
<td>G</td>
</tr>
<tr>
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<td>FS25</td>
<td>-</td>
<td>+</td>
</tr>
<tr>
<td>FS16</td>
<td>+++</td>
<td>G+C</td>
<td>FS26</td>
<td>+++</td>
<td>-</td>
</tr>
<tr>
<td>FS27</td>
<td>+++</td>
<td>+</td>
<td>FS28</td>
<td>-</td>
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<tr>
<td>FS29</td>
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<td>FS33</td>
<td>+</td>
<td>+</td>
<td>FS34</td>
<td>+</td>
<td>++</td>
</tr>
</tbody>
</table>

The persistent character of the analysed efflorescence cases can often be attributed to lowly soluble gypsum being its primary component. This agrees with the findings reported for the UK and the Netherlands, like most of the other outcomes of our field survey. The grey-white appearance precludes interaction with polluted air, and the role of moisture transfer suggests that gypsum components are probably derived from the brick masonry itself.
2.3 Brick properties and persistent efflorescence

In many cases the applied brick type is identified, which permits analysis of the relation between brick properties and persistent efflorescence. However, it was not possible to acquire data for the exact batches of bricks used for construction of the investigated buildings. We have thus used the available technical specifications declared by brick producers. The cold water absorption (CWA) and initial rate of absorption (IRA) are represented in Figure 1, and grouped along the efflorescence composition found for these 15 cases. The CWA and IRA values cover the range commonly found for this brick type. It therefore appears that the exposure to wind-driven rain, rather than the moisture transfer properties themselves, is a dominant factor. Bricks may contain calcium sulphate in the form of anhydrite, which potentially can be a GE source. For some of the identified brick types data regarding the content of Na, K, Mg and SO₄ ions is available, as declared by the brick producers based on EN 772-5 (Table 2). Those data are used here to estimate the amount of gypsum which can potentially be derived from anhydrite dissolution. Table 2 shows that there is no strong link between gypsum amounts (derived from anhydrite) and efflorescence composition.

Table 2: Chemical characteristics of the identified brick types.

<table>
<thead>
<tr>
<th>Case n°</th>
<th>group</th>
<th>ion content [wt%]</th>
<th>gypsum/brick [g]</th>
<th>Case n°</th>
<th>group</th>
<th>ion content [wt%]</th>
<th>gypsum/brick [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>FS07</td>
<td>A</td>
<td>0.014 0.003 0.002 0.001</td>
<td>0.19</td>
<td>FS28</td>
<td>G+C</td>
<td>0.003 0.001 0.000 0.000</td>
<td>0</td>
</tr>
<tr>
<td>FS10</td>
<td>G</td>
<td>0.248 0.001 0.001 0.000</td>
<td>7.16</td>
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<td>G</td>
<td>0.044 0.001 0.000 0.001</td>
<td>1.16</td>
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<td>A</td>
<td>0.172 0.001 0.001 0.000</td>
<td>4.91</td>
<td>FS34</td>
<td>G+C</td>
<td>0.014 0.003 0.002 0.001</td>
<td>0.19</td>
</tr>
<tr>
<td>FS13</td>
<td>A</td>
<td>0.056 0.001 0.001 0.001</td>
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<td>FS18</td>
<td>G</td>
<td>0.020 0.002 0.001 0.003</td>
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<tr>
<td>FS14</td>
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<td>0.014 0.003 0.002 0.001</td>
<td>0.19</td>
<td>FS19</td>
<td>G</td>
<td>0.021 - - -</td>
<td>-</td>
</tr>
</tbody>
</table>

3 LITERATURE REVIEW ON GYPSUM EFFLORESCENCE

The field study has established the occurrence of gypsum efflorescence on brick masonry in Belgium, thus confirming and widening the earlier observations in the UK and the Netherlands [1, 2]. As GE has only come to the forefront in the last few decades, whereas brick masonry has been in use for many centuries, there are several open questions on the physico-chemical processes underlying this phenomenon. And even while the literature specifically dedicated to GE is relatively limited, there are a number of studies from various other fields which can contribute to the understanding of GE. These primarily relate to calcium sulphate sources, transport and crystallisation.
3.1 Calcium sulphate sources

Gypsum efflorescence at the surface of brick masonry requires a source of calcium sulphate in the masonry. The review below corroborates that calcium sulphate can be derived from ceramic brick and/or cement mortar. Calcium sulphate occurs in three distinct mineral forms: gypsum, bassanite and anhydrite, which are respectively the dihydrate (CaSO₄·2H₂O), hemihydrate (CaSO₄·0.5H₂O) and anhydrous (CaSO₄) forms of calcium sulphate. Both gypsum and anhydrite can be present in masonry, and it is hence important to distinguish between these two mineral forms.

Calcium sulphate in ceramic bricks may originate from the initial calcium sulphate in the clay mix, or may stem high-temperature reactions taking place during brick burning [4]. Independent of the reaction path, the final calcium sulphate mineral form present in the ceramic brick is anhydrite, given the high burning temperatures.

Calcium sulphate is present in most Portland cements, as it is deliberately added to control the setting properties. Upon addition of water, the added calcium sulphate reacts with tricalcium aluminate (C₃A): initially ettringite is formed, which then reacts to monosulphate, both being virtually insoluble. Synthetic ettringite does however undergo carbonation when exposed to CO₂, decomposing back to gypsum and other minerals [5]. Monosulphate is even less stable than ettringite, and hence more prone for decomposition. Carbonated mortar is therefore a potential source of calcium sulphate, a hypothesis initially proposed by Brocken and Nijland [2, 6].

3.2 Calcium sulphate transport

Apart from internal calcium sulphate sources, GE moreover necessitates transport of calcium sulphate through the brick and mortar towards the surface. This section corroborates the possibilities for dissolution, diffusion and advection of calcium sulphate in masonry.

Contrary to other salts typically present in masonry, gypsum and anhydrite are fairly lowly soluble at respectively 0.015 mol/kg and 0.019 mol/kg at 25 °C, though the dissolution rate of anhydrite is roughly two orders of magnitude smaller than that of gypsum [7].

Generally, salts in porous materials can be transported when they are dissolved in water, by advection or/and diffusion. The former represents the bulk transport of water, carrying the salt ions with it, whereas the latter is the movement of ions within the water, driven by the salt concentration gradient. Drying experiments on brick samples fed at the bottom surface with a gypsum solution demonstrate this diffusive and advective gypsum transport [8]. After a few days, substantial reductions in the evaporation rates were noted, although no salts appeared on the drying surface. Further analysis confirmed gypsum crystallization just below the surface. This corroborates that even lowly soluble salts, like calcium sulphate, are substantially transported by diffusion and advection.

3.3 Calcium sulphate crystallization

While calcium sulphate is transported by diffusion and advection, similar to other more highly soluble salts, its crystallisation behaviour is however somewhat more particular.

Typically, drying at the surface is stated to result in crystallisation at the surface (efflorescence), while a drying front below the surface is said to cause crystallisation within the material (subflorescence). This would lead one to expect that the same experimental conditions should result in the same crystallisation locations for different salts. The reverse is often observed in actual salts damage cases though, and this observation is further upheld by
laboratory experiments demonstrating salt-specific crystallisation behaviour under well-defined laboratory conditions. For instance, Cardell et al. [9] reported that efflorescence develops on limestone samples immersed in various salts solutions and their mixtures, but not for the gypsum solution for which only a limited subflorescence was identified. Based on field measurements, Charola et al. [3] similarly concluded that gypsum tends to accumulate just below the surface for non-calcareous materials. It remains to be seen though whether this is also valid for the typical alternating wetting and drying of facade masonry.

3.4 Discussion

Both brick and carbonated mortar may hence be the source of GE, respectively in form of anhydrite or gypsum. For the former, anhydrite as the source of GE is questionable, since it is characterized by an exceptionally low dissolution rate. The carbonation of mortar, on the other hand, does not seem to be a limiting factor and actually supports the observed delay in the GE. Gypsum efflorescence then proceeds through the processes of dissolution inside the masonry and transport to the surface of the masonry, the former induced by moisture supplied by wind-driven rain, the latter resulting from moisture drainage by surface drying.

Up to this point, the available knowledge offers some first insights into the physico-chemical processes underlying GE. The tendency of gypsum to crystallise under the surface does form a first crack in the reasoning chain though. The general validity of such limited data can however be questioned. A second – and more important – blow to the formulated argumentation is that both the sources and the transports have been active in ceramic brick and Portland cement based masonry since its original application, which does not allow explaining the only recent occurrence of GE.

4 RECENT CHANGES

The argumentation put forward above falls short on two fronts: it does not clarify the surface crystallisation of gypsum, nor does it explain the recent nature of GE. Gypsum efflorescence has sprung up during the last three decades, what apparently coincides with a number of major changes in material composition and construction technology. These mainly concern the chemical composition of cements and the use of mortar admixtures.

4.1 Brick

To the authors’ knowledge, the brick production technology in Belgium (and Europe) has not experienced significant changes in the last few decades. Bowler and Winter [1, 10] do indeed state a similar remark in relation to the UK.

4.2 Mortar

Masonry mortar composition, on the other hand, has evolved substantially over the last three decades, both in Belgium and in the UK. In the UK cement/lime mortars have been gradually replaced by cement/air entrainer mixes, the decrease in lime content might facilitate the release of sulphates due to ettringite carbonation [2, 6]. In Belgium cement-based mortar has come into use in the 1950’s, thus mortar carbonation cannot solely explain the recent occurrences of GE in Belgium.

Bowler and Winter [10] conclude that the widespread occurrence of GE in the UK coincides with increase and decrease of respectively permitted levels of sulphates and C3A in UK cements. They hypothesize that, all in all, these changes might have led to a higher
availability of sulphates in fresh mortar, which in turn could finally crystallise as the persistent efflorescence. In Belgium the permitted sulphates levels have been fluctuating over the last few decades and there have been no constraints imposed on ordinary cement types by national standards.

Bowler and Winter [1, 10] thus considered the use of surfactants as one of the potential triggers in GE. They report that the first GE occurrences coincide with the introduction of surfactant-based mortar admixtures, such as dedicated air entrainer and plasticizer products but also domestic detergents, and similar is observed in Belgium. Bowler and Winter [10] investigated the effect of various admixtures on the standard brick efflorescence test. They reported that GE developed only on the admixture-treated samples and that gypsum could only have been derived from anhydrite-reach Fletton brick samples. The surfactants present in the tested admixtures can enhance GE formation in a number of ways: by increasing pore solution mobility [10], reducing the liquid-gas interface energy, modifying crystal growth and potentially affecting the anhydrite dissolution. The application of admixtures might therefore possibly explain the recent occurrence of GE. However, it can be debated whether surfactants can exert a long-term effect in masonry.

4.3 Use of insulation

Besides these internal changes in material composition and production, over the last few decades, modifications in the wall composition form an important external change. As reported by Bowler and Sharp [11], the use of thermal insulation inside the cavity of masonry walls has significantly increased. It is assumed that walls become colder and wetter, hence potentially triggering GE. However, the ways in which this may affect GE, via impacts on the gypsum formation, transport and/or crystallization, remain unknown.

5 RESEARCH QUESTIONS ON GYPSUM EFFLORESCENCE

In response to the growing number of persistent masonry stainings in Belgium we report on the field survey results. The persistent character, presence of gypsum, delayed development and recent occurrence are in line with the previously reported British [1] and Dutch [2] cases. The observations point at an important role of wind-driven rain and the source of GE being masonry itself. At the same time the occurrence of GE does not appear related to the physico-chemical properties of the brick. And while research dedicated to GE in particular is limited, a literature review of publications related to gypsum sources, transport and crystallisation has offered a number of insights. Both brick and mortar can be the source of calcium sulphates, via respectively the dissolution of anhydrite or the carbonation of ettringite. And despite their low solubility, calcium sulphates can be dissolved in the pore water, and transported via diffusion and advection processes.

The literature review did, however, also reveal some bottle necks. Calcium sulphate has a tendency for crystallisation below the surface, leading to subflorescence instead of efflorescence. Moreover, all the mechanisms related to sources, transport and crystallisation are equally active in older cement-based masonry, contrary to the more recent nature of the GE problem. In the last step hence, the recent changes experienced by masonry have been assessed. These mainly relate to the composition of binders, which might have caused the increased sulphate availability, and the application of admixtures, where use of surfactants has become common practice.
Nonetheless, many of the inferences articulated above remain hypothetical, which leads to the formulation of a number of research questions in relation to GE. The first two relate to the calcium sulphate sources in masonry:

1. Is gypsum formed by carbonation of ettringite inside the mortar joint?
2. Is GE formed by mobilization of anhydrite from the brick?

The next concerns calcium sulphate crystallisation:

3. Can surface crystallisation be obtained by standard atmospheric excitation?

In relation to the recent occurrence, two more research questions are:

4. What is the impact of mortar admixtures on the calcium sulphate sources, transport and crystallisation?
5. What is the effect of cavity insulation on the calcium sulphate sources, transport and crystallisation?

Further experimental investigations are needed to elucidate gypsum crystallisation behaviour under representative conditions. This will be a starting point for assessing the masonry components as a GE source and investigating the effect of admixtures.

6  ACKNOWLEDGEMENTS

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7 REFERENCES

DECAY SUSCEPTIBILITY OF RAIN EXPOSED WOOD JOINTS – COMPARISON OF TWO DECAY MODELS

Maria Fredriksson (1), Lars Wadsö (2) and Peter Johansson (3)

(1) Division of Building Materials, Lund University, Lund –maria.fredriksson@byggtek.lth.se
(2) Division of Building Materials, Lund University, Lund – lars.wadso@byggtek.lth.se
(3) Division of Building Materials, Lund University, Lund – peter.johansson@byggtek.lth.se

Abstract
Wood is susceptible to decay if it is exposed to high moisture contents and certain temperatures during long periods of time. In rain exposed structures, decay often starts close to joints since the joints can act as water traps where water stays after rain events. Service life prediction models for outdoor wood structures require knowledge about the moisture and temperature conditions in the wood as well as models that describe the degradation under these conditions. Recently, several models that describe decay of wood structures have been presented. In the present study, the results from two of these decay models from literature were compared. Moisture content data from a previous study where moisture content profiles were monitored in joints of different designs were used as input data in the models. In general, both models identified the same points as critical points in terms of high decay susceptibility. However, the models gave somewhat different results in points where drying occurred between the watering periods.

1 INTRODUCTION

When designing a structure it is desirable to know which service life to expect when using certain materials or designs. The service life of wood structures outdoors is mainly limited by degradation by decay fungi. Such degradation generally occurs when the moisture content of the wood is high and it is therefore important to limit the duration of such periods. In rain exposed wood structures, such as wooden claddings, decking, fences etc., decay often starts close to joints, see e.g. [1, 2], since the joint acts as a water trap where water stays after rain events. This is well-known since specimens that include a joint of some sort commonly is used to accelerate outdoor field tests (e.g. [3]). The design of joints is therefore important in order to prolong the service life of rain exposed wood structures.

Since degradation due to decay fungi influences the service life of outdoor wood structures, knowledge concerning the decay fungi and how the degradation depends on moisture content and temperature is one of the factors needed in order to estimate the service life of a wood structure outdoors. Recently, several models that describe wood degradation due to decay fungi have been developed. Viitanen et al. [4] presented a model for growth of decay fungi based on laboratory measurements. Brischke and Rapp [5] developed a model relating the material climate (wood moisture content and temperature) to decay progression.
based on data from field tests from several test sites around Europe. Data from these field tests was also used by Isaksson et al. [6] who suggested and evaluated different decay models. A model similar to one of the models presented in Isaksson et al. [6] is used in an engineering design guideline [7, 8], but here decay occur for all moisture contents, i.e. also for very low moisture contents.

In the present paper, the results from two decay models from literature were compared. The two decay models used were the two step dose response model presented by Isaksson et al. [6] and the model presented by Viitanen et al. [4]. The models were applied on moisture content data from a previous study [9] where joints of different designs were exposed to artificial rain in the laboratory.

2 MATERIALS AND METHODS

2.1 Moisture content measurements – experimental set-up

Moisture content measurements were performed in three types of joints with principal design as follows: end-grain surface facing end grain surface, end grain surface facing side grain surface and side grain surface facing side grain surface. Schematic illustrations of these three joint types are shown in Table 1. The joints were made of fast grown and slow grown Norway spruce (Picea abies (L.) Karst.) and heartwood and sapwood were separated. The mean density was 392 kg/m³ (standard deviation: 26 kg/m³) for the slow grown wood and 376 kg/m³ (standard deviation: 45 kg/m³) for the fast grown wood. Which material that was used for each joint type is shown in Table 1; slow grown sapwood was used for all joints.

The moisture content was measured at different positions close to the joints using the moisture content sensors described by Fredriksson et al. [10]. The points of measurements for the three joints are shown in Table 1. For each joint type and each material, three specimens were made. The gap between the boards in these three specimens was set to 0 mm, 2 mm and 5 mm respectively. This was done to create different microclimates, i.e. duration of water in the gap between the boards, for the three specimens. One water spraying gun was mounted above each specimen. Water was applied 1 hour each day during five days. The climate in the room before and after the wetting periods was 65% RH/20 °C. More details concerning the experimental set-up and the material is given by Fredriksson [9].
Table 1. Schematic illustrations of the three joint types that were used (A, B and C) and which materials and gap sizes that were used for each joint as well as the positions in which the moisture content was measured. In each board, measurements were performed at three depths from the water exposed surface: 3 mm, 11 mm and 19 mm.

<table>
<thead>
<tr>
<th>Joint</th>
<th>Material</th>
<th>Gap</th>
<th>Points of measurement</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>end grain surface - end grain surface</td>
<td>slow grown sapwood (d = 0, 2 ) and 5 mm</td>
<td>![Diagram A]</td>
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<tr>
<td></td>
<td></td>
<td>fast grown sapwood (d = 0, 2 ) and 5 mm</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>slow grown heartwood (d = 0, 2 ) and 5 mm</td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>end grain surface - side grain surface</td>
<td>slow grown sapwood (d = 0, 2 ) and 5 mm</td>
<td>![Diagram B]</td>
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<tr>
<td></td>
<td></td>
<td>fast grown sapwood (d = 0, 2 ) and 5 mm</td>
<td></td>
</tr>
<tr>
<td>C</td>
<td>side grain surface - side grain surface</td>
<td>slow grown sapwood (d = 0, 2 ) and 5 mm</td>
<td>![Diagram C]</td>
</tr>
</tbody>
</table>

2.2 Evaluation of decay susceptibility

2.2.1 Input data

Since the moisture content measurements were performed during a limited period of time which was too short to induce decay, the moisture content data was cycled to represent a longer period of time. Seven days of measured moisture content data for each point of measurement were used. This period with measured data included five watering periods of 1 hour the first five days (see section 2.1). A dry period of ten days, during which the moisture content was set to a constant value of 15% was then added to the measured data. A period of 17 days including seven days of measured data and a dry period of ten days was thus created. This period of 17 days was then repeated until a total period of 5 five years was reached. This five year period was then used as input in the two decay models.

2.2.2 Determination of decay

Two decay models from literature were used to compare the decay susceptibility of the different joints: the two step dose-response model in Isaksson et al. [6] and the model presented by Viitanen et al. [4]. Since the models were applied on data from laboratory tests, where the temperature was held constant (20 °C), the decay calculations in the present study mainly take into account the influence of fluctuations in moisture content.

In the two step dose-response model in Isaksson et al. [6] two doses, a moisture induced \(D_u\) and a temperature induced dose \(D_T\), are calculated and from this a total daily dose is determined. The total daily dose is determined by multiplying \(D_u\) and \(D_T\) and can be further
used to determine the expected decay rating according to standard EN 252 [11] where rating 1 means sound and 4 means failure. Full information concerning this model and the equations used in found in [6]. Since the temperature in the present laboratory experiments was constant, the temperature induced dose was constant in the present case.

In the model by Viitanen et al. [4] decay is modelled as two processes: an activation process and a mass loss process. Mass loss only occurs when the RH exceeds 95% and the temperature exceeds 0 °C. For details concerning this model, see [4]. Since the input in the model by Viitanen et al. [4] is relative humidity (RH), the measured moisture content needs to be transformed into RH. This was done by using a curve fit to the sorption isotherm data in the Wood handbook [12]. Above this range, the relative humidity was determined from a curve fitted to data from pressure plate experiments presented by Fredriksson et al. [10]. This is an approximate calculation of RH since the moisture content is different for desorption and absorption at the same RH level. The hysteresis between absorption and desorption should therefore be included in order to determine the RH more precisely. However, for the present case this rough estimation of RH was chosen because of the lack of absorption isotherm data close to 100% RH which is the relevant range in this case since decay according to the model only occur above 95% RH.

The output from the model by Viitanen et al. [4] was mass loss in % and the output from the two step dose response model was decay rating. Therefore, relative values were determined in order to enable comparison of the results. All calculated decay ratings/mass losses were therefore divided by the decay rating/mass loss in a reference point. The reference point was in the joint A specimen of slow grown sapwood (d = 0 mm) 5 mm from the end grain surface at 11 mm depth. The reference point is marked in Fig. 1 and 2. This point was chosen as a reference point since the highest decay rating/mass loss was reached here with both models.

3 RESULTS AND DISCUSSION

The results from the calculations of the relative decay ratings for the two step dose response model are shown in Fig. 1 and 3 and the relative mass losses calculated by the model by Viitanen et al. [4] are shown in Fig. 2 and 4.

Relative decay rating using the two-step dose response model (Isaksson et al. 2013)

Figure 1. Relative decay rating for the joint A specimens determined using the two step dose response model by Isaksson et al. [6]. The rectangle marks the point of measurement which was used as a reference point, see section 2.2.2.
Figure 2. The relative mass loss for the joint A specimens determined using the model by Viitanen et al. (2010). The rectangle marks the point of measurement which was used as a reference point, see section 2.2.2.

<table>
<thead>
<tr>
<th>Slow grown sapwood</th>
<th>Fast grown sapwood</th>
<th>Slow grown heartwood</th>
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<tbody>
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<td>$d = 0 \text{ mm}$</td>
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<td>$d = 0 \text{ mm}$</td>
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<td>0.6</td>
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<th>$d = 5 \text{ mm}$</th>
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Figure 3. Relative decay rating for the joint B and C specimens determined by the two step dose response model by Isaksson et al. [6]. The circle marks the point of measurement for which data is shown in Fig. 5.
Figure 4. Relative mass loss for the joint B and C specimens determined by the model in Viitanen et al. [4]. The circle marks the point of measurement for which data is shown in Fig. 5.

The models gave basically the same results when it comes to identify critical points, i.e. which points of measurements that were most and least subjected to decay. For example, compare Fig. 3 and 4. Here, both models predict no decay in the horizontal board when the gap size is 5 mm. However, when the gap size is 0 or 2 mm, both models predict decay in the upper part of the horizontal board.

There are also cases where the two models give different results. One example of this is the circled point of measurement in Fig. 3 and 4 in the horizontal board of joint B of fast grown sapwood. Moisture data from this point of measurement is shown in Fig. 5. As seen in Fig. 5a, drying occurred between the watering periods and this is also why the results from the two models differ. The RH used as input in the model by Viitanen et al. [4] fluctuated between values above and below 95% RH (Fig. 5b), i.e. above and below the limit for mass loss in the model. However, in the two step dose response model, these fluctuations are not taken into account since the daily average values are used as input (Fig. 5c). The limit for decay in the two step dose response model is 25% moisture content, and the daily average moisture content is above this limit during the first seven days (Fig. 5c). Therefore, this model predicts...
more severe decay than the model by Viitanen et al. [4] in this case. The resolution of the measured data is therefore taken into account in a better way in the model by Viitanen et al. [4]. However, since moisture content is the parameter that usually is measured in wood structures, it is a disadvantage that the model by Viitanen et al. [4] use RH as input data. The measured moisture content therefore has to be transformed into RH which, as discussed in section 2.2.2, is not trivial under fluctuating moisture conditions especially not for high RH levels, i.e. the RH levels of interest in this case.

Figure 5. a. An example of measured moisture content and dry period data for a point of measurement in the horizontal board of joint B (this point of measurement is marked with a circle in Fig. 3 and 4.) b. Corresponding input data to the two step dose response model. c. Corresponding input data to the model by Viitanen et al. [4].

4 CONCLUSIONS

 Despite of the differences between the two models, they generally identified the same points as critical in terms of decay susceptibility. However, in positions in the joint where drying occurred after the rain events, the drying phase was not taken into account by the two step response model since daily average values are used as input. Therefore, in such positions, the two step dose-response model predict more severe decay than the model by Viitanen et al. [4]. However, a disadvantage with the model by Viitanen et al. [4] is that the moisture content data needs to be transformed into RH which is not trivial under fluctuating moisture contents since there is a hysteresis between absorption and desorption isotherm.

ACKNOWLEDGEMENTS

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THE EFFICIENCY OF AUTONOMOUS CRACK HEALING OF MORTAR IN CHLORIDE SOLUTIONS.

M. Maes (1) and N. De Belie (1)

(1) Magnel Laboratory for Concrete Research, Department of Structural Engineering, Ghent University, Technologiepark Zwijnaarde 904, B-9052 Ghent, Belgium

Mathias.Maes@UGent.be; Nele.DeBelie@UGent.be

Abstract

A lot of damage is reported for constructions in marine environments. Marine environments are very aggressive, since sea water consists mainly of chlorides. When cracks appear in the concrete structures, chlorides will penetrate faster and initiate corrosion and deterioration faster as well. A possible solution is autonomous crack healing by means of encapsulated polyurethane (PU).

Chloride resistance of autonomously PU-healed mortar was tested and compared to cracked and uncracked mortar. To do so, realistic crack widths of 100 and 300 µm were formed by means of a controlled splitting test. To obtain autonomous crack healing, glass tubes filled with a prepolymer on the one hand and an accelerator on the other hand were placed in the specimens at middle height. Upon crack formation, the tubes break and the healing agent is released. Then, the specimens were immersed for 7 weeks in a 165 g/l NaCl solution. After the immersion period, colour change boundaries as well as chloride profiles at different distances from the (healed) crack were obtained.

Based on the results, it seems that autonomous crack healing by means of PU has a beneficial influence on chloride resistance. Nevertheless, there is room for further improvement.

1 INTRODUCTION

Because of the high chloride concentration in sea water, a lot of damage is reported for constructions in marine environments. A commonly used material for such structures is reinforced concrete. However, chlorides affect the durability of concrete by initiating corrosion of the reinforcement steel. In addition, when cracks appear in the concrete structures, chlorides will penetrate faster. So, it is important to repair these cracks and since constructions in marine environments mostly have an high economic impact, fast repair is desirable. However, repair costs are large and in some cases repair is impossible due to inaccessibility. A possible solution is self-healing concrete. Self-healing concrete has the
ability to recover without external intervention. From the literature concerning self-healing concrete/mortar, it is clear that research focuses on the general concept, the mechanical properties and water permeability. Based on the water permeability it is concluded whether harmful substances will penetrate. Specific data on degradation of self-healing concrete/mortar in aggressive environments are not available.

In former research of the authors [1] the efficiency of manually healed concrete concerning the resistance against chloride penetration was tested by means of rapid chloride migration tests. A notch method, based on the method of Audenaert et al. [2] was established to produce artificial cracks in the concrete by means of thin steel plates introduced into the fresh specimen and removed after 10 hours. When the specimens were hardened, the cracks were manually injected with polyurethane. After the migration tests chloride penetration was visualised by spraying AgNO\textsubscript{3} on split specimens. For cracked concrete, the chloride penetration at the crack tip increased in function of the crack width when the crack width was in the range of 0 mm (= penetration from the surface) to 300 µm. Manual healing by means of polyurethane injection increased the chloride penetration resistance. For healed concrete with a crack width of 100 µm, 83 % of the samples regained almost full resistance against chloride penetration. In the case that cracks had a width of 300 µm, 67 % of the samples regained almost full resistance. Based on these results, the authors decided to test autonomously healed mortar by means of realistic cracks, as described in this paper.

2 MATERIALS AND METHODS

2.1 Glass capsules filled with PU-healing agent

In order to carry the healing agent in the mortar specimen tubular glass capsules were used similar to the capsules used by Van Tittelboom et al. [3]. Due to a high brittleness, the tubes will easily break whenever cracks appear in the mortar matrix. Borosilicate glass tubes with an internal diameter of 3 mm and an outside diameter of 3.35 mm were used. The tubes had a length of 50 mm.

MEYCO MP 355 1K (BASF The Chemical Company) was used as healing agent. This is a commercial one-component healing agent which is applied as a two-component agent to accelerate the reaction. The main compound consists of a prepolymer of polyurethane (PU) with a viscosity of 320 mPas at 20 °C which starts foaming in moist surroundings and forms a closed cell structure. The second compound is an accelerator diluted with water. The expanding reaction of this healing agent, with a reaction time of 10-50 seconds, may lead to an increase in volume of 25-30 times.

Half of the tubes was filled with the prepolymer and the other half was filled with a mixture of accelerator and water. First, the tubes were sealed with polymethylmethacrylate at one end, then, the tubes were filled with the components of the healing agent, which were injected by means of a syringe with a needle. When all tubes were filled, the other ends were sealed and the tubes were ready to be embedded inside the mortar samples.

2.2 Mortar with(out) self-healing properties

To determine the efficiency of self-healing mortar in chloride containing environments, an Ordinary Portland Cement mortar (OPC) was prepared. The composition is based on the mix described in EN 196-1 (2005), with a water to cement ratio of 0.5 and a sand to cement ratio of 3. Cylindrical specimens with a height of 50 mm and a diameter of 100 mm were used.
First, a 20 mm mortar layer was brought into the moulds. When this layer was compacted by means of vibration, three couples of glass tubes were placed on top of it. Afterwards, the moulds were completely filled and vibrated again. The non-self-healing samples were prepared in the same way, however, samples belonging to these series contained no glass tubes.

After casting, the specimens were placed in an air-conditioned room with a temperature of 20 °C and a relative humidity higher than 95 %. Demoulding took place the next day whereupon the specimens were stored again under the same conditions until the age of 21 days. Then, the specimens were coated with epoxy at the side, this way the coating acts as reinforcement during the crack formation.

### 2.3 Crack formation - Splitting test

In the cylindrical mortar specimens, cracks were created at 24 days of age by means of a crack width controlled splitting test. The crack width was measured at both sides of the specimen with a LVDT (Fig. 1) and the mean value was used to control the splitting test. The crack width was increased with a velocity of 0.5 µm/s until a crack width of either 180 µm or 500 µm was reached. Then, specimens were unloaded giving rise to a final crack width of approximately 100 µm and 300 µm, respectively.

![Figure 1: Splitting test to create cracks (Based on [3]).](image)

### 2.4 Crack healing

Cracks in the specimens containing encapsulated healing agent were autonomously healed, as can be seen in Fig. 2.

![Figure 2: Self-healing mechanism by means of encapsulated PU. (a) Placing of the glass tube couples, (b) Cracking and healing.](image)
The embedded tubes broke during crack formation and both components of the healing agent were released. Due to capillary forces in the crack and the expanding reaction of the healing agent (cf. part 2.1), a closed cell structure is formed that fills up the crack. Cracks can be sealed completely and become air- and watertight. So, not only the low viscosity and the capillary forces in the crack but also the expansive reaction acts as the driving force for the healing agent to fill up the crack.

### 2.5 Diffusion test

The resistance to chloride penetration was evaluated by using the diffusion test as described in NT Build 443 (1995). The day after crack formation and healing, the specimens were coated again except for the casting surface. At the age of 28 days, the specimens were placed in a 4 g/l Ca(OH)$_2$ solution for 7 days until constant mass was reached. Afterwards the specimens were placed in the test solution, being a 165 g/l NaCl solution.

After 7 weeks storage in the test solution, the chloride penetration was measured by means of the colour change boundary, more specifically by spraying 0.1 M AgNO$_3$ on both halves of a split specimen. At least three healed samples were split per crack width. The colour change boundary was measured by means of image analysis software (ImageJ).

Next, chloride profiles were obtained by potentiometric titrations with a Metrohm MET 702 automatic titrator and a 0.01 mol/l AgNO$_3$-solution. Powder to extract the chloride from was collected by grinding layers of 2 mm thickness up to a depth of 20 mm. In order to obtain information about the chloride penetration around the crack, powder was collected in a zone of 18 x 75 mm with the crack in the middle. Afterwards powder was collected from two separated zones of 18 x 75 mm both 9 mm away from the crack, see Fig. 3. Here as well, three specimens per sample type were analysed.

Figure 3: Grinding method to collect powder around the crack.

The extraction of chlorides from the powder was based on the method described by Maes et al. [4]. In this paper, the acid-soluble chloride content, which is the total chloride content, is measured. By using the titrated volumes of AgNO$_3$, chloride contents were calculated using Eq. 1.

$$C_t = \frac{10 \times 100 \times 35.45 \times 0.01 \times Vol.AgNO_3}{1000 \times 2}$$  \hspace{1cm} (1)
where $c_t$ represents the total chloride concentration (wt.%-concrete); 10 the dilution factor; 35.45 the atomic mass of chlorides (g/mol); 0.01 the concentration of the titration solution (mol/l); Vol.AgNO$_3$ the titrated volume of silver nitrate (ml); and 2 the mass of the concrete powder in the extraction solution (g).

Non-steady-state diffusion coefficients $D_{nssd}$ and chloride surface concentrations were obtained by fitting Eq. 2, which is the error function solution of Fick’s second law, to the measured chloride profiles, using a non-linear regression analysis in accordance with the least squares method.

$$C_i(x,t) = C_s - (C_s - C_i) \cdot \text{erf} \left( \frac{x}{\sqrt{4 \cdot D_{nssd} \cdot t}} \right)$$

where $c_i(x,t)$ is the chloride concentration at depth $x$ and time $t$ (wt.%-concrete), $C_i$ the initial chloride concentration (wt.%-concrete), $C_s$ the chloride concentration at the surface (wt.%-concrete), $D_{nssd}$ the non-steady-state diffusion coefficient (m²/s), $x$ the distance from the surface until the middle of the considered layer (m) and $t$ the exposure time (s). Compared to the mass of concrete, it is reasonable to assume that the initial chloride concentration $C_i$ in Eq. 2 equalled 0 %. The first layer was excluded from the regression analysis, since the measured chloride concentration in the first layer is generally considered not representative.

3 RESULTS

3.1 Colour change boundaries

In order to evaluate the healing effect with regard to the resistance against chloride penetration after 7 weeks immersion in the 165 g/l NaCl solution, four healing categories are defined based on the colour change boundary, see Fig. 4: (1) Totally healed (no Cl$^-$-penetration around the crack), (2) Partially healed (Cl$^-$-penetration until the tubes), (3) Partially healed (Cl$^-$-penetration beyond the tubes (< crack length)) and (4) No effect (similar to cracked concrete).

Figure 4: Visual indication of four categories to evaluate healing based on the chloride penetration depth around the crack measured by means of the colour change boundary.

Table 2 gives an overview of the ability to regain resistance against chloride penetration due to autonomous healing by means of encapsulated polyurethane. The percentages represent the part of the samples belonging to the specific category. To classify them, colour change boundaries were measured and compared.
Table 2: Influence of autonomous crack healing in OPC mortar with regard to chloride penetration, after 7 weeks immersion in a 165 g/l solution. Classification based on Fig. 4.

<table>
<thead>
<tr>
<th>Healing category</th>
<th>OPC 100 µm</th>
<th>OPC 300 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>(1)</td>
<td>50 %</td>
<td>-</td>
</tr>
<tr>
<td>(2)</td>
<td>17 %</td>
<td>33 %</td>
</tr>
<tr>
<td>(3)</td>
<td>-</td>
<td>17 %</td>
</tr>
<tr>
<td>(4)</td>
<td>33 %</td>
<td>50 %</td>
</tr>
</tbody>
</table>

According to the measured colour change boundaries, it seems that 67 % of the autonomously healed mortar specimens with an initial crack width around 100 µm regain almost full resistance against chloride penetration (categories 1 and 2). Besides, specimens with initial crack widths around 300 µm can be healed almost totally in only 33 % of the cases (category 2). For 33 % and 50 % of the autonomously healed mortar specimens with 100 µm and 300 µm cracks, respectively, no effect was observed.

3.2 Chloride penetration profiles

Fig. 5 shows the chloride profiles for healed, unhealed and uncracked specimens with crack widths of 100 µm and 300 µm. Each profile is the average of three individual profiles obtained in different zones (cf. Fig. 3), namely in the zone around the crack for healed specimens (PU – C), in the zone around the crack for unhealed specimens (CR – C) and in the zones next to the healed crack (PU – R) which gives an indication of uncracked samples (REF). The number in the specimen codes refers to the initial crack width.

![Chloride penetration profiles](image)

Figure 5: Average chloride profiles obtained after 7 weeks immersion in a 165 g/l NaCl solution, with initial crack widths of 100 µm on the one hand and 300 µm on the other hand.

Based on the average chloride profiles, it seems that autonomous crack healing has a beneficial influence on the resistance against chloride penetration. The chloride concentrations at different depths around healed cracks (PU–C) are clearly lower than in cracked samples without healing (CR-C). Although they are still higher than in uncracked

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mortar. So, based on these findings it can be concluded that autonomous crack healing by means of encapsulated PU has a beneficial influence on chloride diffusion, regardless the crack width, but uncracked concrete performs better. Nevertheless, it should be mentioned that these are average profiles. A more detailed inspection of the values of the individual chloride profiles per specimen, as can be seen in Fig. 6, learns that for specimens with a 100 µm crack as well as for specimens with a 300 µm crack only two of the three tested specimens were (almost) totally healed while one showed no healing effect. This is in accordance with the findings based on the inspection and categorisation of the colour change boundaries.

![Figure 6: Individual chloride profiles obtained after 7 weeks immersion in a 165 g/l NaCl solution, with initial crack widths of 100 µm on the one hand and 300 µm on the other hand.](image)

### 3.3 Chloride diffusion coefficients

By fitting Eq. 3 to the measured individual chloride penetration profiles, diffusion coefficients are calculated. Table 3 gives the average chloride diffusion coefficients with an indication of the standard deviations. Three specimens were examined per test configuration.

<table>
<thead>
<tr>
<th>Zone</th>
<th>( D_{\text{essd}} \times 10^{-12} \text{ m}^2/\text{s} )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>100 µm</td>
</tr>
<tr>
<td>CR-C</td>
<td>21.1 ± 3.0</td>
</tr>
<tr>
<td>PU-C</td>
<td>12.7 ± 9.3</td>
</tr>
<tr>
<td>PU-R</td>
<td>7.0 ± 2.3</td>
</tr>
<tr>
<td>REF</td>
<td>7.0 ± 0.2</td>
</tr>
</tbody>
</table>

In general the diffusion coefficients in the zone around an unhealed crack (CR–C) are higher than those in the zone around a healed crack (PU-C) and those for uncracked concrete (REF and PU-R), regardless the crack width. It is also clear that the mean values obtained for healed cracks are closer to the values for uncracked concrete than to those for cracked
concrete, indicating the beneficial influence of PU-crack healing. However, the standard deviations for the diffusion coefficients obtained around a healed crack show a large scatter. This finding is in accordance with former results and findings in this paper and can be explained by the fact that only a part of the PU-healed cracks are really sealed and filled up with PU. The latter is probably due to falling short of the healing mechanism rather than the fact that chlorides penetrate through the PU-foam since previous tests [3] showed that the closed cell structure of the used PU is watertight. Visual inspection of the specimens and the glass tubes after splitting shows that in the autonomously healed specimens without improved resistance against chloride penetration, the healing agents were still (partially) in the tubes. Possible explanations are that the crack formation was insufficient to trigger the healing mechanism or that the tubes were shifted during the moulding process, which made it not possible for the two components to react properly. Another possible explanation is the influence of the capillary forces [5]. The capillary forces in the tubes can be higher than in the crack, especially because long tubes (50 mm) and quiet large cracks (up to 300 µm on average) were used in this research. So, additional tests are performed in order to obtain more certainty with regard to the results of autonomous crack healing of mortar.

4 CONCLUSIONS

Autonomous crack healing of mortar by means of encapsulated PU has a beneficial influence on the resistance against chloride diffusion, regardless the crack width. If the healing mechanism works properly and the crack is sealed well, (almost) no chlorides will penetrate along the crack. However, based on chloride penetration depths, chloride profiles and chloride diffusion coefficients, it can be concluded that in 33 % of the cases crack healing has no effect on the resistance against chloride penetration. Nevertheless, autonomous crack healing is able to increase the durability of mortar/concrete structures in chloride-containing environments.

ACKNOWLEDGEMENTS

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REFERENCES


DEGRADATION OF BRAZILIAN GYPSUM EXTERIOR WALLS DUE TO HYGROTHERMAL ACTION

A. Santos(1), J.M.P.Q. Delgado(1) and N.M.M. Ramos(1)

(1) LFC − Laboratório de Física das Construções, Departamento de Engenharia Civil, Faculdade de Engenharia da Universidade do Porto, Porto, Portugal – dec11020@fe.up.pt; jdelgado@fe.up.pt; nuno.ramos@fe.up.pt

Abstract
This work is focused on the evaluation of degradation hazards of Brazilian gypsum exterior walls due to hygrothermal action. An extensive experimental characterization of the thermal conductivity, sorption isotherms, vapour permeability and water absorption coefficient of two different samples of gypsum plasters from the northeast Brazil (plaster pole of Araripe, Pernambuco) was performed. The experimental and analytical procedures followed either international standards or well-established methodologies, supporting the analysis of gypsum walls hygrothermal behaviour.

The measured properties were adopted as inputs to hygrothermal simulation software and the analysis of gypsum based exterior walls was conducted for different climate zones and exposure conditions. The study demonstrates the durability issues that may arise in each location, due to differences in hygrothermal action.

1 INTRODUCTION

Plaster is a binder, which compared with other materials as whitewash and Portland cement, can be considered much less aggressive to the environment. While, in the manufacture process, the Portland cement emits CO₂, the plaster launches water molecules in the atmosphere. Production of Portland cement requires high temperatures, while gypsum plaster can be prepared with temperatures of about 150°C. Although these ambient advantages, gypsum plaster is not very often applied in Brazilian buildings and an effort has been made to increase the consumption of this material in housing. Gypsum, abundant in nature and found in several deposits in northeast Brazil (plaster pole of Araripe, Pernambuco), can be used for manufacturing masonry units and renderings, resulting in a great added value for the local economy and environment.

Gypsum blocks are a building material that promises to bring down the cost of construction by at least 30% in comparison with other materials such as drywall system, brick walls or concrete/cement block walls.

The Brazilian Bioclimatic Zoning consists of eight different homogeneous zones. Zones Z1 and Z2 represent the colder regions and Z7 and Z8 represent the warmer regions of the
country. In the numerical analysis presented in this work Zone 7, city of Petrolina, Pernambuco state, was used as example. For comparison, the Porto (Portugal) climate was used.

2 EXPERIMENTS

In these experiments, an option was made in using specimens of common commercial materials used in Brazil. The experiments were therefore performed in specimens of hydro gypsum plaster and standard gypsum plaster, with the same density ($\rho = 1130 \text{ kg/m}^3$). These are the base materials for manufacturing blocks applied in masonry walls of low budget houses of north east Brazil.

2.1 Sorption Isotherms

Most building materials are hygroscopic, which means that they adsorb vapour from the environment until equilibrium conditions are achieved. This behaviour can be described by sorption curves over a humidity range of 0% to 95% RH. The sorption isotherms represent the equilibrium moisture contents of a porous material as a function of relative humidity at a specific temperature.

The experiments were performed in accordance with ISO 12571 (2000) standard. At a temperature of 23±2ºC, three relative humidity ambiences were used in the characterization of each of the two gypsum materials (50%, 75% and 90%), obtained in a clime chamber.

The gypsum plaster specimens had dimensions of 100x100x10 mm$^3$ and the specimens were initially dried at ambient temperature, during 30 days, in desiccators containing CaCl$_2$, guaranteeing a relative humidity below 0.5%.

For sorption measurements, the test specimen is placed consecutively in a series of test environments, with relative humidity increasing in stages, until equilibrium is reached in each environment. Equilibrium between moisture content and relative humidity has been reached when successive weightings, at time intervals of at least 24 h, show a difference in mass below 0.1%. From the measured mass changes, the equilibrium moisture content ($u$), at each test condition, could be calculated and the sorption isotherm drawn.

2.2 Vapour Permeability

Vapour permeability was determined for samples of the two base materials. The tests were conducted according to ISO 12572 (2001) standard and two permeability values were defined, corresponding to different ranges of RH differences across the sample. The conditions for each of the tests are defined in Table 1.

<table>
<thead>
<tr>
<th>Tests</th>
<th>Normalized conditions ($^\circ$C / %HR)</th>
<th>Effective temperature ($^\circ$C)</th>
<th>Effective relative humidity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A: Dry cup</td>
<td>23 / 0-50</td>
<td>23 ± 0.3</td>
<td>50 ± 3</td>
</tr>
<tr>
<td>B: Wet cup</td>
<td>23 / 50-93</td>
<td>23 ± 0.3</td>
<td>50 ± 3</td>
</tr>
</tbody>
</table>
Prior to testing, all the specimens are preconditioned in a climatic chamber at 23±2 °C and 50% RH, for a period long enough to obtain three successive daily determinations of their weight lesser than 0.5%. After stabilisation, the specimens are placed in cups with a saturated salt solution below the bottom surface of the specimen. The sides of the specimens were covered with vapour impermeable tape. The change of weight of the cup was measured periodically, with a precision of 0.1 mg, on an electronic balance until the steady state was obtained.

2.3 Water Absorption Coefficient

A material that allows liquid moisture diffusion through its boundary surface will change its weight with time when it is brought in contact with liquid water. It’s well known that a schematic plot of the increase in weight of the test specimen versus the square root of time indicates that the specimen weight increases linearly before it comes close to the saturation limit. The slope of this linear variation is called the water absorption coefficient \( A_w \) and can be mathematically written as:

\[
\frac{m - m_0}{S} = A_w \sqrt{t}
\]

where \( m \) is the sample mass at time \( t \), \( m_0 \) is the initial sample mass, \( S \) is the liquid contact surface area and \( t \) is the time.

For all the samples, their masses were recorded at various time intervals. The mass of moisture of each sample in grams was obtained by subtracting the mass of the oven dried samples from the mass of the moist samples. Finally, curves showing the cumulative weight gain versus the square root of time were plotted, and linear regression curves were computed for each. The \( R^2 \) coefficients of the fitting curves were high, ranging from 0.981 to 0.998. Table 2 shows the DIN 52.617 (1987) classification of building materials related to water absorption coefficient.

Table 2: DIN 52.617 (1987) classification of building materials related to water absorption coefficient.

<table>
<thead>
<tr>
<th>( A_w )</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>( &gt; 333 \times 10^{-4} \text{ kg/(m}^2 \cdot \text{s}^{0.5}) )</td>
<td>Rapid suction</td>
</tr>
<tr>
<td>( &lt; 333 \times 10^{-4} \text{ kg/(m}^2 \cdot \text{s}^{0.5}) )</td>
<td>Preventive against water</td>
</tr>
<tr>
<td>( &lt; 83.3 \times 10^{-4} \text{ kg/(m}^2 \cdot \text{s}^{0.5}) )</td>
<td>Water repellent</td>
</tr>
<tr>
<td>( &lt; 0.167 \times 10^{-4} \text{ kg/(m}^2 \cdot \text{s}^{0.5}) )</td>
<td>Water impermeable</td>
</tr>
</tbody>
</table>

3 EXPERIMENTAL RESULTS

3.1 Sorption Isotherms

The isothermal adsorption data measured on the three materials tested are given in Figure 1 and the results are averages of five samples. The Hansen (1986) model (Eq. 2) was used for fitting the data

\[
u = u_h \cdot \left(1 - \ln \frac{\phi}{A_i}\right)^{-1/\alpha}
\]

The results of parameter estimation, for the experimental data, are summarized in Table 3. The fittings of the proposed model are very satisfactory for the examined building materials. The maximum moisture content, at 90% RH, was observed for hydro gypsum, confirming the
fact that the standard gypsum show reduced hygroscopicity (adsorbed moisture is less than 0.1%).

Table 3 - Values of the fit parameters for the Hansen equation (Eq. 2).

<table>
<thead>
<tr>
<th>Isotherm Models</th>
<th>Standard Gypsum Adsorption</th>
<th>Hygro Gypsum Adsorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>$n$</td>
<td>2.32%</td>
<td>2.33%</td>
</tr>
<tr>
<td>$A_1$</td>
<td>70.376</td>
<td>67.555</td>
</tr>
<tr>
<td>$h_0$</td>
<td>0.002</td>
<td>0.001</td>
</tr>
</tbody>
</table>

Figure 1: Sorption isotherms for the two Brazilian gypsum materials tested.

3.2 Vapour Permeability

The graph presented in Figure 2 shows the relative humidity profiles admitted for the gypsum samples during the permeability experiments, in accordance with the theory presented below.

Figure 2: Water vapour permeability for the gypsum plaster samples tested, at 23°C.
The vapour permeability, $\delta_p$, is a material property defined as the transport coefficient for vapour diffusion in a porous material subjected to a vapour pressure gradient. The permeability can be calculated using,

$$\delta_p = g \frac{d}{\Delta P}$$

where $g$ is the mass flux density and $d$ is the sample thickness exposed to a vapour pressure gradient $\Delta P$.

Other properties derived from vapour permeability can be used, as the transport coefficient under a vapour concentration gradient: vapour resistance factor $\mu = \frac{\delta_s}{\delta_p}$, $\mu = \frac{\delta_s}{\delta_p}$, or vapour diffusion thickness, $s_d = \mu \cdot d$.

### 3.3 Water Absorption Coefficient

Typical plots of mass gain versus the square-root of time (water intake curves), at 20°C, are shown in Figure 3, as the calculated water absorption coefficient for both materials. For the first sample (standard gypsum plaster) the average water absorption coefficient obtained was $4495 \times 10^{-4}$ kg/m²s⁰.⁵, and for hydro gypsum plaster samples was $35.2 \times 10^{-4}$ kg/m²s⁰.⁵.

![Figure 3: Water absorption coefficient of different gypsum plaster materials in function of the square-root of time.](image)

An analysis of the water absorption coefficients values obtained with the DIN 52.617 classification shows that the standard gypsum plaster samples are classified as “Rapid suction”, while the hydro gypsum plaster sample is classified as “Water repellent” (see Table 2).

### 4 NUMERICAL RESULTS

The WUFI 5.2 allows realistic calculation of the transient hygrothermal behaviour of multi-layer building components exposed to natural climate conditions (Kuenzel and Kiesll, 1996). This program is a one-dimensional model for heat and moisture transport analysis of building envelope components, based on the finite volume method.

The governing equations for moisture and energy transfer are, respectively,

$$\frac{\partial w}{\partial t} + \nabla \cdot (D_p \nabla \varphi + \delta_p \nabla (\varphi p_{sat})) = 0$$

(4)
\[
\frac{\partial H}{\partial T} \frac{\partial T}{\partial t} = \nabla(\lambda \nabla T) + h_v \nabla(\delta_p \nabla(\phi p_{\text{sat}}))
\]

where \(w\) is water content (kg/m\(^3\)), \(\phi\) is the relative humidity (%), \(t\) is the time (s), \(D_{\phi}\) is the liquid conduction coefficient (kg/ms, \(\delta_p\) is the vapour permeability (kg/m.s.Pa), \(p_{\text{sat}}\) is the saturation vapour pressure (Pa), \(H\) is the enthalpy (J/m\(^3\)), \(T\) is the temperature in Kelvin and \(h_v\) is the latent heat of phase change (J/kg).

The construction type analysed is a gypsum wall made of standard or hydro Brazilian gypsum with climate data of Petrolina (Brazil) and Porto (Portugal). The conditions of indoor air were constant, with \(\phi_{\text{in}} = 60\%\) and \(T_{\text{in}} = 20^\circ\text{C}\). The building component under study has a gypsum block (hydro or standard) of 10 cm and a finishing rendering of 0.3 cm of gypsum. A coating with an \(s_d\) value of 0.1 was considered for the interior surface and an \(s_d\) value of 1 was considered for the outside surface. The interior heat transfer coefficient was constant and equal to 8 W/m\(^2\)K and the exterior heat transfer coefficient only contained the convective part and was considered independent from the wind (constant value of 17 W/m\(^2\)K).

European standard EN 15026:2007 provides minimum criteria for simulation software used to predict one-dimensional transient heat and moisture transfer in multi-layer building components exposed to transient climate conditions on both sides, and WUFI 5.2 complies with all requirements of this European standard.

The application of WUFI 5.2 to a gypsum wall provides the variation with time of the relative humidity in the interior surface of the building element and moisture content in each layer (see Figures 4, 5 and 6).

Figure 4: Cumulative distribution of relative humidity in the interior of the gypsum house.
Figure 5: Wall moisture content.

Figure 6: Mould growth risk.
5 CONCLUSIONS

The construction of low budget houses with gypsum walls is common in certain parts of Brazil. The adoption of a mixture with low water absorption values (gypsum hydro) can be effective to ensure durability regarding hygrothermal action in specific climates such as inland northeast Brazil, as mould growth risk is mitigated.

ACKNOWLEDGEMENTS

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REFERENCES

A NEW PERFORMANCE TEST TO ASSESS THE POTENTIAL DELETERIOUS EFFECT OF SULPHIDE-BEARING AGGREGATES FOR CONCRETE

Josée Duchesne (1), Andreia Rodrigues (1) and Benoit Fournier (1)

(1) Department of geology and Geological Engineering, Université Laval, Québec – josée.duchesne@ggl.ulaval.ca; andreia.oberon81@gmail.com; benoit.fournier@ggl.ulaval.ca.

Abstract

Recently, important damages in concrete containing sulfide-bearing aggregates were observed in the Trois-Rivières area (Québec, Canada). More than 1000 residential owners and many commercial buildings have faced serious issues related to the deterioration of their concrete housing foundations and slabs. The problematic aggregate is an anorthositic gabbro containing sulfide minerals. These sulfide minerals are unstable in the presence of oxygen and humidity resulting in an oxidation reaction; and pyrrhotite is known as one of the most “reactive” sulfide minerals.

The goal of this research is to develop a performance test to assess the potential deleterious effect of different sulfide-bearing aggregates for concrete. So far, the chemical approach, using a measure of the total sulfur content is a tool used for the rapid detection of the presence of iron sulfides in aggregate. This technique cannot identify the type of sulfide present and serves only as a screening test and further testing is needed. In the approach proposed, the next step is a newly-developed mortar bar expansion test where the bars are subjected to a cyclic exposure conditioning regime involving wetting and drying cycles in an oxidizing agent keeping temperature and humidity conditions that are favorable to oxidation reaction.

1 INTRODUCTION

Cases of rapid degradation of concrete associated to iron sulfides in aggregates were recently recognized in the Trois-Rivières area. More than 1000 residential and commercial owners have faced serious issues related to the deterioration of their concrete foundations and slabs. In some cases, the deterioration was such that immediate remedial actions were required and concrete has to be replaced (figure 1). All the masonry and covering stones were first removed because they stand on the foundation walls and permit to lighten the structure. Houses were lifted up from their foundations and the later was demolished and replaced.

The signs of concrete deterioration observed in the distressed concretes mainly consist of map cracking on the surface of the walls with some crack openings reaching more than...
40mm. Yellowish and brownish discoloration was often observed surrounding these cracks. Pop-outs are observed on the walls showing the presence of oxidized and rusted aggregate particles. Small bumps easily detachable from below showing oxidized and rusted aggregate particles can be seen. Some of these deterioration features are illustrated in figure 2.

Figure 1: Replacement of the concrete foundation. All the masonry and covering stones were removed, houses were lifted up and concrete foundations were demolished and replaced.

A large number of concrete samples were investigated. In all cases, the aggregate material used for the concrete manufacturing was an intrusive igneous rock, more precisely an anorthositic gabbro, containing various proportions of sulfide minerals including pyrite (FeS₂), pyrrhotite (Fe₁₋ₓS), chalcopyrite (CuFeS₂) and pentlandite ((Fe,Ni)₉S₈). Figure 3 presents views of the anorthositic gabbro particles on fresh and polished surfaces.

Iron sulfides are common minor constituents in many rock types. Pyrrhotite, a non-stoichiometric mineral of general formula Fe₁₋ₓS, with x varying from 0 (FeS) to 0.125 (Fe₇S₈) [1-2] is well known as an unstable mineral in the presence of oxygen and humidity. Belzile et al. 2004 [2] presented a review of pyrrhotite oxidation processes. A deleterious process involving the oxidation of sulfide minerals and the sulfatation of the cement paste is thought to have caused the swelling and cracking of the affected concrete elements. In fact, the presence of secondary reaction products including iron oxyhydroxide, gypsum, ettringite, ettringite/ thaumasite solid solution and thaumasite was observed and support this hypothesis. The chemical reactions that lead to the oxidation of iron sulfide minerals and in turn the sulfate attack of the concrete are described in detail elsewhere [3,4].

Several publications have already identified the risks of sulfide oxidation for engineering works [5] but no precise guideline is available for the quality control of the aggregate materials. Until now, no study has been able to reproduce in the laboratory the expansion and deterioration of concrete containing sulfide bearing aggregates.
Figure 2 - Examples of concrete deterioration features A – Map-cracking in concrete foundation showing some yellowish surface coloration. B – Cracks were filled up with sealant materials to prevent water infiltration. C – Large open cracks seen on the interior side close to an exposed oxidized aggregate particle. D – Pop outs on the interior side of a wall showing oxidized aggregates particles surrounded by whitish secondary products.

Figure 3 – Crushed anorthositic gabbro coarse aggregate particles with a stereomicroscopic view of a polished sample showing aggregate particles containing iron sulfide minerals.
2 MATERIALS AND METHODS

The main material used in this study is the anorthositic gabbro (problematic aggregate) from the Saint-Boniface quarries, in the Trois-Rivières area, Québec, Canada. Aggregate materials without sulfide minerals are used as control specimen. A total of 8 aggregates were studied including 6 sulfide-bearing aggregates and 2 control aggregates.

The chemical approach, using a measurement of the total sulfur content is a tool used for the rapid detection of the presence of sulfide minerals. Aggregate total sulfur content is used to get an approximation of the sulfide content calculated from mineral stoichiometry. This technique cannot identify the type of sulfide present and serves only as a screening test. The total sulfur content of aggregate material is determined by an infrared absorption method using an Eltra CS 800 carbon/sulfur analyzer. The sub-sample required for analysis is less than 1 g. A sample preparation method is proposed to avoid sampling bias.

Finally, a newly-developed mortar bar expansion test where the bars are subjected to a cyclic exposure conditioning regime involving wetting and drying cycles in an oxidizing agent keeping temperature and humidity conditions that are favorable to oxidation reaction is proposed. The goal of this test is to establish favorable conditions that will reproduce, in the laboratory, the expansive process responsible for the damage.

3 RESULTS AND DISCUSSION

3.1 Global evaluation program of concrete aggregate: 1st - Total sulfur content

Until now, the chemical approach, via a measurement of the total sulfur content, is a tool used for the rapid detection of the presence of iron sulfide minerals in an aggregate sample. In fact, the iron sulfide minerals are formed chemically by the presence of iron and sulfur. The measurement of the total sulfur content is used to calculate the sulfide content using iron sulfide minerals stoichiometry. For example, pyrite (FeS₂) contains 53.45% of S and pyrrhotite (Fe₁₋ₓS) contains 37.67 % S. To determine the content of iron sulfide minerals, some assumptions must be made. First, almost all sulfide minerals contain sulfur, whether it is pyrrhotite, pyrite, chalcopyrite, ... so you cannot redistribute sulfur in different minerals unless data on the mineralogical distribution is known. The calculation is made from the stoichiometry of the minerals and does not consider substitutions or impurities.

The total sulfur content is often determined by an infrared absorption method with a carbon/sulfur analyzer. This device consists of sample combustion in an induction furnace and measurement of combustion gas absorption in the infrared region. For analysis, a sample of powder under 0.3 to 1 g is required. The sulfur content is measured from the SO₂ in the flue gas. Since the sub-sample required is less than 1 g, it is important that it is representative of the initial sample. Sampling bias and preferential grinding of soft phases may be avoided.

Following critical evaluation of sample preparation methods suggested in the CAN/CSA-A23.2-26A-09 (Determination of Potential Alkali-Carbonate Reactivity of Quarried Carbonate Rocks by Chemical Composition) and in the BNQ 2560-500/2003 (Aggregates - Determination of the petrographic index of the potential sulfate swelling for granular materials), a sample preparation method was developed and used for all aggregate samples analyzed in this study (Figure 4).

An initial sample of 4 kg, with particle size ranging between 5 and 20 mm, is split in two representative subsamples containing more than 300 particles each. A portion of 2 kg is then crushed to reduce all particles to a size less than 5 mm. The sample is split until a sub-sample...
of 500 g is obtained (2 splits). The sub-sample of 500 g is then pulverized so that all particles are less than 300 microns. The sample is then split until a mass of 50 g is obtained (3 splits). The sample, with a weight of about 50 g, is entirely pulverized until all particles are less than 80 microns in size. The resulting sample is split in 4 parts for the different chemical analyzes.

To validate the assay variability on the total sulfur content measurement, samples of different types of aggregate materials were prepared and split into four portions to be analyzed in four different laboratories. For quality controls, some blanks and duplicates were also analyzed. Similarly, standard samples were included in each series of analysis. Table 1 presents the results obtained by each laboratory as well as key statistics, mean, standard deviation and coefficient of variation.

![Sample preparation method](image)

Figure 4 – Sample preparation method to ensure that the sub-samples obtained are the most representative as possible as the initial sample.

The results obtained show that the inter-laboratory variability is acceptable, with values of the coefficient of variation lower than 10% for most of the samples analyzed. Higher coefficient of variation values correspond to samples with very low total sulfur content in order of 0.1% or less. Duplicates samples (5 and 5-bis and 10 and 10-bis) show little variation and present the same mean values. Similarly, the values obtained for the standard sample, CVT-1 corresponds to the published values. We can conclude that the results are accurate and precise and can be used as a screening tool in the global evaluation program.

### 3.2 Global evaluation program of concrete aggregate: 3rd - Mortar bar expansion test

The oxidation of iron sulfide minerals generates secondary reaction products such as iron oxyhydroxides (goethite, limonite, ...) products. This reaction generates also sulfuric acid that reacts with the cement paste components producing internal sulfate attack with presence of gypsum, ettringite and thaumasite. All these secondary minerals have a higher volume than the reactants and are responsible for the swelling and cracking of the concrete.

The objective of this section is to develop a performance test on mortar bars to reproduce in the laboratory the expansion observed on the affected concrete caused firstly by the oxidation of the sulfide-bearing aggregate followed by the sulfate attack of the cement paste.
Preliminary tests were carried out on mortar bars specimens in different conditions of humidity, temperature and using different oxidizing solutions in order to establish favorable conditions for the development of a performance test that will reproduce, in the laboratory, the expansive process responsible for the damage of the concrete incorporating sulfide-bearing aggregates. Mortar bars, 25x25x28.5 mm in size, were manufactured using a water-to-cement ratio (w/c) of 0.70 and a cement-to-aggregate ratio of 1:2.72. A high w/c was chosen to reproduce the characteristics of concrete used in the damaged housing foundations. The grading requirement for aggregate used was the same as that used in the standard test method for potential alkali reactivity of aggregates (mortar bar method) (CAN/CSA A23.2-25A). All the bars were prepared with a general use (GU) portland cement. A total of 8 types of aggregates with different sulfides contents, including 2 control specimens without sulfide minerals, were used in this experiment. In order to reproduce the deterioration observed in the

Table 1 – Results of the total sulfur content measured by 4 laboratories

<table>
<thead>
<tr>
<th>Sample</th>
<th>Lab 1 S (%)</th>
<th>Lab 2 S (%)</th>
<th>Lab 3 S (%)</th>
<th>Lab 4 S (%)</th>
<th>Mean</th>
<th>σ</th>
<th>CV (%)</th>
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<td>1</td>
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<td>0.44</td>
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<tr>
<td>2</td>
<td>1.71</td>
<td>1.72</td>
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<td>0.012</td>
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<td>0.25</td>
<td>0.007</td>
<td>2.8</td>
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</tbody>
</table>

*11 is a blank  *CVT-1 is a standard with published value = 0.26 σ 0.05 (n = 5-37)
field, the test was divided in two phases: the first promotes conditions favorable for the oxidation of the aggregate and the second promotes the sulfate attack of the cement paste.

According to [2], the oxidation reaction of pyrrhotite is enhanced at high temperature and humidity. To promote the oxidation of sulfides, mortar bars were stored at different temperatures (21°C, 38°C, 60°C and 80°C), at three different relative humidity (60, 80 and 100%) with wetting and drying cycles in oxidizing solutions. The mortar bars were soaked twice per week for a 3-hour period into a 6% sodium hypochlorite solution (bleach) or a 3% hydrogen peroxide solution, and then replaced under their respective controlled temperature and humidity conditions. Mass and length measurements were monitored once a week. After 3 months in these conditions favorable to the oxidation reaction, the mortar bars were transferred to 4°C and 100% relative humidity to promote the sulfate attack.

Figure 5 presents the influence of the temperature on the expansion obtained with the Maskimo aggregate. All mortar bars were kept at 80% relative humidity with 2 wetting and drying cycles per week in the sodium hypochlorite solution. A temperature of 80°C increases the expansion due to the oxidation of the sulfide bearing aggregate. Figure 6 presents the expansion obtained for the mortar bars kept at 80°C and 80% relative humidity with 2 wetting cycles per week in the sodium hypochlorite solution. A set of bar was transferred at 4°C after 90 days; these conditions increased the rate of expansion. Photographs of the samples after the expansion test are also presented. Maskimo samples present polygonal cracks that often originate from oxidized aggregate particles. Many aggregate particles are covered with rust. Control samples are intact despite the severe conditions they were exposed to.

Different parameters were tested in the mortar expansion test. Two oxidizing solutions (sodium hypochlorite (bleach) and hydrogen peroxide solutions) were used to soak the bars twice a week. However, expansion was obtained only with the sodium hypochlorite solution. Shrinkage of the mortar bars was obtained using the hydrogen peroxide due to its lower pH that promotes the neutralisation of the solution while in contact of the mortar bars. In the first part of the test (in the oxidation phase) different temperatures were tested (21, 38, 60 and 80°C) and it was clear that the higher the temperature was, the greater was the expansion measured. Samples were stored at 60, 80 and 100% relative humidity. Both 60 and 80% relative humidity gave similar results. Finally, higher expansion is obtained if the mortar bars are transferred at 4°C after 3 months at high temperature to promote the sulfate attack.

![Figure 5: Expansion of mortar bars as a function of time for samples with Maskimo aggregate stored at different temperatures.](image-url)
CONCLUSIONS

The goal of this study was to develop a global evaluation program of concrete aggregate to identify potentially deleterious sulfide-bearing aggregates prior to their use in concrete. The approach proposed begins by a measure of the total sulfide content of the aggregate. After, the aggregate was evaluated by a newly-developed mortar bar expansion test. Mortar bars were kept at 80°C and 80% relative humidity for 90 days with two 3-hour wetting and drying cycles a week in a 6% sodium hypochlorite solution. At 90 days, the bars were transferred at 4°C to promote the sulfate attack. The performance approach developed will be extended to a broader range of aggregates to establish the threshold values at each step of the protocol.

REFERENCES

VACUUM INSULATION TECHNOLOGY FOR BUILDINGS – A COMPARATIVE LCA STUDY OF A SWEDISH MULTIFAMILY BUILDING

Kjartan Gudmundsson (1), Peyman Karami (1) and Christer Sjöström (1)

(1) School of Civil and Architectural Engineering, KTH, Stockholm, kjartan@byv.kth.se

Abstract

A significant share of the energy consumption in Sweden and Europe (about 40%) can be related to the heating of buildings, about half of which can be related to the thermal losses through the envelope. An improved thermal insulation of new buildings and the existing building stock is therefore an important step towards reduced energy consumption. With traditional insulation materials, this will result in an increased transport volume, an added complexity in terms of detailing and the loss of living space area. With a thermal insulation capacity about 8-10 times that of traditional insulation materials, vacuum insulation panels, VIPs, might provide an interesting alternative. This study compares the environmental impact of different of VIPs and traditional thermal insulation and the relevance on a system level. This is done through a study of a typical Swedish multifamily building. The spatial properties together with material specifications of the Building Information Model are used to calculate the energy consumption and the carbon footprint of the building while investigating the sensitivity of the results to service life of the various components. The energy calculations are based on local climatic conditions while material data has been retrieved from the EPDs of the manufacturers.

1 INTRODUCTION

1.1 Energy and the climatic envelope Title

A large part of the energy used in Sweden and the European Union can be related to the thermal losses through the climatic envelopes of buildings (about 20%). This presents an economical and environmental challenge, the solution of which must include the reduction of heat losses through the walls, floors and roofs of buildings. With traditional insulation materials, this will lead to a significant increase in the thickness of the climatic envelope and consequently an increased volume of transport, loss of living space and a restriction on architectural design. As an example, a house in Sweden would need to have an insulation thickness of some 335 mm in the wall and 500 mm in the roof in order to meet the passive house standard corresponding to U-values of 0,10 and 0,066 W·m²·K⁻¹.
1.2  New opportunities with VIPs

Vacuum insulation panels (VIPs) have a thermal resistance of about 8-10 times that of a conventional insulation material. This provides the opportunity to obtain superior thermal insulation of buildings, yet with a slender climatic envelope. A regular vacuum insulation panel is made of a gas-tight envelope surrounding a porous core from which the air has been evacuated. There are, however, vast possibilities of combining different core materials and envelopes in different typologies as described in the literature [1]. The relatively small pore size in combination with low pressure prevents thermal transport through convection of the gases within the core. By using core material with pore size in the range of the mean free path of the gas molecules the conduction through the collision of gas molecules can also be limited. Conduction through the core material is minimized by using materials with a sparse solid matrix while the radiative heat exchange between the interior surfaces of the core material depends on the surface properties that can be improved by the use of additives.

![A regular VIP panel with a fumed silica core](image)

Figure 1. A regular VIP panel with a fumed silica core [2].

Until now, the application of VIPs has been limited to a small number of buildings. Further description and examples of applications can be found in the report of the International Energy Agency [3] as well as in Ghazi Wakili et al. [4], Schwab et al. [5] and Binz and Steinke [6]. Batens et al. [7] have also done a review of VIPs in building applications.

1.3  Durability and service life of VIPs

The service life time of a VIP can be defined as the time at which the required thermal conductivity has been surpassed, while a further account of service life is given in ISO 15686-2 [8]. One of the most important aging mechanism of VIPs is the permeation of gas through the envelope that may be enhanced at higher temperatures and humidity and is further increased at the edges [9]. The edge effect will get greater with the ratio of edge length over area of the panel [10]. It is common to put getters or desiccants in the core material in order to adsorb or entrap residual gas or moisture and thereby secure or prolong the service life of panels [11]. With current technology the gas pressure increase of a 20 mm thick VIP with metallized high barrier envelope is around 1-2 mbar a year giving a lifetime of 25-50 years or even more depending on the required performance of the panel [12,9,2]. The problems of gas
permeation through the envelope can be solved by using virtually impermeable metal films with a thickness of 10 µm or more, but this can result in thermal losses that are of the same magnitude as the heat flow through the panel [13].

It must also be noted that VIP panels may fail due to imperfect production or through mechanical damages during installation.

1.4 Life Cycle Assessment (LCA)

The LCA method is quite rigidly defined by SETAC, the Nordic guidelines for LCA and within the ISO 14000 series [14]. It can be applied on all levels of the building process from the design phase to the operation of existing buildings.

By definition a LCA analysis consists of four distinct phases that are the definition of goal and scope, inventories, the evaluation of environmental impact and the interpretation of the results. Evaluation of environmental impact consists of four steps. The first step includes definitions of the environmental impact categories, such as global warming or acidification and is followed up by the second step that associates the categories with the presumptive mass flow and environmental loads. The third step includes ranking and weighing of the calculated impact in order to make it possible to do the evaluation. As this makes the method somewhat subjective it is of great importance that the model used is clear and transparent as well as in line with the objectives of the study. The fourth and final step includes conclusions and recommendations.

Recent research concerning the Life cycle analysis of buildings include the work of Thiel et al [15] that compared the impact of the different materials of a net-zero energy building. Other work has been concerned with primary energy use and CO₂ emission for the production and operation of conventional and low-energy residential buildings [16,17] as well as comparative studies of different structural alternatives [18,19] and building systems [20] while Osman and Ries [21] did a study of energy systems for providing energy to buildings.

The LCA analysis and environmental impact assessment of a regular vacuum panel made by a porous core media is currently limited to a fairly small number of investigations. For instance, the report of Gao et al. [22] did a LCA analysis of monodisperse hollow silica nanospheres that can be used as nano insulation materials. Intini et al. [23] did a study including evaluation of environmental impact of VIPs made with recycled PET and with virgin PET products. A remarkable low environmental impact was concluded in the case of non-virgin PET products. Furthermore, Proietti et al. [24] did LCA assessments to make a comparison concerning the environmental performance of the reflective foil in comparison with other types of insulating materials. In a recent work, La Rosa et al. [25] did comparison of environmental impacts of composite solutions for building applications, eco-sandwich panels.

In the case for vacuum insulation panels for building applications, the report of Kunic et al. [26] includes analysis of the global warming potential (GWP) for different thermal insulations of an external building envelope, where a significant small environmental influence of VIPs, was concluded.

Furthermore, concerning the investigation of carbon footprint of buildings, recent research of Ogden et al. [27] includes a comparative study of the VIPs and PUR as well as mineral wool based on a methodology of aggregated operational and embodied CO₂, where the detailed studies of this work [27] supports the case for the development of VIPs that combine higher thermal performance with relatively low embodied energy. When applied to the
different insulation materials in the study [27] the aggregated methodology demonstrated particular advantages for VIPs in relation to retrofit applications.

Our study puts emphasis on comparing the environmental impact of retrofitting a Swedish standard building, before and after retrofitting with VIPs and conventional insulation materials.

1.5 Environmental Product Declaration (EPD)

The environmental impact of a design alternative must be quantified if the potential effects of altering a design are to be evaluated. This can be done with the help of a Type III environmental product declaration, EPD, according to ISO 14025 and EN 15804, that give an account of the “cradle to gate” stages in terms of environmental LCA (ISO 14040 and 14044), while a comprehensive LCA will also include the end-of-life stage “Cradle-to-grave”. Aspects related to the use-stage of construction products are usually ascribed to the product itself and this stage is not a part of the EPD and the reader must therefore put the presented data into the overall performance context of a building [28].

2 BUILDING CASE STUDY

A residential building in Stockholm is investigated. The study contains a comparison of the life cycle impact of a standard building, a well-insulated building with traditional insulation material and a well-insulated building with vacuum insulation panels, VIPs. The study includes the primary energy use, both renewable and non-renewable as well as the global warming potential (GWP) emissions.

The functional unit of the LCA is a 2 story multifamily building of 4 apartments with a total living area of 520 m². Each apartment is assumed to be inhabited by a family of 3-4 people.

The building is a hypothetical building based on an open access Autodesk Revit® model. The data on the materials and products used was obtained from the EPDs of the manufacturer. The data used was no older than 4 years. The operational energy calculation is carried out with the VIP Energy software.

The building reference time is set to 50 years.

3 RESULT AND DISCUSSION

The assessment of the construction and building materials is limited to the climatic envelope while the surface finishes are excluded. The product stage, the construction process stage, the use stage and the end of life stage of the building products are included. The operational energy use of the building is included, excluding the household electricity. The benefits and loads beyond the system boundary are disregarded. Table 1 gives an account of the three different alternatives, concerning the insulation of the climatic envelope, that are investigated in this study.
Table 1: The building types and insulation properties

<table>
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<tr>
<th>Building Components</th>
<th>Area m²</th>
<th>U-Value W·m⁻²·K⁻¹</th>
</tr>
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<td></td>
<td></td>
</tr>
<tr>
<td>External walls</td>
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<tr>
<td>Roof</td>
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<td>windows</td>
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<td>windows</td>
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<td>0.09</td>
</tr>
<tr>
<td>Roof</td>
<td>274</td>
<td>0.065</td>
</tr>
<tr>
<td>windows</td>
<td>130</td>
<td>1.2</td>
</tr>
<tr>
<td><strong>Building type I-II-III</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Bearing internal walls: 550 mm reinforced concrete</td>
<td>222</td>
<td>-</td>
</tr>
<tr>
<td>Non-bearing internal walls 140 mm: gypsum board, 70 mm mineral wool, air gap, wood studs, gypsum board</td>
<td>347</td>
<td>-</td>
</tr>
<tr>
<td>Joist (Between two floors) 274 mm: gypsum board, 15 mm EPS, OSB, wooden joists, 95 mm mineral wool, wood furring strips, gypsum board</td>
<td>244</td>
<td>-</td>
</tr>
<tr>
<td>Ground 350 mm: 100 mm reinforced concrete, 50 mm EPS, 200 mm mineral wool, 150 mm macadam</td>
<td>261</td>
<td>0.105</td>
</tr>
<tr>
<td>Foundation</td>
<td>115</td>
<td>2.4</td>
</tr>
<tr>
<td>Bearing footing 900x300 mm²</td>
<td>32</td>
<td>1.4</td>
</tr>
<tr>
<td>Balcony slab</td>
<td>102</td>
<td>6</td>
</tr>
<tr>
<td>Doors</td>
<td>20</td>
<td>1.2</td>
</tr>
</tbody>
</table>

Figure 2 shows the global warming potential emission, measured as kg of CO₂ equivalent, while each bar represents the result values for various building components from product stage to end-of-life stage for a period of 50 years.
Each bar of figure 3 illustrates the primary energy use for the same building materials, measured in MJ, also evaluated from product stage to end-of-life stage for a period of 50 years.

![Energy Use](image)

The results show that the insulation materials have a comparatively notable effect on both primary energy use and the global warming potential while this effect depends on the choice of material and insulation thickness. The insulation materials of Building type II thus have a greater use of primary energy and GWP than those of Building type I due to a better insulation of walls and roof. It is also of interest to notice that the VIPs reduce the total primary energy use and GWP as can be seen by comparing the total result of insulation materials for Building type III with that for Building type II. The total result values of the GWP for all materials of each building are illustrated in figure 4 (left).

It is also of great interest to compare the magnitude of the cradle-to-grave energy use of the materials to the operational energy of the buildings. Figure 4 (right) shows the total primary energy use of the three buildings while each bar gives the primary energy use of the materials, both renewable and non-renewable, from cradle-to-grave as well as the result of the operational energy calculation carried out with the VIP Energy software. There is a considerable difference in the results between the standard building type I and the low energy buildings of types II and III.

![Figure 4](image)

**Figure 3.** Primary energy use of the materials used

**Figure 4.** the total GWP emission (left) and the total primary energy use (right)
It should be mentioned that the calculated energy use of Building type I amounts to 92.96 kWh∙m\(^{-2}\) per year while Building types II and III have values of 61.81 and 56.74 kWh∙m\(^{-2}\) per year, respectively. It should be mentioned that the Swedish National Board of Housing, Building and Planning (Boverket) prescribes a value of less than or equal to 110 kWh∙m\(^{-2}\) per year is for the energy consumption of residential buildings [29]. This value includes the energy for heating, energy for space cooling and domestic hot water. It should be considered that the results of figure 4 (right) illustrate that the effect of reducing the operational energy use of the building clearly outweighs the energy use of the materials from cradle-to grave.

4 CONCLUSIONS

It has been shown that the insulation materials used in a building have a significant effect on the Global Warming Potential and the primary energy of the building materials. It can also be stated that this effect can be improved by the choice of insulation material and that vacuum insulation panels are a very competitive alternative not at least since they may offer the possibility to save some living space area.

Another result of this study is the substantial difference between an ordinary building and a well-insulated building when looking at the sum of the primary energy of the materials and the operational energy of the building and that the use of high thermal performance envelopes is strongly motivated.

ACKNOWLEDGEMENTS

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[29] The Swedish Building Regulations (regulations and guidelines), the Swedish National Board of Housing, Building and Planning (Boverket) BFS 2011:6 BBR 18
SYSTEM MODEL OF MOULD GROWTH ON BUILDING MATERIALS

Yujing Li (1)

(1) Division of Building Materials, Faculty of Engineering, Lund University, Lund, Sweden – Yujing.Li@byggtek.lth.se

Abstract

During the recent decades indoor mould is one of the major concerns for the indoor environment and the sustainability of the material use since it is closely connected to the health issues for the building occupants. This study presents a system dynamic model of mould growth on building materials by taking into account of the physical properties of the building materials and the physiological properties of the mould. This model can be used to show the trend of the mould growth in the buildings. This is an inter-discipline study of the material science and the biological science.

Mould, building materials and the surrounding air are considered as a system. All the influencing factors within the systems and their interactive impacts are analyzed by the system analysis. Based on the analysis a system model is built. The model consists of the moisture properties of the materials: moisture exchanged between the building material and the surrounding environment via desorption and adsorption processes, which are influenced by the hysteresis properties of the material and the temperature. This part is connected to the activities of the mould, as mould consumes the moisture. The physiological properties of mould are also influenced by the moisture states of the material and the surrounding environment, such as temperature, oxygen etc. By providing the climate data, the model can show the trend of the mould growth over the time.

1 INTRODUCTION

Mould in buildings is of interest in relation to indoor air quality as there is evidence that human health can be influenced by indoor microbial activity [1]. In order to
prevent mould growth in building components it is essential to understand the correlation between physical parameters of the material and mould growth.

Most of the building materials are porous materials. Their moist state can be influenced by the moisture conditions in the surrounding environment, by absorption (wetting) and desorption (drying) processes. Most of the building materials are organic, or can accumulate particles with organic compounds, i.e., house hold dusts. When the conditions fit, it provides an environment for mould to grow. This study attempts to model the moist state of the material, which is influenced by the surrounding environment by desorption and adsorption processes and the growth of the mould on such material. This is an interdisciplinary study combines the physical properties of the building material and the physiological properties of mould fungi, with system analysis method.

2 METHODOLOGY

2.1 The system analysis and system dynamics

The main method adopted in this study is called “System thinking and systems analysis”. System thinking is the way of understanding relationships and patterns between components in a network of relationships. It is a science based on understanding connections and relations between seemingly isolated things [2]. By involving all the factors (stakeholders) and understanding the causes and effects of the problems, it is possible to understand how dynamic the system works and how the system develops [2].

In this study, we start with a simple system: a piece of porous material, i.e., a piece of wood, is placed in a closed indoor environment. We will look at the relationship between the material and the indoor air. Other factors, such as the air exchanged between the indoor and outdoor air, are not taken into account for this stage. There are already a small amount of mould spores available on the material as it normally exists in any indoor environment.

The general components taken into account in this system are listed in the causal loop diagram [2] (CLD). The positive sign “+” on the arrow indicates the same trend of change, i.e., if A increases then B increases; or if A decreases so B decreases.

2.2 How does mould grows

Mould growth is influenced by many factors. The three most important ones are nutrient availability (substrate), moisture state and temperature. Water plays a key role in the mould growth on building materials [3]. The growth of one specific mould varies under different relative humidity levels. Different moulds have different moisture optima and behave differently under moisture changes [4].
The mould growth on the material requires the water from the material. When the water activity is higher than the thresholds for mould to grow, higher water activity in the material normally leads to better growth for mould. At the same time the mould growth consumes water, which decreases the water activities in the material. Mould consumes nutrient from the environment for its growth. Since fungi are aerobic organisms, the availability of oxygen is also important for mould growth [5].

The growth of the mould on a piece of building material can be described in the CLD below in Fig. 1.

**Fig. 1.** The growth of mould on a piece of building material in a causal loop diagram (CLD1).

### 2.3 How the moist state changes in the material

The water in the material can be adsorbed from the air (absorption), when the relative humidity in the air is higher than it in the wood. In the other way, when the water activities in the material is higher than it in the air, the water also diffuses to the air by desorption (drying). The relative humidity in the air is regulated by the temperature in the air. When the temperature is higher, the RH becomes lower. The temperature also influences the mould growth, as shown in the CLD in Fig. 2

**Fig. 2.** The moist exchange between the building materials and the surrounding air in a causal loop diagram (CLD2).
2.4 The system

The mould growth and the moist state changes in the material can be connected since the mould consumes the water in the material, which decreases the amount water available in the material. The system can be described in CLD3 in Fig. 3.

Fig. 3. The mould growth on a piece of building material in a closed environment in a causal loop diagram (CLD3).

3 RESULTS

3.1 Constant temperature scenario

It is possible to estimate the development of the material according to the CLD analysis and draw a trend line of the system [2].

Under the constant temperature condition, if there is only water exchange between the material and the moisture in the air, it will reach a balance, which is the equilibrium of the relative humidity of the air and the water activity of the material. The water activity of the material might look like in Fig. 4A, if the relative humidity in the air was higher than the original water activity in the material, i.e., wetting; of in Fig. 4B, if the relative humidity in the air was lower, i.e., drying.
Fig. 4. The trend line of the moist state of the material in a constant temperature condition, A: the water activities of the material was increased due to absorption. B: the water activities of the material was decreased due to desorption

3.1.1 Drying

If there is a piece of wet material placed in a relative dry condition under the constant temperature. The development of the mould biomass will look like in Fig. 5. At the beginning when the water activity in the material was still relatively high, the mould will increase growing. However, with the drying of the material, the water activity level can no long support the active growth of the mould. The active (living) mould biomass will start decaying.

![Active biomass vs. Time](image)

Fig. 5. The trend of the mould growth on the material during drying

3.1.2 Wetting

If a piece of dry material is place in a high relative humidity environment, the active mould biomass will increase gradually, as shown in Fig. 6.

![Active biomass vs. Time](image)

Fig. 6. The trend of the increasing mould biomass during moisturizing

3.2 Fluctuating temperature

When the temperature changes, the relative humidity of the air changes as well. The RH increases when the temperature decreases and decreases when the temperature increases. The changes of the RH in the air also influence the desorption and absorption between the material and the air, as shown in Fig. 7.
Fig. 7. The RH in the air changes with the temperature change.

The biomass on the material will increase slowly eventually due to the accumulated growth when the RH was high.

4 CONCLUSION

By using the system analysis and drawing the causal loop diagram, it is possible to draw a trend line of the development of the mould growth on building material with various temperature and moisture conditions. The picture can be enlarged to more complex conditions by adding more factors and stakeholders to consider.

However, if a more precise prediction is required, it needs more quantitative data to simulate the model with a computer program.

REFERENCES

Alkali-silica reaction (ASR) is one of the main processes affecting the durability and reducing the service life of concrete structures worldwide. Some laboratory tools were developed over the years for evaluating either the actual damage degree or the potential for further distress of concrete damaged due to ASR and among those, the Stiffness Damage Test (SDT) and the Damage Rating Index (DRI) seems to have a great potential. This work presents the evaluation of either the mechanical or microscopic features of concrete affected by ASR in the laboratory through the use of both SDT and DRI. The analyses were carried out on three types of concrete (25, 35, 45 MPa) and two types of highly-reactive aggregates (Texas sand and New Mexico gravel). Four expansion levels (0.05%, 0.12%, 0.20% and 0.30%), were chosen for analysis over this research. The results show that both tools could efficiently detect the damage’s degree in the samples for all the mixtures analyzed. Moreover, their coupling enables a better understanding about ASR damage and development.

1 INTRODUCTION

Alkali-silica reaction (ASR), one of the most common deleterious mechanisms identified in concrete structures worldwide, consists in a chemical reaction between “unstable” silica mineral forms within the aggregate materials and the alkali hydroxides (Na, K – OH) dissolved in the concrete pore solution. It generates a secondary alkali-silica gel that induces expansive pressures within the reacting aggregate material(s) and the adjacent cement paste upon moisture uptake from its surrounding environment, thus causing microcracking, reduction of material’s properties (mechanical/durability) and, in some cases, functionality in the affected structure [1].

One of the biggest challenges in dealing with aging/deteriorating concrete structures is to establish the correlation between the distress "signatures" caused by a damage mechanism
(i.e. ASR, for instance), and the loss in mechanical properties, physical integrity, durability and performance of the affected material or element, as well as their structural implications.

Recent studies dealing with the evaluation of the mechanical performance of aging concrete suggest that both the Stiffness Damage Test (SDT) and the Damage Rating Index (DRI) can provide a diagnostic evaluation of the “degree of damage” in concrete affected by ASR [2].

2 STIFFNESS DAMAGE TEST (SDT)

From the mid 1980s, Crisp and co-workers used the SDT to quantify the degree of damage in concrete due to ASR [3, 4]. Actually, Walsh [5] had previously reported a good correlation between the crack density and the cycles of loading/unloading (stress/strain relationship) of rock specimens. Based on those results, Crouch [6] then proposed a new test procedure (Stiffness Damage Test - SDT) based on cyclic loading (in compression) of concrete samples (cylinders or cores) with diameters greater than 70 mm (length / diameter of 2.0 – 2.75) [3, 4].

Initially, the SDT involved the application of a stress of up to 5.5 MPa at a rate of 0.10 MPa/s [3, 4]; the authors wanted this testing procedure to be non-destructive, thus enabling the use of the test specimens for further testing. The loading was then controlled by a microprocessor and repeated five times [3, 4]. Crisp and co-workers carried out more than 1000 tests on cores extracted from damaged concrete structures; after analyzing the stress-strain response, they proposed the following as the diagnostic parameters for determining the extent of damage in a specimen [3, 4]:

• Modulus of elasticity (Ec): average modulus of elasticity value of the last four cycles, as concrete samples of damaged concretes presented lower secant modulus of elasticity than undamaged samples;
• Hysteresis area (H, in J/m3): area of the hysteresis loops averaged over the last four cycles, as damaged concrete samples showed greater energy loss (or hysteresis areas) than undamaged samples;
• Non linearity index (NLI): it represents the ratio of the slope of the stress response at half the maximum load over the secant Ec. This parameter provided information about either the extent of damage or the crack patterns of the samples.

Smaoui et al. [7] further evaluated the reliability of the SDT on laboratory concrete samples incorporating a variety of reactive rock types that had reached different expansion levels (stored at 38°C at 100% R.H.). After carrying out many tests, the authors found that the best output response for the SDT was the hysteresis area of the first cycle for test specimens loaded up to a maximum of 10 MPa. They mentioned that the lower stress level evaluated (5.5 MPa) did not allow the ASR-induced microcracks to stress (reclose) sufficiently to enable drawing statistically reliable information on the level of ASR expansion reached by the tested concrete. The authors also found that the correlation between the expansion and the plastic deformation obtained after the five loading/unloading cycles was fairly satisfactory. However, they noted significant variations for either the hysteresis area or the plastic deformation for concrete specimens incorporating different types of reactive aggregates. These differences were possibly associated to the nature of the aggregates selected (fine or coarse) and differences in the internal pattern of damage, as they can generate their own reaction mode (i.e. pattern/density/orientation of cracking depending on whether the damage is generated in the fine or coarse aggregate, or by different rock types, etc.).
Sanchez et al. [8], following the initial works, kept studying the SDT procedure on concrete mixtures cast with both different reactive aggregates (coarse vs. fine) and concrete mix-designs (25, 35 and 45 MPa). The goal of those works was to verify the influence of either the test loading or several input parameters (concrete environment, humidity, specimen size, test loading, etc.) on the output test analyses. Likewise, the evaluation of the output test responses against the expansion levels of affected specimens was performed.

After carrying out a heavy testing matrix, the authors presented the following main conclusions: 1) SDT should be carried out with a percentage of the concrete strength instead of using a fixed load; 2) the use of 40% of the concrete strength seems to be the best approach in the procedure for distinguishing damaged concrete specimens towards their expansion levels; 3) the use of percentages up to 40% of the concrete strength enables the use of the same specimen for supplementary analyses such as compressive or tensile strength, the test seems to keep its “non destructive” character up to this point; 4) the output parameters such as the hysteresis area and the plastic deformation over the five cycles as well as the modulus of elasticity (as an average value of the second and third cycles) were chosen as the most diagnostic output results of the test; 5) the input parameters such as the concrete’s cure history (i.e. the specimen humidity), the sample’s geometry and size, the sample’s environment (zone and direction) as well as the choice of the sample’s strength prior testing seems to strongly influence the output analyses of the SDT and; 6) the use of indices (SDI and PDI) instead of absolute values, which take into account the ratio “dissipated energy/total energy” implemented in the system, better represents the real “damage” of affected materials Likewise, this approach decreases the impact of the loading choice and provides easier understanding of AAR evolution as a function of its expansion.

3 DAMAGE RATING INDEX (DRI)

The DRI is a microscopic analysis performed with the use of a stereomicroscope (about 15x magnifications) where damage features generally associated with ASR are counted through a 1 cm$^2$ grid drawn on the surface of a polished concrete section. The number of counts corresponding to each type of petrographic features is then multiplied by weighing factors, whose purpose is to balance their relative importance towards the mechanism of distress (for instance ASR). It is important to mention that the factors used in the method were chosen on a logical basis, but relatively arbitrarily [9]. Ideally, a surface of at least 200 cm$^2$ should be used for DRI analysis, and it may be greater in the case of mass concrete incorporating larger size aggregate particles. However, for comparative purposes, the final DRI value is normalized to a 100 cm$^2$ area [9, 10]. DRI results are often represented by charts enabling easy visualization of the different damage features in the specimen under study.

Recent studies dealing with the DRI indicated that the variability between the operators performing this test could be significantly reduced by improving the definition/description of the different damage features, modifying some weighing factors, as well as by appropriate training of the petrographers using reference sections [9]. These authors proposed to use identical factors for the two categories of opened cracks in the aggregate particles (factor of 2) or cracks in the cement paste (factor of 3), with or without reaction products; this was done to reduce the variability associated to the difficulty in positively recognizing the presence of reaction products in cracks of the polished sections. Consequently, the two types of cracks could be grouped together having the same weighing factors if one considers that a crack is an
indication of damage, either with or without reaction products. Also, larger weighing factors were selected for cracks in the cement paste, compared to that in the aggregate particles (i.e. factor of 3 vs. 2), to indicate a relatively greater importance regarding the durability of the affected concrete element. Finally, it was found that eliminating the counts of the number of voids with reaction products in the cement paste and the reaction rims from the calculation of the DRI values also contributes at reducing the variability between the operators and are not really direct indications of “damage” in concrete.

Since the process of damage generation vary through the type/nature of reactive aggregate used (fine vs. coarse aggregate, lithotype, etc.), the DRI should ideally assess the nature and degree of distress features and correlate them with either the expansion attained by the distressed concrete or, ideally, with losses in mechanical properties [11-13]. Such information is, however, currently very limited.

4 SCOPE OF THE WORK

This paper specifically presents and discusses the results of the basic evaluation of the applicability of the Stiffness Damage Test (SDT) and the Damage Rating Index to detect/quantify damage in concrete when deleterious expansion due to ASR is generated either in the coarse or in the fine aggregate material. In order to best achieve this objective, extensive testing was performed on (well-controlled) laboratory-made and cured concrete specimens of different mix designs (25, 35 and 45 MPa) and incorporating coarse or fine reactive aggregates.

5 MATERIALS AND METHODS

5.1 Materials and mixture proportions

Three types of non air entrained concrete mixtures (25 MPa, 35 MPa and 45 MPa mix design strengths) and two highly-reactive aggregates (New Mexico gravel-NM and Texas sand-Tx) were selected for the study. The coarse aggregates ranged from 5 to 20 mm in size. Non-reactive fine and coarse aggregates were used in combination with the above reactive aggregate materials for concrete manufacturing. Table 1 gives the detailed concrete mixture proportions.

After casting, the specimens were placed for 48h in the moist curing room (i.e. after the first 24h in the moist curing room, the specimens were demolded and left in this room for a further 24h). Small holes, 5 mm in diameter by 15 mm long, were then drilled in both ends of each test cylinders and stainless steel gauge studs were glued in place, with fast-setting cement slurry, for longitudinal expansion measurements. After completion of the first 48 h at 23°C, the “0” length reading was performed and the specimens were placed in sealed plastic (22 litters) containers lined with damp cloth (4 cylinders per bucket). All buckets were then stored at 38°C and 100% R.H., and all the test cylinders were monitored regularly for length variations. Moreover, as per ASTM C 1293, all containers were cooled to 23°C for 16 ± 4 h prior to periodic axial expansion measurements. Then, test cylinders were removed from the high-temperature storage conditions for SDT and DRI testing when they reached the expansion levels chosen for this research, i.e. 0.05% ± 0.01%, 0.12% ± 0.01%, 0.20% ± 0.01% and 0.30% ± 0.01%. Overall, ASR expansions among the test cylinders of a same batch were only slightly variable, as the entire test set was cast at the same time and from the same large concrete batch [8].
Table 1: Concrete mix designs used in the study

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>25 MPa - Materials (kg/m$^3$)</th>
<th>35 MPa - Materials (kg/m$^3$)</th>
<th>45 MPa - Materials (kg/m$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Texas sand</td>
<td>Texas sand</td>
<td>Texas sand</td>
</tr>
<tr>
<td>Cement</td>
<td>314 (101)</td>
<td>314 (101)</td>
<td>370 (118)</td>
</tr>
<tr>
<td>Sand</td>
<td>790 (304)</td>
<td>714 (264)</td>
<td>714 (264)</td>
</tr>
<tr>
<td>Coarse aggregate</td>
<td>1029 (384)</td>
<td>1073 (424)</td>
<td>1029 (384)</td>
</tr>
<tr>
<td>Water</td>
<td>192 (192)</td>
<td>192 (192)</td>
<td>174 (174)</td>
</tr>
</tbody>
</table>

The number in brackets correspond to the volume occupied by the materials (in L/m$^3$).

Once the above expansion levels were reached, the specimens were wrapped in plastic film and stored at 12°C until testing (because of testing capacity issues). Prior to testing, both ends of each cylinder were carefully mechanically ground to avoid any interference from the stainless steel gauge studs used for expansion measurements. Also, even though they were wrapped in plastic film prior to testing, the specimens were restored for 48h in the moist curing room, protected from running water, before stiffness damage testing, in order to allow appropriate saturation of the test specimens, following the procedure proposed for concrete cores extracted from real concrete structures (CSA A23.2-14C). Length and mass readings were also performed on a number of test specimens prior and upon unwrapping to make sure that they had not suffered from significant shrinkage or expansion over the storage period prior to testing. The monitoring of the test specimens showed that the 12°C storage resulted in slight shrinkage (- 0.02 ± 0.01%) and mass loss (0.7 ± 0.2%) of the test specimens, which was recovered through the 48-hour re-saturation period prior to running stiffness damage testing.

5.2 Methods for assessment and analysis

5.2.1 Stiffness damage test (SDT)

Test cylinders were subjected to five cycles of loading/unloading at a controlled loading rate of 0.10 MPa/s. All the specimens were tested at a strength level corresponding to 40% of the 28-day concrete mix design strength, according to [8]. The results presented in this paper correspond to the average values obtained on three affected specimens at a given expansion level.

5.2.2 Damage Rating Index (DRI)

The DRI was performed on specimens cast from all concrete mixtures and for all expansion levels selected for this work. Counts of cracking in the aggregate particles were made in particles down to 1 mm in size, instead of 2 mm normally used in the original method [9]. The latest weighing factors proposed by Villeneuve et al. [9] were used for the calculation of the DRI output values.

6 RESULTS

6.1 Stiffness damage test

Sanchez et al. [9] proposed the use two indices instead of absolute values over the SDT: SDI and PDI; where the first index represented the dissipated energy (over the five compression cycles)/the total energy implemented in the system (area under the stress/strain curve), and the second represented the plastic deformation (over the five compression cycles).
cycles)/total deformation in the system. Figure 1 presents the results of those indices against the expansion levels of all the specimens used in this work.

Looking at the SDI values (Figures 1A), one observes that this index may distinguish quite well the different expansion levels (or different amounts of internal cracking) for all the concrete mixtures, aggregates and strengths. In general there is either a concave or linear trend (depending on the concrete strength) of this variable against the expansion level of affected specimens, ranging from about 0.08 for sound concretes up to 0.35 for 0.30% of expansion.

Considering the PDI values, one notices that it also can well distinguish the different expansion levels (or different amounts of internal cracking) for all the concrete mixtures, aggregates and strengths, ranging from about 0.05 up to 0.30 for 0.30% of expansion. Likewise, this variable is mostly characterized by either a concave or linear trend towards the expansion levels of the affected samples.

A – 25, 35 and 45 MPa mixtures: SDI vs. expansion

B – 25, 35 and 45 MPa mixtures: PDI vs. expansion

Figure 1: Stiffness damage indices for the 25, 35 and 45 MPa mixtures: A) SDI results. B) PDI results.

### 6.2 Damage Rating Index

The detailed results of the DRI determinations, i.e. in terms of the relative importance of each of the various petrographic features of deterioration are illustrated in Figure 2. At first glance, the following general trends can be observed from the below graphs: 1) a commonly found feature of “deterioration” in all polished sections corresponds to *Closed cracks* within the aggregate particles (CCA - blue charts). In the case of Tx mixtures, this distress feature type is even more apparent; 2) a progressive increase in the number of cracking within both the aggregate particles (OCCA, CCAG - red and green charts) and the cement paste, with and without gel (CCP, CCPG - orange and light blue charts) is found with increasing expansion of the test specimens; 3) the DRI values correlate well with the expansion levels measured for all the mixtures and aggregates selected for the study. Moreover, it appears that the DRI values do not change significantly as a function of the concrete strength as all the mixtures presented quite similar results for all the expansion levels studied; 4) the DRI values seem to be not
affected by the deleterious expansion process whether it originates from the fine (Tx) or the coarse (NM) aggregate; 5) an interesting behavior could be noticed for the 45 MPa concrete mixtures. Higher DRI numbers (than the obtained for the 25 and 35 MPa concretes) were found at low expansion levels, yet they remained stable up to 0.12%, while increasing almost linearly and similarly to the other mixtures from about 0.12% up to the highest expansion levels tested in this study (0.30%) and; 6) the data below indicate that a certain degree of damage already exists in the test specimens (DRI ranging between 100 and 140, from some cracking in the aggregate particles and in the cement paste) for concrete specimens showing no significant expansion level (indicated 0% in Figures 2B, 2E).

Figure 2: DRI charts for all the mixtures analyzed over the study: A, B, C. Texas sand. D, E, F. NM gravel + Lav. The common legend for all graphs is given in Figure A.

7 CONCLUSIONS

- Both tools (SDT and DRI) were able to distinguish quite well the different ASR expansion levels chosen in this work, illustrating respectively important mechanical and microscopic information about either the cracking extent or the cracking features characteristics of ASR distress development;
- The microscopic and mechanical data seem to correlate quite well against the expansion levels studied, which seems to emphasize the complementary character of those tools;
- Finally, although the SDT and DRI were originally developed for assessing the effects of ASR in concrete, the test certainly has the potential of evaluating distress due to other deleterious mechanisms such as freezing and thawing, action of fire, impact loads, DEF, etc. However, an in depth study is necessary to proof its efficiency.
ACKNOWLEDGEMENTS

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AN IN-SITU AND LABORATORY STUDY OF THE EFFECT OF THE INTRINSIC PROPERTIES OF MORTARS ON THEIR POTENTIAL BIORECEPTIVITY

T.H. Tran\(^{(1,2,3,4)}\), A. Govin\(^{(2)}\), R. Guyonnet\(^{(2)}\), P. Grosseau\(^{(2)}\), C. Lors\(^{(3,4)}\), D. Damidot\(^{(3,4)}\), O. Deves\(^{(5)}\) and B. Ruot\(^{(5)}\)

(1) Department of Civil Engineering, Duy Tan University, Danang, Vietnam – tranthuhien197@gmail.com

(2) UMR 5307, LGF, Ecole Nationale Supérieure des Mines de Saint Etienne, Saint Etienne, France – govin@emse.fr; guyonnet@emse.fr; grosseau@emse.fr

(3) Université Lille Nord de France, Lille, France

(4) Laboratory LGCgE-GCE, Ecole Nationale Supérieure des Mines de Douai, Douai, France - christine.lors@mines-douai.fr; denis.damidot@mines-douai.fr

(5) Centre Scientifique et Technique du Bâtiment, Université Paris Est, Marne-la-Vallée, France – deves@cstb.fr; ruot@cstb.fr

**Keywords:** biofouling, mortar, intrinsic properties, laboratory tests, in-situ tests.

**Abstract**

This study aims to clarify the effect of mortar intrinsic properties (porosity, roughness and carbonation level) on its ability to biofouling. Two scales experimental tests, an accelerated fouling in laboratory and a natural fouling in the real-world, were set-up. The first one was conducted in a closed device allowing a periodic sprinkling of an algal suspension on the samples surface. The outdoor test samples were exposed in a park at Grenoble (France). The colonization rate of the sample surface was evaluated by image analysis. The results show that the impact of each intrinsic parameter is quite different as function of the test. The porosity has no influence on the algal colonization of the samples exposed in indoor whereas a high porosity seems to increase slightly the bioreceptivity of ones exposed outdoor. The roughness, in both tests, promoted the microorganisms attachment and so their colonization. However, the discrimination of roughness grades is higher in the laboratory test than in the in-situ one. The surface pH significantly influences on the accelerated biofouling but not on the in-situ one. These dissimilarities result from the difference in experimental configurations of the two tests. Thus the laboratory test should be adjusted to be more suitable and to allow an extrapolation of results.
1 INTRODUCTION

Mortar and concrete are common material for building envelope. After a certain time, buildings are usually subjected to the colonization by microorganisms. Among the involved microorganisms, the chlorophytes (green algae) and the cyanophytes (blue-green algae) are the main and the initial colonizers [1-4]. This biological colonization induces an esthetical problem and can in later stage, degrades the building-materials by physical or chemical mechanisms [5].

The biofilm composition and the colonization rate depend on different parameters such as the material properties, the building architecture and the global and local environment around the building. In general, the conditions that favor the water retention and the access of material surface to the biological propagules, promote the implantation and the growth of microorganisms. For example, in rainy regions and during heavy precipitation season, the facades represent a higher susceptibility to biofouling [6]. The north-facing facades which are wetter and less sunny, get colonize faster [4,6]. Several authors notify the permanent biofouling observed at the foot of walls, the junction of different coatings and the overhanging elements (cornices, moldings, balconies, etc. …) [4,7]. Furthermore, the humid environments near a lake, a river, trees and shrubs, are favorable to the appearance of stains on the walls.

The intrinsic properties influencing on the susceptibility of material to microorganisms colonization is defined as bioreceptivity [8]. It can be divided into physical properties (porosity, roughness, hydrodynamic properties…) and chemical ones (chemical composition, pH surface…). Several studies have investigated the role of material characteristics since the last thirty years [4,9]. However, most of them focused on laboratory scale experiments. The conditions of these tests (illumination, humidity, inoculation…) were chosen in order to accelerate the biological colonization process on the sample surface. Few researcher were performed to examine what happens in the real world, i.e. at field-scale which requires many years. So, correlations between these two experiment scales are scarce. Furthermore, to accelerate the laboratory test, the cementitious material is often aged by carbonation and/or leaching process. However, the effect of the accelerated aging is rarely evaluated.

This work aimed to study the influence of intrinsic characteristics of mortar (porosity, roughness, carbonation state) on its biofouling. Two test scales are conducted: an accelerated laboratory test and a test by exposition of samples to natural conditions (in-situ).

2 MATERIALS AND METHODS

2.1 Preparation and characterization of materials

The investigated materials are mortars composed of 30 % Portland cement (CEM I 52.5), 65 % siliceous sand (0.1/0.35) and 5 % calcareous filler. To this dry mixture, 0.27 % cellulose ether (Hydroxylethyl Methyl Cellulose) was added. In order to obtain different porosities without changing the mortar formulation, two water/cement (w/c) ratios, 1 and 1.2, were used.

The fresh mortar was casted into 50 x 50 x 1 cm$^3$ expanded polystyrene molds and stored at 20 ± 1 °C and 95 ± 5 % of relative humidity. After 28 days of curing, the mortar was cut into 20 x 8 x 1 cm$^3$ and 30 x 20 x 1 cm$^3$ specimens for accelerated tests and in-situ tests, respectively. To investigate the effect of carbonation, the mortars were cut in same size specimens, after only 7 days of storage, and were then exposed to an accelerated carbonation until reaching a surface pH of about 9. The carbonation was performed under pure CO$_2$ at 20 ± 1 °C and 65 ± 5 % of relative humidity.
Different surface roughnesses were achieved by three finishing methods of mortar during its setting. The first consisted in smoothing the surface of fresh mortar by a ruler and the two others in scratching the surface of mortars during setting with two sponges of different roughness.

The porosity was determined by mercury intrusion porosimetry. For each mortar, three samples were measured after prior drying by acetone.

The surface pH was measured thanks to a surface pH electrode (Sentix Sur).

The roughness of the mortar was evaluated by means of an optical profilometer and characterized by the arithmetic average of the height ($R_a$) [10].

2.2 Biofouling tests

2.2.1 Laboratory scale tests

The laboratory fouling test was performed by means of a closed device containing an algal suspension. The microalga *Klebsormidium flaccidum* was selected because of its representativeness [4,11-13]. Furthermore, *K. flaccidum* is easy to cultivate.

The initial concentration of algal suspension was fixed to 4 mg/ml (of dry masse) and 50 L of this suspension was introduced into the test chamber. Thanks to a pump system, the sample surfaces were sprinkled for 90 min every 12 h by this suspension. The light was provided by two neon lamps. The photoperiod was fixed to 12 h light/12 h dark. The details of the experimental approach were described in previous papers [14,15].

In laboratory experiments, specimens were tested in triplicate for each material. Carbonated and uncarbonated samples were tested separately.

2.2.2 Field scale tests

In the field-scale experiments, the mortar samples were placed on a stainless steel frame in a private green park close to Grenoble, France. In order to favor the biological colonization, the samples were placed near trees, facing to north and inclined with an angle of 45 °. Furthermore, to avoid splashing during rainy periods, the specimens were arranged in two rows of which the first was positioned at 1 m above the ground. The contamination between specimens was also avoided.

For each material, three specimens were tested. The mortars with a w/c ratio of 1 were studied at carbonated and uncarbonated states, with the three roughnesses. For the samples with a w/c ratio equal to 1.2, only the carbonated mortars with the two highest roughnesses were examined.

2.3 Biofouling evaluation

The fouled area was determined by means of image analysis. Each specimen was digitalized thanks to an office scanner every weekday and at least every two months for bench-scale and field-scale experiments, respectively. The RGB color space images obtained were transformed thanks to Aphelion software into YIQ color space to improve the detection of fouled surface. The biological coverage was well detected on the grey level image of the Q-channel by thresholding. The colonization rate was calculated by the ratio of the colonized area to the total area. More details about this method can be found in [14].
3 RESULTS AND DISCUSSION

3.1 Material characteristics

The characteristics of mortars are given Table 1. For both uncarbonated and carbonated mortars, the increase in the w/c ratio induced a rise in the total porosity. Indeed, the porosity increases from 37 % to 39 % for uncarbonated mortars and from 32 % and 36 % for carbonated mortars when w/c increases from 1 to 1.2. The accelerated carbonation leads to a reduction of surface pH from 11 for uncarbonated mortars to 9 for carbonated. Three levels of roughness were obtained whatever the carbonation state and the w/c ratio. The roughnesses are around 30µm, 50µm and higher than 70µm for the lowest, the intermediate and the highest roughnesses, respectively.

Table 1: Characteristics of mortars

<table>
<thead>
<tr>
<th>Sample</th>
<th>w/c ratio</th>
<th>Porosity (%)</th>
<th>Surface pH</th>
<th>Roughness $R_a$ (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1UC-R1</td>
<td>1</td>
<td>37.2 ± 0</td>
<td>11.0 ± 0.4</td>
<td>29 ± 5</td>
</tr>
<tr>
<td>1UC-R2</td>
<td>1</td>
<td>38.9 ± 0.3</td>
<td>11.0 ± 0.3</td>
<td>47 ± 6</td>
</tr>
<tr>
<td>1UC-R3</td>
<td>1.2</td>
<td>32.1 ± 1.9</td>
<td>9.0 ± 0.1</td>
<td>29 ± 5</td>
</tr>
<tr>
<td>1.2UC-R1</td>
<td>1.2</td>
<td>36.2 ± 0.1</td>
<td>8.9 ± 0.1</td>
<td>47 ± 6</td>
</tr>
<tr>
<td>1C-R1</td>
<td>1.2</td>
<td>32.1 ± 0.1</td>
<td>9.0 ± 0.1</td>
<td>32 ± 4</td>
</tr>
<tr>
<td>1C-R2</td>
<td>1.2</td>
<td>38.9 ± 0.3</td>
<td>11.0 ± 0.3</td>
<td>47 ± 6</td>
</tr>
<tr>
<td>1C-R3</td>
<td>1.2</td>
<td>36.2 ± 0.1</td>
<td>8.9 ± 0.1</td>
<td>145 ± 18</td>
</tr>
</tbody>
</table>

3.2 Colonization of cementitious material surfaces by microorganisms

An illustration of the algal colonization in both tests is shown Figure 1.

Figure 1: Illustration of algal colonization over time in laboratory test (A) and in-situ test (B), for 1.2C-R3.

In the accelerated test, the growth of *Klebsormidium flaccidum* generates a dense and velvety mat on samples as illustrated in Figure 1. The first algal spots appear at privileged sites of the surface, such as air bubble holes or asperities formed by the roughness. The extension of fouling results from the growth of the first spots and the adhesion of new ones. On the lowest roughness samples (R1 and R2), the algal growth forms streaks due to the
suspension flow. This fouling form is usual on the building facades. For the roughest mortars (R3), the fouling follows the surface asperities.

In the field-scale tests, the samples were exposed to natural conditions from summer 2009 (26th of June). During almost one year, no visible biological colonization was observed on the samples. The first settlement appeared on the mortars 1.2C-R3. The main colonizer was attributed to green algae. Indeed, the biofouling of all the in-situ samples was green, which is the characteristic color of green algae. The colonization rate of 1.2C-R3 mortars is shown in Figure 2B. The curve can be divided into four steps: a latency step, a growth step, a stagnation step and a decrease step. The latency time was about 11.6 months. Furthermore, the biological growth accelerated between 12 and 16 months of exposure corresponding to the autumn period (from August to November). In fact, the propagules of microorganisms might be already settled on the surface since the spring. Then, mild temperatures coupled with strong light and heavy rainfall during this autumn favored the biological growth. After a short period of stagnation, from 16 to 18 months of exposure, the colonization rate decreased. In fact, the microorganisms detached from the surface. Cell aging and cell death due to the low ambient temperatures, the low light densities, the snow and the effect of outdoor conditions themselves, disrupted the anchoring between the microorganisms and the sample surface.

### 3.3 Effect of intrinsic characteristics of mortar

#### 3.3.1 Porosity

The effect of the w/c ratio (1 and 1.2), and thus the porosity, on the biofouling rate of carbonated mortars is shown in Figure 2.

![Figure 2: Effect of porosity (w/c ratio) and roughness on the colonization rate for carbonated mortars in laboratory (A) and field tests (B).](image-url)

In the bench-scale experiment (Figure 2A), the algal colonization curve is of sigmoid type, characterized by three steps: a latency step, an exponential growth step and a stagnation step. For each roughness (R1, R2 and R3), the colonization rate is very close regardless the w/c ratio, especially, the roughest mortars (R3) where the two curves almost coincide. The algal colonization appears after 6 testing days and the entire surface is colonized after 30 days. For the two others roughnesses (R1, R2), because of experimental standard deviation, the colonization was considered identical whatever the w/c ratio.

In this laboratory test, porosity does not influence on the colonization rate, although the literature highlights an increase in bioreceptivity of porous mortars due to their water uptake and water retention behavior. This contradiction was allocated to the experimental protocol rather than to the modest difference in porosity between the two formulations. Indeed, relative humidity in the test chamber was permanently very high (i.e. ranging from 80 to 100 %). In
these conditions, water content into the samples was always abundant and thus no longer a limiting factor for the biological development. This observation was confirmed by previous studies regarding mortars made with $w/c$ ratios equal to 0.5 and 1 [14,15]. Despite of the considerable difference in porosity, these mortars exhibited identical bioreceptivity.

Unlike in the accelerated test, the $w/c$ ratio and thus the porosity exhibit an influence on the biofouling of in-situ samples (Figure 2B). Indeed, the visible microorganisms develops faster on the more porous mortars ($w/c = 1.2$). Between 11 and 13 months of exposure, while the biofouling appeared on the 1.2C-R3 mortars, no biological colonization was observed on the 1C-R3 ones. The following measurement, realized two months later, showed a colonization rate of 80 % for 1.2C mortars against only 30 % for 1C mortars. For the roughness R2, the effect of the $w/c$ ratio was intermediate. One month later, only the more porous mortar was totally colonized. The results are in agreement with the literature since the mortars made with the highest $w/c$ ratio were the most porous and exhibited a greater bioreceptivity [11,16,17].

### 3.3.2 Roughness

In the accelerated test, the biofouling rate was obviously influenced by the surface roughness of samples and the effect of the three levels of roughness was quite distinct (Figure 2A). Indeed, the colonization rate increases, while the latency time decreases with the roughness. The latency time was around 18, 9 and 6 days of testing, and the complete colonization was achieved in 56, 51 and 30 days for roughnesses R1, R2 and R3, respectively. These results are in agreement with previous studies [14,17]. By providing asperities, the roughness favors the physical anchorage of microorganisms provided by the suspension. Consequently, rougher is the surface, earlier the biofouling begins and faster the colonization is.

The roughness impacted also the biofouling of mortars exposed in the real-world by increasing the colonization kinetics (Figure 2B). However, only two grades of roughness were observed. Indeed, no difference in bioreceptivity to visible photosynthetic organisms was noticed for roughnesses R2 and R3. Conversely, roughness R1 exhibited a lower colonization rate. Therefore, the biofouling is less affected by the roughness in the field-scale test than in the bench-scale test that could be explained by a much less intense natural inoculation. Furthermore, contrary to the bench-scale test, for a given $w/c$ ratio, the latency time was quite similar whatever the roughness. This could be the result of the mutual impact of the season, the external relative humidity and temperature in natural conditions. Thus the effect of roughness on the latency time is hided.

### 3.3.3 Accelerated carbonation

Figure 3 illustrates the evolution of the colonization rate for carbonated and uncarbonated mortars made with a $w/c$ ratio of 1, for the lowest and the highest roughnesses (R1 and R3).

In the accelerated fouling test, the carbonation appears as the most discriminating parameter influencing the bioreceptivity of material by significantly shortening the latency time and accelerating the kinetics of algal colonization. Indeed, thanks to carbonation, the latency time decreases from 46 to 18 days and from 26 to 7 days for the roughnesses R1 and R3, respectively and the necessary time to reach the complete colonization is reduced (51 days for 1UC-R3 against 30 days for 1C-R3). The total colonization of 1UC-R1 was not achieved because of a precocious stop of testing. Nevertheless, the slope of the curve was greater for carbonated mortars than for uncarbonated ones.
Unlike the effect of roughness which favors the ability of algae to physically cling to the surface, the carbonation affects the biological metabolism. Carbonation, by decreasing the surface pH and thus the pH of the algal suspension, creates less alkaline environment and thus less stressful conditions for algae *K. flaccidum* [14,15]. Consequently, carbonated mortars are highly receptive to colonization by microorganisms.

Figure 3: Effect of accelerated carbonation on the colonization rate for mortars made with a w/c ratio of 1, with roughness R1 and R3, in laboratory (A) and field tests (B).

In the field-scale test, the accelerated carbonation did not result in different growth kinetics, since the biological growth of phototrophic microorganisms evolved quite identically (Figure 3B). Indeed, whatever carbonation state of mortars, the latency time was about 13 months for all the formulations, and 4 months later, the total colonization was reached for all rough samples (R3) and 60 % for all smoothest samples (R1).

The favorable climatic conditions and sufficient inoculation were perhaps not met at the beginning of testing. Indeed, the samples were exposed through the summer, thus, spring, the most favorable season for spreading and growth of microorganisms, was past. Hence, the microbial colonization of carbonated samples was not initiated despite a favorable surface pH. After one year of exposure, the mortars were aged and weathered by leaching and natural carbonation. Consequently, the same surface pH (pH ≈ 8) was measured for all the samples whatever their initial carbonation state. The bioreceptivity of mortars was thus identical when favorable conditions to the biological development were satisfied. Therefore, the influence of the initial surface pH on biofouling is completely inhibited.

4 CONCLUSIONS

By both laboratory and *in-situ* tests, the effect of the intrinsic parameters of mortars on their biofouling by photosynthetic organisms has been investigated.

The impact of porosity on biofouling of mortars was different for two experimental test scales. This parameter did not affect the colonization rate in laboratory test due to the experimental conditions. However, although more experiments of verification still required for the field-scale test, high porosity seems to favor the biological colonization.

For the both test scales, the influence of the roughness is evidenced. A rough surface enhances the biological attachment. The discrimination of roughness grades was better in the accelerated tests than in the field-scale ones, which could be explained by an intense inoculation of the accelerated test against a natural inoculation in the *in-situ* test.

The accelerated carbonation was considered as a decisive parameter in the laboratory test, by significantly promoting the algal growth and by accelerating the colonization rate. While
in natural conditions, due to numerous uncontrolled and random environmental factors, the effect of the carbonation was not observed.

The divergence of results between the laboratory and field-scale tests prevents to correlate the two experimental scales. These dissimilarities could be remediated by improving the experimental protocol such as a decrease in the inoculation intensity, a drying phase of samples between two sprinkling cycles in the accelerated test or a reasonable choice of the beginning of sample exposure to natural environment.

REFERENCES

INFLUENCE OF MATERIAL CHARACTERISTICS ON ELECTROCHEMICAL REALKALISATION OF CARBONATED CONCRETES

Philippe H. L. C. Ribeiro (1), Gibson R. Meira (2) and Pablo R. R. Ferreira (2)

(1) Civil and Environmental Engineering Postgraduate Programme - Federal University of Paraíba, João Pessoa, Brazil – phlcr_jp@hotmail.com
(2) Federal Institute of Education, Science and Technology of Paraíba, João Pessoa, Brazil – gibsonmeira@yahoo.com; pablo.r06@hotmail.com

Abstract
This work studied the influence of concrete characteristics such as cement type and water to cement ratio, and concrete cover thickness on the electrochemical realkalisation of carbonated concretes. Reinforced concrete specimens were cast with high early strength Portland cement and Portland pozzolan cement and w/c ratios of 0.55 and 0.65. Reinforced concrete covers of 1 and 2 centimetres were also adopted. The specimens were previously carbonated in a carbonation chamber until reinforcement depassivation and afterwards were subjected to realkalisation procedures under an average cathodic current density of 2 A/m² in respect to the exposed concrete surface. Results show the treatment efficiency for recovering concrete alkalinity. More time and total electrical charge passed are demanded in proportion as w/c ratio decreases, concrete cover thickness increases and when concrete has lower alkalinity reserve. The tendency of reinforcement reaching the passive condition is observed by corrosion potential measurements and concretes that demand more charge and time for realkalisation reach the passive condition in a shorter time. Corrosion current density shows a decrease tendency over time and, although not reaching the level that represents passive condition in most of the cases, it was not far from that.

1 INTRODUCTION
Concrete is a material that usually presents high levels of alkalinity, which helps on the formation and protection of passivation film on metal surfaces in reinforced concrete structures. Since this initial condition of high alkalinity and the concentration of depassivating ions below a certain threshold are maintained, corrosion process does not start [1]. However, this protective film can be broken down due to carbonation or in the presence of concentrations of chloride ions higher than a certain critical content [2]. In the specific case of concrete carbonation, the pH goes down due to carbonates precipitation as a consequence of hydroxides and other hydrated phases reaction with carbon dioxide [3].
As a rehabilitation method or as a preventive treatment, the realkalisation method has been used to recover concrete alkalinity nearby the reinforcing region [4]. The advantage of this method is that when the treatment finishes the external electrical system used for the method application is dismantled and concrete structure can follow its role without larger destructive interventions as in conventional parch repair treatment [5].

Studies about realkalisation treatment which focused on understanding the phenomenon characteristics, like transport mechanisms involved [6-7], efficiency on reinforcement repassivation [8-9], efficiency of different electrolytes on realkalisation [10], effects on concrete properties [11] and side effects [12-13], have been carried out during the last years. However, the effectiveness of the treatment in repassivating the reinforcement has been questioned [14-15] and, although some work is being carried out on this specific topic [16], there is still not a consensus and more research is necessary. Moreover, the influence of material or structure characteristics on the method behaviour and its final results was studied at a lower level [16].

Taking into account this gap in knowledge, this work studies the influence of cement type, water to cement ratio and reinforcement concrete cover on electrochemical realkalisation of carbonated concretes and its efficiency on repassivation of reinforcements.

2 EXPERIMENTAL WORK

2.1 Casting and curing of concrete specimens

Prismatic concrete specimens with 8 x 8 x 8 cm were cast using Brazilian Portland Cements V (high early strength Portland cement) and IV (Portland pozzolan cement), which physical and chemical characteristics are presented in Table 1 as cements I and II, respectively. Concrete mixtures and properties are presented in Table 2.

Table 1 – Chemical composition and physical properties of cements.

<table>
<thead>
<tr>
<th>Composition/property</th>
<th>Cement I</th>
<th>Cement II</th>
</tr>
</thead>
<tbody>
<tr>
<td>SO₃ (%)</td>
<td>3.19</td>
<td>2.69</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>18.96</td>
<td>25.75</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>3.92</td>
<td>5.44</td>
</tr>
<tr>
<td>Fe₂O₃ (%)</td>
<td>2.95</td>
<td>2.44</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>61.06</td>
<td>51.56</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>3.08</td>
<td>4.51</td>
</tr>
<tr>
<td>Na₂O (%)</td>
<td>0.15</td>
<td>0.23</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>1.03</td>
<td>1.86</td>
</tr>
<tr>
<td>Insoluble residue - IR (%)</td>
<td>0.67</td>
<td>16.81</td>
</tr>
<tr>
<td>Loss on ignition - LI (%)</td>
<td>1.15</td>
<td>4.47</td>
</tr>
<tr>
<td>Free CaO</td>
<td>2.96</td>
<td>1.13</td>
</tr>
<tr>
<td>Blaine (cm²/g)</td>
<td>4020</td>
<td>4670</td>
</tr>
<tr>
<td>Specific density (g/cm³)</td>
<td>3.14</td>
<td>2.97</td>
</tr>
</tbody>
</table>

Two reinforcement bars with 6.3 mm diameter and 10 cm length were embedded in each specimen and were placed respecting 10 or 25 mm of concrete cover. Two reinforced concrete specimens were cast for each experimental condition. Reinforcements were previously cleaned by mechanically brushing their surfaces and had their extremities covered with an
insulating tape, delimiting an intermediate constant exposing area of 6 cm². Additional
reinforced specimens were cast to monitor carbonation and realkalisation of concrete cover as
described in section 2.2. All specimens were cured in a wet chamber for seven days and
remained protected with plastic film in laboratory environment until completing 180 days,
when it is expected that the concrete reach a more stable microstructure.

Table 2 – Concrete mixtures and properties.

<table>
<thead>
<tr>
<th>Concrete:</th>
<th>C1</th>
<th>C2</th>
<th>C3</th>
<th>C4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement type:</td>
<td>I</td>
<td>I</td>
<td>II</td>
<td>II</td>
</tr>
<tr>
<td>Cement (kg/m³)</td>
<td>362</td>
<td>349</td>
<td>360</td>
<td>347</td>
</tr>
<tr>
<td>Sand (kg/m³)</td>
<td>858</td>
<td>828</td>
<td>853</td>
<td>823</td>
</tr>
<tr>
<td>Aggregate (kg/m³)</td>
<td>934</td>
<td>902</td>
<td>928</td>
<td>896</td>
</tr>
<tr>
<td>Plasticiser (kg/m³)</td>
<td>1.77</td>
<td>-</td>
<td>3.51</td>
<td>-</td>
</tr>
<tr>
<td>w/c ratio</td>
<td>0.55</td>
<td>0.65</td>
<td>0.55</td>
<td>0.65</td>
</tr>
<tr>
<td>Slump (cm)</td>
<td>5.5</td>
<td>16.5</td>
<td>4.5</td>
<td>8.9</td>
</tr>
<tr>
<td>Compressive strength (MPa) - 90 days</td>
<td>40.2</td>
<td>36.3</td>
<td>28.9</td>
<td>22.3</td>
</tr>
<tr>
<td>Absorption (%)</td>
<td>3.14</td>
<td>4.62</td>
<td>4.52</td>
<td>5.67</td>
</tr>
<tr>
<td>Voids (%)</td>
<td>7.11</td>
<td>10.21</td>
<td>10.04</td>
<td>12.32</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>2.26</td>
<td>2.21</td>
<td>2.22</td>
<td>2.17</td>
</tr>
</tbody>
</table>

2.2 Accelerated carbonation and realkalisation procedures

After this initial period of 180 days, the specimens were subjected to accelerated
carbonation in a saturated carbonation chamber (100 % CO₂). During this carbonation period,
sacrificial non-reinforced concrete specimens were used to monitor the carbonation front
advance by means of a phenolphthalein test. Simultaneously to carbonation front advance
monitoring, electrochemical measurements were taken to identify reinforcement
depassivation, as commented in section 2.4.

For realkalisation treatment, an electrical field was generated between the reinforcement
(cathode) and an external anode attached to the concrete surface using a power supply, which
was continuously controlled to produce an average cathodic current density of 2 A/m² in
respect to the exposed concrete surface. In this arrangement, the anodes were made of
activated titanium mesh placed on the surface of the specimen and the electrolyte was a
sodium carbonate (NaCO₃) 1M solution.

2.3 Alkalinity monitoring

The system arrangement for the treatment was turned on until the concrete reinforcement
cover was completely realkalised. This was monitored in two duplicate specimens cast with
this objective, using a 1% thymolphthalein solution as a pH indicator, as shown in Figure 1.
The ratio between realkalised and previous carbonated area, expressed as a realkalised
fraction of the total carbonated area in the cover region, was used to monitor the realkalisation
advance in the concrete cover region.

Additional pH analyses were also carried out. When all the concrete cover was realkalised,
the specimens were broken and samples close to the reinforcement were collected using a
diamond-based tool. These samples represent an 8 mm thickness layer nearby the
reinforcement surface and were collected for reference (REF), carbonated (CARB) and
realkalised (REA) specimens. An extraction procedure similar to that described by Castellote
et al. [17] was used to obtain an equilibrium solution that was used to analyse the hydroxide concentration and, consequently, the pH of this equilibrium solution.

Figure 1 – Monitoring of specimens realkalisation: (a) cutting and (b) comparison of areas.

2.4. Electrochemical measurements

Electrochemical measurements were carried out during the carbonation and electrochemical realkalisation periods. This was done on single measurements of open circuit corrosion potential \( (E_{corr}) \) and instantaneous corrosion current density \( (i_{corr}) \) measurements.

Regarding electrochemical measurements to indicate reinforcement depassivation, this work accepted that \( i_{corr} \) values higher than 0.1 µA/cm² indicate metal depassivation, as well as \( E_{corr} \) values more electronegative than -350 mV (CSE) [18-19]. This electrochemical monitoring was continued during one year after the realkalisation treatment was over.

3 RESULTS AND DISCUSSION

3.1. Total electrical charge passed for completing realkalisation

The total charge capable to realkalise carbonated concrete cover was monitored by multiplying current density and time. Results are presented in Fig. 2, which shows the relationship between total charge passed (in respect to concrete surface) and realkalised fraction of the total carbonated area in the cover region, as described in section 2.3.

Results show that concretes made with Portland pozzolan cement (C3, C4) required more charge to complete concrete realkalisation. It could be expected that, as these concretes presented higher porosity (Table 2), less charge would be necessary. However, this behaviour can be explained by the fact that, as concretes made with pozzolan cement have less alkalinity reserve due to the portladite consumption in pozzolanic reactions, they tend to reach lower levels of alkalinity after carbonation and thus more total charge passed is demanded.

The influence of w/c can be observed comparing concretes C1 and C2. It can be seen that concrete C1 needs 330 A.h/m²_concrete to complete realkalisation, in proportion as concrete C2 needs 240 A.h/m²_concrete, which means an additional charge of 90 A.h/m²_concrete. In the case of concretes C3 and C4, there is a similar behaviour with a difference of about 50 A.h/m². As commented by Bertolini et al. [20], concretes with lower w/c need a higher total charge passed to complete the treatment and thus, under the same current density, the treatment takes longer to realkalise a given concrete cover thickness.
Figure 2 – Progress of the realkalised fraction as a function of the total charge passed.

Concretes with higher reinforcement cover thickness need more charge passed to complete realkalisation. Although this seems to be an obvious aspect, it was not measured in previous papers. It was necessary to change total charge passed from 240 to 400 A.h/m²(concrete) when increasing reinforcement concrete cover from 1 cm to 2.5 cm for concrete C2, which means an increase of more than 66% in total charge. For concrete C4, the total charge increased from 310 to 500 A.h/m²(concrete), which means a similar increase. This is related to the amount of OH⁻ ions that need to be generated to realkalise the whole concrete cover thickness.

3.2. Relative alkalinity changes due to the treatment

Figure 3 compares the alkalinity of concrete samples for REF, CARB and REA specimens. In the last case, samples were extracted 30 days after the treatment with the objective of representing a more stable condition, regarding the polarization applied during the electrochemical realkalisation and the chemical equilibrium nearby the reinforcement. Two samples were taken for each analysed condition and the average of each pair of samples was expressed as a relative alkalinity value in terms of the highest average pH value observed among all the analysed conditions, which was 13.4.

Figure 3 – Relative alkalinity nearby the reinforcement for REF, CARB and REA specimens.
For all studied concretes, specimens subjected to electrochemical realkalisation presented higher alkalinity than carbonated specimens and quite close to the alkalinity of reference specimens. This shows the efficiency of the treatment in recovering the concrete alkalinity \cite{8, 20}. On the other hand, it can also be noticed the influence of the cement type. Both concretes C3 and C4 presented lower alkalinity values for REF, CARB and REA specimens. This can be explained by its lower alkaline reserve (see Fig. 3, reference samples), which makes them reach lower levels of alkalinity during carbonation and after realkalisation.

There are also differences of alkalinity between specimens made with the same concrete mixture and different reinforcement concrete covers (C2/10, C2/25 and C4/10, C4/25). These differences can be related to the skin effect for REF and CARB specimens, which leads to differences on concrete microstructure in outer layers and also makes the carbonation of this concrete region easier. Furthermore, for REA specimens, the higher total charge demanded to realkalise a thicker reinforcement concrete cover generates more OH− ions at the reinforcement surrounding region (see section 3.2).

3.4. Electrochemical parameters and tendency of reaching passive condition

Figures 4 and 5 show the comparative behaviour of corrosion potential (Fig. 4) and corrosion current density measurements (Fig. 5) during the experiment.

By corrosion potential measurements, it is possible to see a tendency of reinforcement reaching the passive condition. This tendency is clearly seen twelve months after the end of the treatment. Before this period, there are still some values in the zone of uncertain corrosion.

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure4}
\caption{Comparative corrosion potential for REF, CARB and REA specimens.}
\end{figure}

Considering the corrosion current density, Figure 5 shows that, although only some bars presented values below 0.1 μA/cm², which indicate the passive condition, most of the bars reached the low corrosion current level after 12 months (6-month measurements were not taken for this variable). This implies in current values significantly lower, which means a metal dissolution rate significantly lower and, consequently, a longer residual service life. On the other hand, a tendency of reaching values even lower throughout time is not rejected.

Concerning this aspect, Redaelli and Bertolini \cite{16} found that although corrosion current density obtained by potentiostatic tests significantly decreased over time they do not match the condition of passive steel in alkaline media. On the other hand, no significant amounts of
corrosion products were found on steel surface after breaking these same specimens, which indicates that this kind of measurement could not be suitable for monitoring the effectiveness of steel repassivation. However, this aspect is not still a consensus [21]. The lack of an effective electro-osmotic flux has been reported as the reason for not reaching the effectiveness of reinforcement repassivation in some cases [21].

Figure 5 – Comparative corrosion current density for REF, CARB and REA specimens.

4 CONCLUSIONS

The influence of water to cement ratio on this treatment happens in an inverse way to the time required by the treatment and the total charge passed. It means that as w/c increases, the time to complete the treatment decreases, as well as the total electrical charge passed.

The use of pozzolanic addition in cement contributed for longer periods of realkalisation as a consequence of its lower alkalinity reserve due to portlandite consumption in pozzolanic reactions, as well as for higher total charge passed to complete the treatment. This lower alkalinity has consequences for reinforcement repassivation tendencies, demanding a longer time to reach this condition or tendency.

A thicker concrete cover demands more time and, consequently, more charge to produce enough alkalinity for the whole concrete cover region. On the other hand, this contributes for a shorter time demanded for reaching electrochemical stabilization, which can be explained by the higher levels of alkalinity reached nearby the reinforcement.

The tendency of reaching the passive condition is clearer for corrosion potential measurements. For corrosion current density measurements, although it is not so clear, they tend to decrease over time, reaching values not far from this reference and longer service life periods for the structure. However, more monitoring time and further research are necessary to analyse if it is possible to confirm a similar tendency with this parameter and if corrosion current rate could be suitable for this purpose.

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ENVIRONMENTAL IMPACT ASSESSMENT OF FIRE RETARDANT TREATED WOOD USED IN EXTERIOR CLADDINGS

Selamawit Mamo Fufa (1,2) and Rolf André Bohne (1)

(1) Department of Civil and Transport Engineering, Norwegian University of Science and Technology, Trondheim – selamawit.fufa@ntnu.no; rolf.bohne@ntnu.no;

(2) Department of Materials and Structures, SINTEF Building and Infrastructure, Trondheim – selamawit.fufa@sintef.no

Abstract

Fire retardant treated wood has been used in order to overcome the increasing demands of fire safety requirements and to extend the use of wood to most applications. The treated wood also has a positive environmental impact with respect to inhibiting fires, thus limiting the emissions produced during the burning of wood. In addition to effectiveness in the case of fire, for sustainable utilization of fire retardant treated wood, consideration of the environmental performance of the fire retardants throughout their life cycle is essential. Although the fire- LCA model has been developed and used to evaluate fire retardant products, the model has focused on the environmental impact of accidental fires based on fire statistics and experimental data. The aim of this work is to conduct a LCA of fire retardant treated wood in accordance with ISO 14040 series methodologies in order to investigate the performance of materials and/or processes, as well as the product life cycle stage, which contributes to the greatest impact on the environment. The LCA study considered the raw material acquisition, fire retardant treated wood production and transportation life cycle stages of 1 m3 of fire retardant treated wood. The environmental impact analysis results show the highest impact from the raw material production stage.

1 INTRODUCTION

Fires that occur in buildings have been a concern due to the increased risk of injuries, loss of life and property. Toxic combustion products, flame and high temperature, a reduction in oxygen concentration, reduced visibility and the instability of construction are some of the factors which have endangered both residents and the people performing rescues.

The type of material selected in buildings plays a major role in determining the risk of fire and its propagation through the structure or adjacent units. Wood is one of the well-used sustainable building materials, and in Norway wood has a strong tradition as a building
material in domestic buildings. Fire retardant treated wood (FRTW) products have been used in reducing different reaction-to-fire properties of the wood through increasing the time to ignition, reducing the flame spread across the surface, decreasing the heat release rate during combustion and preventing the formation of flammable drops [1, 2]. Hence, fire retardants reduce the temperature at which the thermal degradation of wood occurs, thereby allowing people more time to evacuate the building and for the authorities to control the spreading of the fire. The increased opportunity of using FRTW leads to the improvement of both the FR and treatment processes with respect to fire. The quality of FRTW products has also increased the potential use of wood as a building material, particularly in multi-story buildings.

Although FRs have been used in order to reduce the risk of fires, some of these treatments have been criticized due to their poor performance [3-5]. The increase in smoke and carbon monoxide release caused by an incomplete combustion of halogenated FRs, the high loading level requirements of FRs such as magnesium hydroxide and hygroscopicity and the acidic properties of inorganic salt based FRs are some of the drawbacks of FRs. In addition, even if fire retardant treated wood products have all had positive impacts in inhibiting fires, the FR chemicals may cause environmental problems. For the sustainable utilization of fire retardant treated wood, assessing the life cycle environmental impacts of the treated wood is crucial. Life cycle assessment (LCA) helps to identify the potential environmental effect of fire retardant treated wood during its overall life cycle in order to identify the system that contributes to critical environmental impact and to avoiding the possibility of problem shifting.

Some fire retardant producers state that their products are environmentally friendly, though to the best of our knowledge there are no open references that cover the LCA of fire retardant treated wood products using standard methodologies. This study investigates the environmental impact associated with fire retardant treated spruce wood used in exterior cladding based on life cycle perspective, using ISO 14040 [6] and ISO 14044 [7] LCA methodologies. The LCA consists of goal and functional unit definition, a description of the system boundaries, the development of inventory of flows in and out of the system boundaries, impact assessment and interpretation stages.

2 GOAL AND SCOPE OF THE STUDY

2.1 Goal of the study

The LCA study was performed to evaluate the environmental performance of fire retardant treated wood (FRTW) used in exterior claddings.

2.2 Functional unit

The functional unit (FU) is a reference unit, where inventory inputs and outputs are quantified in the system boundary to help facilitate the conversion of the basic life cycle inventory data to the real situation, in which the FRTW is used in its intended application. In this study, the LCA was done using a FU of 1 m³ FRTW.

2.3 System boundaries

The system limits cradle to gate analysis taking raw material acquisition, transportation and manufacturing of FRTW life cycle stages into consideration. The use and end of life
stages were not included in the present study. A schematic representation of the system boundaries of the study is illustrated in Figure 1.

Spruce wood species, obtained from sustainably managed local forests, were impregnated with FRs and other additives, under a vacuum of 40 mbar for 20 minutes, followed by an air pressure of 17 bar for approximately 1 hour. The FRTW was coated with one layer of primer and two layers of top coat after it had completely dried in a drying chamber for approximately a week. The product was then packed and stored in a warehouse. According to the European reaction to fire classifications [8, 9], the final FRTW manufactured is classified as B-s1,d0, in which "B" describes fire behavior, "s1" smoke production and d0 flaming particles or droplets.

2.4 LCA tool and database
SimaPro (version 7.3.3) [10] was used to model the LCA, while the Ecoinvent database (version 2.2) [11] in SimaPro was used to estimate the contribution of unavailable data and background processes.

3 INVENTORY ANALYSIS
The life cycle inventory (LCI) phase of LCA focuses on developing life cycle data for inputs and outputs throughout the lifecycle stage of the product.
3.1 Raw materials

Wood, fire retardants and additives are the main raw materials used for the manufacturing of FRTW. The foreground information of the selected process in wood production is based on the LCA report obtained from SINTEF Building and Infrastructure [12], which was developed in the process of making an environmental product declaration (EPD-Norge) for different wood and wood based products. The potential environmental burden caused by wood used as raw material was assessed based on Norwegian sawn dried wood EPD data [13]. According to the Norwegian sawn dried wood EPD, 95% of round timbers used in the production of Norwegian planed structural timber are certified in accordance with the PEFC standard (Programme for the Endorsement of Forest Certification).

Primary input and output data for FR chemicals and other additives were collected from manufacturers in Norway. FRTW is delivered with plastic packaging that is included in the LCA-inventory.

The coatings data was based on environmental data sheets and the technical product data of Akzo Nobel products, Cetol BL 21 Plus and Cetol BL Opaque. Cetol BL 21 Plus was used as a primer prior to being overcoated with Cetol BL Opaque. Based on the producer’s information, Cetol BL 21 Plus covers a minimum of 4 m²/litre and the equivalent amount of primer used was 0.26 kg/m². For the case of the Cetol BL Opaque top coat, it covers a minimum of 6 m²/litre, which is equivalent to 0.41 kg/m² for a total of the two layers.

3.2 Energy

The production of FRTW uses energy in the form of electricity and thermal energy. Electricity consumption was from domestic production in Norway using a NORDEL electricity mix. In Norway, even if most of the electricity is hydropower-based, due to seasonal disturbances or higher production demand, Norway requires extra electricity import, which comes primarily from Nordic countries. This is the reason why the electricity mix from NORDEL is used instead of the Norwegian electricity mix. Production waste and biomass are also used in the company's power plant for the production of thermal energy.

3.3 Transport

The transportation of raw material to the production area and internal transport were included in the LCA calculations, and the raw material transportation was carried out with road transportation (Transport, lorry > 32t, EURO3/RER U). The transport processes were based on the Ecoinvent database, through the average kgkm (kg transported each km).

4 IMPACT ASSESSMENT METHODS

Life cycle impact assessment has been performed by considering both the European ReCiPe midpoint method [14] and the single issue method, which is provided by the Ecoinvent (version 2.0) and expanded by PRe Consultants for the energy resources available in the database [15].

The impact categories analyzed in this study are Global warming potential (GWP, kg CO₂ eq.); Ozone depletion potential (ODP, kg CFC-11 eq.); Acidification potential (AP, kg SO₂ eq.) and Eutrophication potential (EP, kg P eq.).

The cumulative energy demand (CED, MJ) includes characterization factors for both non-renewable and renewable energy sources divided in 6 impact categories: fossil; non-
renewable nuclear; non-renewable biomass; renewable biomass; renewable wind, solar, geothermal; and renewable hydropower.

5 RESULTS AND DISCUSSIONS

Figure 2 shows the relative percentage contribution of the fire retardant treated wooden cladding manufacturing process to GWP, ODP, AP and EP impact categories.

It is seen that the raw material life cycle stage presented the highest contribution to all impact categories followed by transport and manufacturing. The FR has the largest impact on EP. This is due to a higher FR consumption, and to the higher FR retention levels required to reach fire classifications compared to ordinary preservation treatments used to protect wood against biological decay [5, 16].

The second largest impact contribution is from transport, especially in the ODP and GWP impact categories, as most of input materials are imported from abroad. Wood and additives are contributed indirectly through the input transport. Relatively speaking, the FRTW manufacturing process is the lowest impact contributor. This can be due to a lower impact from the source of energy used in the impregnation process, which is energy intensive.

Table 1 summarizes primary energy consumption (non-renewable and renewable) for raw material, transport and manufacturing life stages.
Table 1. Primary energy consumption for the manufacturing of 1 m³ of fire retardant treated wood

<table>
<thead>
<tr>
<th>Impact category</th>
<th>Raw Material</th>
<th>Transport</th>
<th>Manufacturing</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-renewable primary energy (MJ)</td>
<td>8.55E-02</td>
<td>3.95E-02</td>
<td>1.02E-02</td>
<td>9.57E-02</td>
</tr>
<tr>
<td>Renewable primary energy (MJ)</td>
<td>3.76E-02</td>
<td>8.35E-04</td>
<td>9.28E-03</td>
<td>4.77E-02</td>
</tr>
</tbody>
</table>

For the manufacture of 1m³ FRTW, raw materials production accounts for approximately 89-79%, while manufacturing accounts for approximately 10-19% of consumption of both non-renewable and renewable energy, respectively.

A closer inspection of the composition of the non-renewable and renewable primary energy consumption depicted in Figure 3 indicates that the essential primary energy carrier is fossil accounting for approximately 57% of the primary energy used, followed by hydropower (approximately 18%).

![Figure 3. Energy consumption specified for the different energy carriers and life cycle stages.](image)

6 CONCLUSIONS

In this study, the findings illustrate the highest impact from the raw materials for most of the impact categories, which is due to a high consumption of the fire retardants. By contrast, fire retardant impregnation and coating activities showed a relatively low contribution in most impact categories. Selecting durable and environmentally preferable fire retardants may help reduce the impact of fire retardant treated wood. The results obtained from this study provide valuable information for fire retardant treated wooden claddings manufacturers to help improve their environmental performance. Our ongoing research work continues to assess the use phase and end of life of the cladding to help complete the LCA study. Further work will focus on identifying the environmental burdens along with uncertainty analysis and scenario analysis.
ACKNOWLEDGMENTS

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PERFORMANCE ANALYSIS OF WOOD SUBJECTED TO TREATMENTS MODIFIED WITH TITANIUM DIOXIDE AND CLAY NANOPARTICLES

Selamawit M. Fufa (1,2) and Bjørn Petter Jelle (1,2)

(1) Department of Materials and Structures, SINTEF Building and Infrastructure, Trondheim – selamawit.fufa@sintef.no; bjorn.petter.jelle@sintef.no
(2) Department of Civil and Transport Engineering, Norwegian University of Science and Technology, Trondheim – bjorn.petter.jelle@sintef.no; selamawit.fufa@ntnu.no

Abstract
In this work, the overall performance with respect to the durability, reaction to fire properties and environmental impacts of wood subjected to treatments modified with 1 wt% of TiO₂ and/or clay nanoparticles were summarized. The weathering performance of the specimens were analysed by performing accelerated ageing tests and FTIR analyses. The cone calorimeter was used to assess the reaction to fire performances, whereas the environmental impact of the treatments were investigated using life cycle assessment. Herein, all the test methods and results obtained are summarized to investigate, compare and evaluate the overall effect of nanoparticles on the selected treated wood properties. Thus, this work gives an overview of the importance of performing different test methods in order to evaluate the effect of treatments modified with nanoparticles on different wood properties along with their potential environmental impact.

1 INTRODUCTION

Buildings play a major role in the consumption of the total available energy, hence contribute a lot in natural resource consumption and emission of various hazardous gases which can affect human health and the environment [1]. Choosing the appropriate building materials may help to meet the rapidly growing need for buildings in an environmentally responsible way.

Wood has been playing an important role in sustainable design mainly because it is a renewable material, relatively cheap and has lower environmental footprint. However, Wood exposed to exterior applications on buildings can be affected by combined actions of environmental factors if left untreated [2]. In addition, wood is a combustible building material, which may contribute to fire development to an unacceptable extent. Wood treatments, which help to extend the service life of the wood, have been modified through time based on the required durability and environmental performances. Nanotechnology based
treatments are one of treatments used to improve multiple wood properties or introducing new ones with a very small percentage [3]. The vast benefits and growing popularity of these treatments focusing only on their novel properties may have adverse negative impact on human health and the environment.

This work summarizes an output from research work which focused on investigating the durability and reaction to fire properties of wood by modifying wood treatments with 1 wt% of TiO$_2$ nanoparticles and/or nanoclay [4-6]. The objective was to combine TiO$_2$ nanoparticles and nanoclay in existing wood treatments to benefit from UV stabilization and fire retardant properties of the nanoparticles, respectively. Herein all the test methods and results of coated and impregnated specimens published in different articles are summarized to investigate, compare and evaluate the overall effect of the nanoparticles on the selected coated wood properties. Thus, this work gives an overview of the importance of performing different test methods in order to evaluate the effect of treatments modified with nanoparticles on different wood properties along with their potential environmental impact.

2 METHODOLOGY

2.1 Test specimens

Norway spruce (Picea abies) wood specimens free of knots and visible defects were used. Before finishing, test specimens with size of (100.0 ± 0.5) mm x (100.0 ± 0.5) mm x (20.0 ± 0.5) mm were made for accelerated ageing and cone calorimeter test. Spruce (S) coated with water-based paint (WBP) and impregnated with preservative (I) was tested in addition to untreated spruce specimens (SU). The treatments were mixed with 1 wt% each of TiO$_2$ nanoparticles (NTs) and nanoclay (NC) or a combination of the two. The codes used throughout the paper are explained in Table 1.

Table 1: Description of the coding system [6].

<table>
<thead>
<tr>
<th>Code</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SU</td>
<td>Spruce untreated</td>
</tr>
<tr>
<td>SWBP</td>
<td>Spruce coated with water-based paint</td>
</tr>
<tr>
<td>SWBPNT</td>
<td>Spruce coated with water-based paint modified with 1 wt% NT</td>
</tr>
<tr>
<td>SWBPNC</td>
<td>Spruce coated with water-based paint modified with 1 wt% NC</td>
</tr>
<tr>
<td>SWBPNCT</td>
<td>Spruce coated with water-based paint modified with 0.5 wt% NT and 0.5 wt% NC</td>
</tr>
<tr>
<td>SI</td>
<td>Spruce impregnated with wolmanit CX-8</td>
</tr>
<tr>
<td>SINT</td>
<td>Spruce impregnated with wolmanit CX-8 modified with 1 wt% NT</td>
</tr>
<tr>
<td>SINC</td>
<td>Spruce impregnated with wolmanit CX-8 modified with 1 wt% NC</td>
</tr>
<tr>
<td>SINCT</td>
<td>Spruce impregnated with wolmanit CX-8 modified with 0.5 wt% NT and 0.5 wt% NC</td>
</tr>
</tbody>
</table>
Water-based acrylic blue tinted paint and primer supplied by Jotun AS was the paint used. Wolmanit CX-8 provided by Moelven Wood Norway was the preservative selected for impregnation. The NT used in this study was Hombitec RM 402 obtained from Sachtleben Chemie, while the NC was hydrophilic bentonite purchased from Sigma Aldrich, Norway.

2.2 Durability test and evaluation methods

The weathering performance of the treated specimens was investigated by exposing specimens to accelerated ageing test. An Atlas SC600 MHG Solar Simulator ageing test chamber, which simulates solar radiation and rain using a metal halide global (MHG) lamp and water spray, respectively, was used. The test specimens were exposed to a 2500 W MHG lamp, with a solar radiation intensity of 1200 W/m² and a total of 1 l/min water spray from the two nozzles. The ageing test was performed with a 6 h cycle consisting of 5 h of solar radiation at 63 °C and 50 %RH and 1 h water spray at 10 °C and 100 %RH for a total of 62 days.

The chemical changes occurring on the surface of the specimens, before, during and after the ageing process were analysed using Fourier transform infrared (FTIR) spectroscopy. The FTIR spectra were obtained by a Thermo Nicolet 8700 spectrometer equipped with an attenuated total reflectance (ATR) Smart Orbit accessory applying a diamond crystal. Infrared spectra were collected in the wave number range from 4000 cm⁻¹ to 400 cm⁻¹ at a spectral resolution of 4 cm⁻¹ with 32 scans.

The rate of degradation in the Atlas ageing test chamber was compared with actual outdoor exposure using an acceleration factor. An acceleration factor of 260, which correlates 1 day exposure in the Atlas ageing apparatus to 260 days of actual outdoor exposure, was estimated based on simplified calculation performed by Fufa et.al.[4].

2.3 Cone calorimeter

The reaction to fire behaviour of the specimens was investigated in cone calorimeter test carried out at SINTEF NBL. Three parallel specimens were tested under 50 kW/m² heat flux exposure. The test results of heat release rate (HRR) of specimens are reported.

2.4 LCA methodology

Life cycle assessment presents the method of applying FTIR durability analysis for use in LCA evaluations. The study was performed by combining environmental data, environmental product declaration (EPD) and ecolabel inventory data, with durability test results analysed by FTIR analysis. Due to the limitation of the available data, only environmental impact of spruce coated with acrylic water based paint modified with NT (SWBPNT) in comparison with wood coated with acrylic water based paint (SWBP) was performed. The system included production, construction, use and maintenance and demolition life cycle stages. SimaPro’s LCA version 7.3 software for the ReCiPe Midpoint and Endpoint impact assessment methods from the Hierarchist (H) perspective [7] was used to analyse global warming, human toxicity, photochemical oxidation, acidification and eutrophication impact categories.
3 RESULTS AND DISCUSSION

3.1 Weathering performance analysis

The FTIR spectra of impregnated and coated specimens before, during and after ageing in an Atlas solar simulator are plotted on a logarithmic absorbance scale in the wave number range between 1800 cm$^{-1}$ to 800 cm$^{-1}$ as shown in Figure 1 and Figure 2 respectively.

![Absorbance spectra](image)

Figure 1: Absorbance spectra for impregnated specimens, SI (a), SINC (b), SINT (c) and SINCT (d) [8].

Different spectral changes at wave numbers around 1722 cm$^{-1}$, 1141 cm$^{-1}$ and 1050 cm$^{-1}$ are assigned to the bending or stretching vibrations of different groups [3]. Herein we summarize the changes within the absorption region around 1722 cm$^{-1}$ and 1050 cm$^{-1}$ wave numbers.

The absorbance peaks around 1722 cm$^{-1}$ attributed to C=O stretching and decrease for all impregnated and coated specimens during exposure. However, the absorbance band of spruce impregnated with unmodified preservatives (SI) and modified preservatives (SINC, SINT and SINCT) specimens are much weaker than that of the similar band of the coated specimens. This may show that this band is mainly attributed to the C=O bonds of the coating. For coated specimen, the decrease in absorbance with increase in ageing time may be due to the removal of the coating from the surface of the wood. The change for SWBPNT was relatively the lowest one, whereas the largest change was observed for SWBPNC.
The peaks of the absorption bands around 1050 cm$^{-1}$, which are significant for impregnated specimens, are assigned to hydroxyl (OH) stretching vibration. The changes in these absorption bands might show growing concentration of hydroxyl groups in polysaccharides due to the formation of free OH groups in addition to absorption of water during the accelerated ageing test ([9, 10]. Smallest change in these bands was observed for SINT.

### 3.2 Reaction to fire performance

HRR is one of the most important parameters for assessment of fire hazard and is used for characterization of reaction to fire behaviour. A typical wood material will have two HRR peaks when burning, as shown in Figure 3, which is related to its combustion characteristics.
The HRR of impregnated and coated specimens was lower than untreated specimens. The higher HRR values of the treated specimens might be because of the release of a vapour from the treatments (either from the coating or the preservative) during ignition. From the two types of treatments, HRR of coated specimens were higher than the impregnated ones. Coatings are made of different chemicals that can be easily volatilized under high temperature and speed up ignition. As they are also applied on the surface, upon heating, coatings may be volatilized easily and facilitate further combustion of the wood. In general, the results obtained from these experiments showed poor reaction to fire performance of nanoparticles containing treatments.

### 3.3 Environmental impact assessment

LCA was performed based on maintenance interval determined from the accelerated aging test [11]. The FTIR analysis at around 1722 cm\(^{-1}\) absorbance band was selected for estimating the time when significant change occurs in the coated wood specimens. With an estimated service life of 50 years, the maintenance intervals of 14 and 23 years were obtained for SWBP and SWBPNT, respectively. The results from LCA are presented in Figure 4.
For SWBP and SWBPNT, climate change and human toxicity impact categories are relatively the highest ones. This is may be due to leaching of the paint or the waste generated from the paint utilized during the use and maintenance stages. The overall environmental impact comparison between SWBP and SWBPNT show a relatively lower total environmental impact of SWBPNT compared to SWBP. However, it is also important to note that if the interest is not in reducing the maintenance interval and the cladding is recoated more frequently, i.e. more often than the estimated maintenance interval in this work, the impact of SWBPNT may be higher than SWBP.

4 CONCLUSIONS

Accelerated ageing test results indicate the potential of specimens treated with small percentage of TiO$_2$ nanoparticles and/or nanoclay in delaying the effect of degradation. Even if all types of treatments protect the specimens in one way or other, the results show in general relatively poor effects of impregnated specimens compared to coated specimens. However, the reaction to fire properties of specimens treated with modified treatments was poor. The results from comparison of the overall environmental load reveal that the impact of SWBPNT was slightly lower than SWBP. In addition, this LCA study gives a general overview on the methodology of combining available environmental data with durability test results analysed by Fourier transform infrared (FTIR) spectroscopy when investigating the environmental impact of wood coated with nano-based paint. Further investigations are required by including fire retardants with the nanoparticles. Moreover, examining the retention and distribution of nanoparticles using transmission electron microscopy image analysis and dispersion of the nanoparticles using different dispersion techniques could further be investigated.
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PERFORMANCE OVER TIME OF ADHESIVE SYSTEMS FOR BONDING CERAMIC TILES ON FAÇADES – A CASE STUDY

V.P. de Freitas (1), H. Corvacho(1), M. Quintela(1) and J.M.P.Q. Delgado(1)

(1) LFC – Laboratório de Física das Construções, Departamento de Engenharia Civil, Faculdade de Engenharia da Universidade do Porto, Porto, Portugal – vpfreita@fe.up.pt; corvacho@fe.up.pt; betaniaquintela@gmail.com; jdelgado@fe.up.pt

Abstract

An efficient adhesive bonding of exterior ceramic tiles applied on façades is an obvious important factor to ensure the safety and the durability of the façade. The failure of adhesive bonding is a common issue with relevant technical and economical consequences.

The aim of this work is to present an evaluation of the performance overtime of adhesives systems in bonded ceramic tiles on façades, based on extensive experimental research works carried out at the Laboratory of Building Physics (LFC).

A case study is presented which evaluate the performance of adhesives systems to be used on the façades of a building located near the sea. For this purpose, accelerated ageing tests are performed following two different ageing procedures, allowing the comparison of the performance over time of the systems under analysis.

1 INTRODUCTION

In this work a complete case study is analysed. The main goal of this case study was to evaluate the performance of alternative adhesive systems based on the variation over time of their adhesion to the support. The evaluation of the adhesive system performance was carried out by measuring the adhesion to the support in the initial situation and after submitting the materials to different cycles of accelerated ageing. The adhesive bonding of exterior ceramic tiles applied on façades is an important factor to ensure the safety and durability of the building. The failure of adhesive bonding has immediate consequences and therefore is a common concern for the building industry and building owners.

This case study focuses on the behaviour of ceramic tiles applied on exterior surface where substrate is concrete C35/40. This type of envelope frequently develops pathologies that can have different causes. The Pull-off test is a very popular way of evaluating the adhesion strength of renderings or ceramic tiles and it is frequently used as a tool to help on the correct diagnosis of causes for degradation of the façades exterior layers.

Several standards are available to frame the evaluation of adhesive strength by means of the pull-off test, namely EN 1348 (2007) and EN 12004 (2008) which were used in this study.
2 EXPERIMENTAL SET-UP

This extensive case study presents the results of a work carried out by request of the Port Authority of “Porto de Leixões” to analyse several solutions of bonding systems proposed by the architecture to the external façade of Cruise Terminal (see Figure 1).

Table 1: Brief description of the samples tested.

<table>
<thead>
<tr>
<th>Bonding System</th>
<th>Description</th>
<th>Layers</th>
</tr>
</thead>
<tbody>
<tr>
<td>BA1</td>
<td>A waterproofing product (W1) and a reaction resin adhesive (RA1)*</td>
<td>2</td>
</tr>
<tr>
<td>BA2</td>
<td>A waterproofing product (W2) and a reaction resin adhesive (RA1)*</td>
<td>2</td>
</tr>
<tr>
<td>K</td>
<td>A waterproofing product (W3) and a reaction resin adhesive (RA2)*</td>
<td>2</td>
</tr>
<tr>
<td>M</td>
<td>Waterproofing and adhesion provided by a reaction resin adhesive (RA3)*</td>
<td>1</td>
</tr>
<tr>
<td>T</td>
<td>Waterproofing and adhesion provided by a reaction resin adhesive (RA4)*</td>
<td>1</td>
</tr>
</tbody>
</table>

The specimens used in this case study were made with concrete C35/40 (support), with the application of a waterproofing system and adhesive. For each of the 6 adhesive systems analysed (see Table 1) we used 10 specimens (RILEM 2004). On all the tests described in this work, the ceramic elements test area is 50 mm long, 50 mm wide and 10 mm thick.

2.1 Accelerated Ageing Process

The first accelerated ageing procedure, designed as cycle A, consists of the variation of conditions of temperature and relative humidity, in a climatic chamber, associated with the effects of rain (spraying with water) and of solar radiation (Xenon arc lamp), having a duration base of 12 hours. Figure 1 shows the climatic conditions of this cycle. For this ageing procedure, the performance of the six bonding systems was evaluated at four different moments in time: after 28 cycles, 56 cycles, 84 cycles and 150 cycles of exposure in the climatic chamber subjected to repeated cycles of 12 hours.

The second ageing procedure, designed as cycle B, and based on EN 1015-21 (2002), is described in Table 2. It consists of the repetition of a heating/freezing cycle (Cycle B1) and a humidification/freezing cycle (Cycle B2). Each of the cycles, B1 and B2, has duration of 24 hours. In the transition from cycle B1 to cycle B2, the specimens remain for 48 hours in a controlled environment (at the laboratory). For this second ageing procedure, the performance
of the six bonding systems was evaluated at three different moments in time: T1, after the exposure to (2 x cycle B1 + standard environment + 2 x cycle B2); T2, after the exposure to (4 x cycle B1 + standard environment + 4 x cycle B2) and T3, after the exposure to (8 x cycle B1 + standard environment + 8 x cycle B2).

Table 2: Cycles combination B.

<table>
<thead>
<tr>
<th>Cycle B1</th>
<th>Cycle B2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heating/Freezing</td>
<td>Standard environment (laboratory)</td>
</tr>
<tr>
<td>T (°C)</td>
<td>RH(%)</td>
</tr>
<tr>
<td>60</td>
<td>n.c.</td>
</tr>
<tr>
<td>20</td>
<td>65</td>
</tr>
<tr>
<td>-20</td>
<td>n.c.</td>
</tr>
</tbody>
</table>

2.2 Pull-Off Test

The adhesive strength is determined as the maximum tensile strength applied by a direct load perpendicular to the surface being tested (Ramos et al., 2012). The pull-off test is classified as a near-to-surface, partially destructive method which is able to measure tensile strength of various materials. The tensile load is applied by means of a defined pull-head plate glued to the test area. The adhesive strength is the ratio between the failure load and the test area.

Circular metallic plates, with diameter of 50 mm and 18 mm thick, were used which means that results weren’t affected by plate geometry variation (see Figure 2).

![Figure 2: Failures observed after 150 cycles of accelerated ageing procedure-cycle A.](image)

3 RESULTS AND DISCUSSION

The experimental results are presented for each bonding system tested. The first series of results is the reference series for the initial conditions which means that no accelerated ageing procedure was applied. The second and third series present the results of the accelerated ageing procedures, cycle A and cycle B.

The following tables and figures present the results related to bonding systems analysed (BA1, BA2, K, M and T) for the first series without accelerated ageing (Table 3) and the results of the accelerated ageing procedures, cycle A and cycle B (Figures 3 and 4).
Table 3: Pull-off test results without accelerated ageing, reference value (t0).

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Sample</th>
<th>Adh. strength [N/mm²]</th>
<th>Type of failure</th>
<th>ΔAS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>BA1</td>
<td>I</td>
<td>1.20</td>
<td>CF-sA(I)</td>
<td>+9</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>1.10</td>
<td>CF-sA(I)</td>
<td>-0</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>1.10</td>
<td>CF-sA(I)</td>
<td></td>
</tr>
<tr>
<td>BA2</td>
<td>I</td>
<td>2.00</td>
<td>AF-sA(I)</td>
<td>+5</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>2.20</td>
<td>AF-sA(I)</td>
<td>-24</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>1.45</td>
<td>AF-T</td>
<td></td>
</tr>
<tr>
<td>K</td>
<td>I</td>
<td>1.35</td>
<td>AF-sA(I)</td>
<td>+4</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>1.15</td>
<td>AF-sA(I)</td>
<td>-12</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>1.35</td>
<td>AF-sA(I)</td>
<td></td>
</tr>
<tr>
<td>M</td>
<td>I</td>
<td>0.85</td>
<td>CF-T*</td>
<td>+10</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>2.20</td>
<td>CF-A</td>
<td>-5</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>1.90</td>
<td>CF-A</td>
<td></td>
</tr>
<tr>
<td>T</td>
<td>I</td>
<td>2.90</td>
<td>CF-S</td>
<td>+7</td>
</tr>
<tr>
<td></td>
<td>II</td>
<td>3.00</td>
<td>CF-S</td>
<td>-7</td>
</tr>
<tr>
<td></td>
<td>III</td>
<td>2.60</td>
<td>CF-S</td>
<td></td>
</tr>
</tbody>
</table>

ΔAS: Positive/negative maximum differences from the average value of adhesive strength;
AF-sA(I): Adhesive failure between the first and second waterproofing layers
AF-T: Adhesive failure between the ceramic tile and the adhesive
CF-A: Cohesive failure by the adhesive
CF-S: Cohesive failure by the concrete support
CF-sA(I): Cohesive failure by the waterproofing layer
CF-T: Cohesive failure by the ceramic tile.
Figure 3: Pull-off test results for the two ageing procedures and for bonding systems: (a)-BA1, (b)-BA2 and (c)-K.
Figure 4: Pull-off test results for the two ageing procedures and for bonding systems: (a)-M and (b)-T.

As given in Table 1, the bonding system BA1 is constituted by a waterproofing product (W1) and a reaction resin adhesive (RA1) of class R2T according to NP EN 12004. The bonding system BA2 is constituted by a waterproofing product (W2) different from the one of system BA1 and the same reaction resin adhesive (RA1). The bonding system K is constituted by a waterproofing product (W3) and a reaction resin adhesive (RA2) of class R2T according to NP EN 12004, both different from the previous tested systems (BA1 and BA2). The bonding system M is constituted by only one product, a reaction resin adhesive (RA3) which provides both waterproofing and adhesion. Finally, the bonding system T is composed by only one product, a reaction resin adhesive (RA4), which provides both waterproofing and adhesion.

Table 4: Summary of the pull-off test results.

<table>
<thead>
<tr>
<th>Bonding system</th>
<th>Initial ref. value (t0)</th>
<th>Minimum (Test time)</th>
<th>Accelerated Ageing Procedure</th>
<th>Accelerated Ageing Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cycle A</td>
<td>Cycle B</td>
</tr>
<tr>
<td>BA1</td>
<td>1.1</td>
<td>0.90 (several)</td>
<td>No significant variation</td>
<td>No significant variation</td>
</tr>
<tr>
<td>BA2</td>
<td>1.9</td>
<td>0.30 (A₂₇₅)</td>
<td>Decreasing trend</td>
<td>No significant variation</td>
</tr>
<tr>
<td>K</td>
<td>1.3</td>
<td>0.40 (A₂₇₅)</td>
<td>Decreasing trend</td>
<td>No significant variation</td>
</tr>
<tr>
<td>M</td>
<td>2.0</td>
<td>0.90 (A₂₈)</td>
<td>Negligible decreasing trend</td>
<td>Decreasing trend</td>
</tr>
<tr>
<td>T</td>
<td>2.8</td>
<td>1.20 (A₂₈)</td>
<td>Decreases in the 1st accelerated ageing time &amp; maintains thereafter</td>
<td>Negligible decreasing trend</td>
</tr>
</tbody>
</table>

Figure 5 and Table 4 show a summary of the experimental results described in detail above. The comparative analysis took, from the research, the accelerated ageing procedures already tested and confirmed their relevance. Previous experimental studies developed by Freitas et al. (2008) with the same accelerated ageing procedure, the cycle A, developed by LFC and presented in Figure 1, for cementitious adhesive systems are summarized in Figure 5, by the thick grey line. The kind of experiments carried out, although inconclusive in what
concerns a durability assessment in absolute terms, allows a relevant comparison between several solutions, in this case between the adhesive strength over time of different bonding systems.

In the case of the use of reaction resin adhesives the results obtained are less expressive, with no significant variation of the adhesive strength in some of the tests (see Table 4).

![Figure 5: Summary of Pull-off test results for the first ageing procedure (Cycle A) and previous research results for cementitious adhesive.](image)

In Figure 5 it is possible to observe that bonding systems analysed (reaction resin adhesives) didn’t present the marked degradation curve observed with cementitious adhesive systems for the same accelerated ageing procedure (Cycle A). The adhesive systems analysed present a decreases in the first accelerated ageing time but maintains thereafter.

Another important conclusion resulted by the analyse of Figure 7 is that the reaction resin adhesives with 1 layer (M and T) presents better results that the bonding systems with 2 layer (Ba1, BA2 and K).

The degradation effects obtained with the accelerated ageing procedure named Cycle B were less expressive and, for some of the bonding systems tested, there was no significant variation of their adhesive strength.

**4 CONCLUSIONS**

In this work, a test campaign with the aim of characterizing the performance over time of different bonding systems for ceramic tiles to be applied on the façades of a large building, the Cruise Terminal, was presented as a case study.

The main conclusions of this work are:
- It is not possible to define objectively the durability of the tested solution;
- It is possible to present a comparison between the different tested solutions and show, for previous research that cementitious adhesives systems has a higher degradation tendency than reaction resin adhesives;
The cementitious adhesive presents a marked degradation curve, due to the accelerated ageing procedure named Cycle A, however, this clear trend was not observed in the case of reaction resin adhesives; The reaction resin adhesives with 1 layer (M and T) presents better results that the bonding systems with 2 layer (Ba1, BA2 and K); The accelerated ageing procedure designed as cycle A and developed by LFC presented more conclusive results.

ACKNOWLEDGEMENTS

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REFERENCES


HYGROTHERMAL PERFORMANCE OF A NATURALLY VENTILATED GYPSUM HOUSE – THE BRAZILIAN CLIMATE INFLUENCE

R.M.S.F. Almeida(1)(2), V.P. de Freitas(2), J.M.P.Q. Delgado(2) and P. Paula(2)

(1) Escola Superior de Tecnologia e Gestão de Viseu, Campus Politécnico de Repeses, Viseu, Portugal – ralmeida@estv.ipv.pt
(2) LFC – Laboratório de Física das Construções, Departamento de Engenharia Civil, Faculdade de Engenharia da Universidade do Porto, Porto, Portugal – vpfreita@fe.up.pt; jdelgado@fe.up.pt; dec11021@fe.up.pt

Abstract

A simple modular constructive system based on gypsum blocks is presented. It is argued that this constructive system reduces the manpower considerably, the time of execution and the final costs of construction. This is particularly important in a country whose objective is to achieve, until the end of 2014, 3 million of new dwellings. However, the durability assessment should be analysed but first its implementation and performance along the country must analysed.

The hygrothermal performance of the gypsum house was assessed by advanced modelling, using the EnergyPlus software. The numerical results carried out allow a discussion over the interior comfort and durability of this modular constructive system.

In this paper, the computer simulation model and the results of a parametric analysis of the dwelling performance on the eight climatic regions defined in Brazilian regulations are presented. The model was validated using the in situ measurements of air temperature.

Results showed that the modular gypsum house can be an interesting solution for Brazil, however improvements and adaptations need to be made in specific regions.

1 INTRODUCTION

In recent years the Brazilian government has made great efforts to improve the population quality of life by launching public funded programs for acquisition of houses. The objective is to achieve, until the end of 2014, 3 million of new dwellings.

These figures expose a clear need for new housing construction in the country. Preferably simple, rapid and easy to implement constructive solutions should be chosen, without neglecting comfort and durability. The use of local endogenous natural resources, such as gypsum, can be an interesting solution both from an environmental and economically perspective.
Hence, an innovative modular constructive system based on gypsum blocks is presented. This constructive system reduces considerably the manpower, the time of execution and the final costs of construction. However, its implementation and performance along the country must be analyzed.

Brazil is a very large country with very different climate. Therefore, this simple, naturally ventilated, modular gypsum houses must be tested under different conditions in order to understand its performance in terms of occupants’ thermal comfort. Although some limitations on the dwellings hygrothermal performance are expected, the inclusion of simple additional constructive elements, such as insulation layers, shading devices and HVAC systems, can be an option to make the solution interesting for application throughout the country. After this sensitivity analysis, and consequent building climatic adaptation, the durability of the solutions must be assessed. The Laboratory of Building Physics of Faculty of Engineering of University of Porto started a research project for the complete characterization of the dwellings performance, including: laboratorial determination of the gypsum properties, in situ evaluation of the dwelling performance by continuous monitoring of the indoor environmental quality, thermal simulation of the dwelling under different climatic conditions, recommendation of additional constructive measures in order to adapt the dwelling for the climate and a durability assessment of the final solutions.

In this paper, the computer simulation model and the results of a parametric analysis of the dwelling performance on the eight climatic regions defined in Brazilian regulations are presented. The model was validated using the in situ measurements of air temperature. EnergyPlus software, for the simulation, and DesignBuilder, for the model creation, were used.

2 THE GYPSUM HOUSE

Plaster is a binder, which compared with other materials as whitewash and Portland cement, can be considered much less aggressive to the environment. However, plaster has relatively little use in Brazil and an effort has been made to increase the consumption of this material in housing. Gypsum, abundant in nature and found in several deposits in northeast Brazil (plaster pole of Araripe, Pernambuco), can be used for manufacturing masonry units and renderings, resulting in a great added value for the local economy and environment.

Gypsum blocks are a building material that promises bring down the cost of construction by at least 30% in comparison with other material such as drywall system, brick walls or concrete/cement block walls. Also, gypsum blocks offer a far superior technical specification in terms of acoustic insulation (sound reduction 38 Db), heat insulation and fire resistance. The gypsum block wall presented in this paper does not require any kind of insulation material; as a result, the cost of building an interior wall reduces dramatically as it is faster to build. Other advantage is that it is an environment control system, as gypsum material naturally balances humidity and temperature allowing to both save energy and offer a termite-free and mould-free environment.

Starting from the experiences carried out in African countries in the eighties, the interior comfort and durability of the construction process with gypsum prefabricated blocks is being studied. A prototype was built in Petrolina and its indoor environmental quality is being monitored (Figure 1).

This dwelling has three bedrooms, two bathrooms and an area that includes kitchen and leaving room, with the organization and dimensions shown in Figure 2.
3 THE BRAZILIAN NORMATIVE RECOMMENDATIONS

Current Brazilian regulation NBR 15220-Part 3 [1] divides the country into eight climatic regions, named Z1 to Z8 (Figure 3). Figure 3 also includes the percent area of the country that corresponds to each zone. These were the zones used for the sensitivity analysis of the dwelling thermal performance. A complete description of the climatic data can be found in [1].

Table 1 shows the city used as representative of each climatic region as well as the source of information used to obtain the hourly climatic file required for the simulation.

Additionally it establishes constructive recommendations for dwellings of social interest in accordance with the climatic region. These recommendations include: dimension of ventilation openings, shading strategies, strategies for passive thermal conditioning and limits for envelope (wall and roof) thermal properties (heat transfer coefficient, U (W/(m².K)), thermal delay factor, φ (h), and solar factor, FSo (%)). The methodology for the calculation of the thermal properties...
is also included, however, similar information can be found in traditional building physics literature.

The gypsum house envelope thermal properties were computed and compared with the limits proposed by the normative for each climatic region. The gypsum thermal conductivity and density, required for this calculation, were laboratory confirmed. The heat capacity was established from a literature review. Table 2 synthetizes the information. The bold values correspond to the situations where the gypsum house is not in compliance with the limits. The only problematic situation is related with the thermal delay factor, which is defined as “the time between a thermal variation and its manifestation on the opposite surface of a building component, subjected to a periodic heat transfer regime”. Walls do not respect the thermal delay factor limit in zones Z4, Z6 and Z7. Regarding the roof this situation only occurs in zone Z7. Hence, according to the normative recommendation, the gypsum house envelope is adequate, with its actual properties, to be implemented in zones Z1, Z2, Z3, Z5 and Z8.

Table 2: Wall and roof thermal properties.

<table>
<thead>
<tr>
<th></th>
<th>Z1</th>
<th>Z2</th>
<th>Z3</th>
<th>Z4</th>
<th>Z5</th>
<th>Z6</th>
<th>Z7</th>
<th>Z8</th>
<th>Gypsum house</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wall</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>U (W/(m².K))</td>
<td>≤3.0</td>
<td>≤3.0</td>
<td>≤3.6</td>
<td>≤2.2</td>
<td>≤3.6</td>
<td>≤2.2</td>
<td>≤2.2</td>
<td>≤3.6</td>
<td>1.9</td>
</tr>
<tr>
<td>Ø (h)</td>
<td>≤4.3</td>
<td>≤4.3</td>
<td>≤4.3</td>
<td>≥6.5</td>
<td>≥4.3</td>
<td>≥6.5</td>
<td>≥6.5</td>
<td>≤4.3</td>
<td>4.1</td>
</tr>
<tr>
<td>FS₀ (%)</td>
<td>≤5.0</td>
<td>≤5.0</td>
<td>≤4.0</td>
<td>≤3.5</td>
<td>≤4.0</td>
<td>≤3.5</td>
<td>≤3.5</td>
<td>≤4.0</td>
<td>2.3</td>
</tr>
<tr>
<td>Roof</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>U (W/(m².K))</td>
<td>≤2.0</td>
<td>≤2.0</td>
<td>≤2.0</td>
<td>≤2.0</td>
<td>≤2.0</td>
<td>≤2.0</td>
<td>≤2.0</td>
<td>≤2.3</td>
<td>1.8</td>
</tr>
<tr>
<td>Ø (h)</td>
<td>≤3.3</td>
<td>≤3.3</td>
<td>≤3.3</td>
<td>≤3.3</td>
<td>≤3.3</td>
<td>≤3.3</td>
<td>≥6.5</td>
<td>≤3.3</td>
<td>2.0</td>
</tr>
<tr>
<td>FS₀ (%)</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>≤6.5</td>
<td>5.4</td>
</tr>
</tbody>
</table>

4 SIMULATION MODEL

The use of simulation tools has increased significantly and is universally acknowledged its contribution to finding solutions that enhance energy efficiency in buildings, notably the choice of the ventilation system, constructive solutions to the building envelope, heating system, more efficient lighting equipment, solar protection systems... Given the objectives of this study, EnergyPlus [3, 4] software was chosen for the simulation of the dwelling thermal performance. The software DesignBuilder [5] was used to create the models.

The building geometry, previously described in section 2, includes six different thermal zones: Living room, Bedroom 1, Bedroom 2, Bedroom 3, WC 1 and WC 2. Figure 4 shows the model created.

The dwelling is naturally ventilated. Ventilation was modeled with the following assumptions: constant infiltration rate of 0.25 h⁻¹, in all zones due to envelope air tightness; natural ventilation with a constant rate of 3 h⁻¹; in all zones with a temperature set-point of 18 °C and, to avoid warm air being introduced, the outside air must be at least 1 °C colder than the inside air.
The simulation model was validated using *in-situ* measurements of air temperature. The experimental dwelling described in section 2 is being continuously monitored and the available data, together with the real weather data of Petrolina, was used to validate the simulation model in terms of air temperature simulation. Additionally, real data regarding internal gains, such as occupation profile and density, lighting and equipment, was also used for model calibration. As stated before, the material properties were established in laboratory. More information about the simulation model properties and calibration procedure can be found in [6].

The air temperature was recorded by means of wireless sensor WLS-05, which has a precision of ±0.5 °C and a resolution of 0.01 °C. The sensors were located on the gypsum house prototype described in section 2 (Figures 1 and 2), approximately in the middle of the rooms. All the sensors used in this project were calibrated by an independent governmental entity and by the manufacturers.

The simulation results were compared with the *in-situ* measurements to validate the model. As an example, the result obtained for the air temperature simulation inside zone “Living room”, on a typical day of September, is shown in Figure 5, together with the *in-situ* measured air temperature.
5 SENSITIVITY ANALYSIS

The main goal of this paper is to analyse the influence of Brazilian climate in the dwelling thermal performance. Therefore, several simulations were performed considering the dwelling location changing across the different climatic regions of the country. This analysis allows for some preliminary conclusions regarding specific changes in the building construction, required to adapt it and guarantee adequate comfort conditions for its users.

Figure 6 shows the monthly mean air temperature inside the dwelling, considering the eight climatic, in zone “Living room” and “Bedroom 1”. It is also included in the graph the temperature limits (18 and 26 °C) usually assumed in Brazil.

Although some more detailed analysis is required, the results clearly indicate that additional constructive improvements, such as insulation, shading devices and HVAC systems, are indispensable. Thermal discomfort due to low temperatures is definitely a problem in Zones Z1, Z2 and Z3, while overheating problems arise at Z7 and probably in other regions since these mean monthly temperatures.

The second analysis was the use of the simplest well-known method that can be employed to preliminarily estimate buildings energy performance, the degree-hours method, based on human comfort levels and available temperature data for a given space. The degree-hours can be defined as the sum of the differences between air temperature and a reference value and can be calculated for cooling ($DH_{cooling}$ in °C) and heating ($DH_{heating}$ in °C) purposes (equations (1) and (2)).

$$ DH_{cooling} = \sum_{i=1}^{n} (\theta_i - 26) \quad \theta_i > 26 $$

$$ DH_{heating} = \sum_{i=1}^{n} (18 - \theta_i) \quad \theta_i < 18 $$

where $\theta_i$ is the air temperature inside the zone in °C. Figure 7 illustrates the degree-hours for the eight climatic regions.
This refined analysis exposes overheating problems not detected previously. In fact, regions Z5, Z7 and Z8 present a $DH_{cooling}$ value that suggests the need to improve the gypsum house performance.

6 CONCLUSIONS AND PERSPECTIVES OF FUTURE WORKS

A simple and cheap gypsum modular house is presented. The dwelling thermal performance is being monitored in-situ and the already available data was used to calibrate and validate a simulation model. A sensitivity analysis of the building performance across the eight Brazilian climatic regions was performed. The following conclusions can be stated:

− The use of endogenous natural resources is crucial for a sustainable development of Brazil. The country still needs to build a large number of dwellings. The gypsum modular house presented can be an interesting option;

− Brazilian normative NBR 15220-Part 3 [1] divides the country into eight climatic regions and establishes constructive recommendations for dwellings of social interest in accordance with the climatic region. The gypsum house construction properties reveal the need to an adjustment in some regions. However, this preliminary assessment indicates a great potential of implementation for this modular welling;

− The sensitivity analysis and the degree-hours calculation exposed overheating problems in regions Z5, Z7 and Z8 and thermal discomfort due to low temperatures regions Z1, Z2 and Z3;

− As a final conclusion one can say that the modular gypsum house can be an interesting solution, however improvements and adaptations need to be made in specific regions.

After the conclusion of the climatic adaptation of the dwelling, the next step in this investigation includes the durability assessment of the construction. Future works will be done in order to evaluate the service life prediction of the material, by numerical simulation and laboratorial accelerated ageing experiments.
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ACTUAL SERVICE LIFE PREDICTION OF BUILDING COMPONENTS

Research Director, PhD Niels-Jørgen Aagaard(1), Senior Researcher Erik Brandt(2), Senior Researcher, PhD Ernst Jan de Place Hansen(3)

Danish Building Research Institute, Aalborg University, Copenhagen, (1) nja@sbi.aau.dk, (2) ebr@sbi.aau.dk, (3) ejp@sbi.aau.dk

Abstract

In recent years, sustainability and life cycle cost in the construction industry have been given great attention in many countries due to the heavy climatic and environmental impact from this sector. In Denmark, a sustainability certification scheme for buildings has been developed including a condensed method for assessment of life cycle costs for buildings.

Estimation of life cycle costs has traditionally been based on predicted service life for building components in terms of technical performance. This paper suggests a method for taking into account other contributing factors such as economics, aesthetics and use of the building. Each of these factors is regarded as a stochastic variable and can be seen as competing causes of bringing service life to an end. A model is proposed for combining such variables resulting in the actual service life of building components.

Furthermore, the paper presents an analysis of service life of buildings as such with respect to the use of the building partially based on analysis of data from the nationwide Danish register of buildings and housing as well as turnover in the Danish construction industry from refurbishment and demolition activities. Finally, it is discussed how to adjust the model for practical purposes, and a scheme for actual service life for selected building components important for analysis of sustainability is linked. The schemes are now being implemented as basis for sustainability certification of new buildings in Denmark.

1 INTRODUCTION

1.1 Background

Assessment of sustainability of buildings and building components very often includes an analysis of Life Cycle Cost (LCC) or a Life Cycle Assessment (LCA) in terms of environmental impact. Likewise, these analyses are often relevant when considering investments in renovation of buildings. Knowledge on service life of building components is an important part of the basis for assessing LCC. Furthermore, service life of building components is an indispensable part of LCA’s concentrating on economy of energy and CO₂-emissions for solutions to design problems. The result of such analysis relies heavily on the prediction of service life for the relevant building components and materials.

Service life of building components is also applicable to sustainability labelling systems or certification schemes for building components and materials, as the system established by the Danish construction industry in Green Building Council Denmark [9].
1.2 Basis
1.2.1 Standards and data
There is no international or national Danish standard on actual service life (ASL) of building components comprising values for specific components of given materials. The ISO 15686 Standard ‘Buildings and constructed assets - Service life planning’, Part 1-11, describes principles (Part 1) [16], how to deal with experiences from practice (Part 7) [14] and assessment of service life (Part 8) [15], but none of the elements of the standard suggest any figures on ASL or how to predict it. In European context, a system and a register is being developed and is over time expected to contain data on actual service lives for building components in terms of product data [24].

Very little documented information on service life of building components or materials is available in the Danish construction industry. Consequently, lists and files of such data are mainly based on experiences and professional appraisal, of which the most important over the last years are [5] and [8].

From countries with some industrial and trade integration with the Danish construction industry, the following sources to information on service life of building components are relevant, most of them similarly based on experiences and professional appraisal:

- Germany: Lebensdauer von Bauteilen und bauteilschichten [13]
- The Netherlands: Levensduur van bouwproducten [22]

Most of these sources are developed with a certain purpose in mind; e.g. insurance coverage or assessment of life cycle cost. The resulting list of data is influenced by such purposes as they may respect an upper limit or are based on specific percentile of the expected distribution of service life. Consequently, it is difficult to use such data in another context.

1.2.2 Composite building components
Building components are usually composed of a number of parts and materials, e.g. an external wall might consist of – listed from the inside – concrete inner leaf, thermal insulation, outer leaf, façade cladding and painting. Furthermore, secondary parts like vapour barrier and wind barrier may be included. The resulting service life for such components cannot be determined directly, and service life for each of the included parts may be assessed separately. If the building component is composed in such a way that parts with a shorter service life cannot be replaced without destroying parts with a longer service life, the service life of the component is defined by the service life of the parts with the shorter service life; e.g. the service life of an in-situ cast concrete basement slab is usually determined by the service life of the underlying thermal insulation, since this is expected to have the shortest service life and cannot be replaced without destroying the slab.

2 SERVICE LIFE OF BUILDING COMPONENTS

2.1 What is ‘service life’?
2.1.1 Service life for LCC/LCA
‘Service life’ is the time from installation to dismantling of a component in the building. In LCC and LCA, the usual relevant service life to be used is the mean value of service life – in short ‘Average service life’, as these analyses in general acts as supporting tools in the design phase of a construction project and the expected time during which the component may serve
its function is relevant. Consequently, lower or higher value of service life that might occur in other usages, e.g. like insurance coverage estimation, is irrelevant.

2.1.2 Types of service life

Different types of loss in performance in relation to demands may put an end to the service life of a building component. In *Handbook of environmentally sound design of building* [3] it is suggested that service life of a building component can be described by four types:

a. *Technical service life* (TSL) – the time in which the component technical and physical is capable of fulfilling its original function as determined by loads and impacts on the component and its resistance towards these.

b. *Functional service life* (FSL) – the time in which there is a need for the original function of the component as delimited by changes in demands from users and society to the performance of the component.

c. *Economical service life* (ESL) – the time in which it from a LCC perspective is economically feasible to keep the component in service.

d. *Aesthetic service life* (AESL) – the time in which the ‘aesthetic’ performance as determined by socio-technical and psychological factors is intact.

Although these aspects of service life are not fully independent of one another, they are regarded as a sound basis for a practical approach in attempts to predict service life of a building component.

2.1.3 Technical service life

TSL of building components is comprehensively dealt with in the scientific literature; e.g. [17]. Deterministic methods for modelling service life of building components are presented, e.g. in [10]. TSL is usually defined by a set of factors multiplied on a reference service life; e.g. [12] and [15]. The ISO-method is applied to sealed building and construction joints based on probabilistic modelling of uncertainties of the comprised factors [23].

The building component may over time lose its ability to fulfil one or more of its functions, e.g. a façade protecting against both wind, rain and energy loss, triggering either a replacement or maintenance activity [18]. The component has reached its TSL if replacement is necessary.

2.1.4 Functional service life

FSL has so far not been treated in the scientific literature as it is hereby suggested as the overall concept for the description of user related issues, e.g.

- development of society (new demands, economic trends, demographic changes etc.)
- development of the building stock over time (construction vs demand, ageing of existing buildings etc.)
- capacity in the construction industry (construction, rehabilitation, demolition etc.)
- demolition rate for the existing building stock (building conservation policies, reuse of building materials etc.)
- changes in construction basis (legal rules, labour market, accessibility of resources, climate adaption etc.)

Overall, FSL is determined by demands from users and society to the performance of the component. The service life of a building component might come to an end, not because the component has lost its ability to perform, but because there is no longer a need for it in its original form.
2.2 Variation of Service Life

Even though a deterministic method may deal with various factors influencing the service life, the method as such leads to a result expressed as a fixed number of years.

As the service life of a building component in practice will depend on several factors, a fixed number of years are in many cases a relatively weak description of reality and a stochastic modelling of some of the aspects of service life are therefore suggested including mean value, variation and density function.

Density functions may be described as illustrated in figure 1 being symmetrical, asymmetrical or having several tops depending on the number and nature of the factors that influences the functions.

![Figure 1 Examples of density functions of service life for a given building component. Average service life is indicated by vertical lines.](image)

Initially, the types of distributions are not known, but may be determined by empirical data. Unfortunately, no such data exist for conditions and components in the Danish building tradition.

2.3 Actual Service Life

The resulting service life of a given building component – the ‘Actual service life’ (ASL) – is the resulting time from installation to dismantling of a component. ASL is dominated by the type of service life with the lowest mean value.

The four types of service life (Section 2.1.2) may be seen as competing factors, each of them being stochastic variables.

If the types of service life are statistically independent and the density functions are known, the mean value and variation of ASL can be determined by the density function for the minimum service life. If no assumptions are made of statistically independencies and the density functions are unknown, a lower boundary value of the actual mean service life may be determined as the solution to a minimization problem, e.g. [2], provided the mean value and variation are known for each of types of service life.

ASL will in all cases be lower than any of the four competing types of service life. Example: It is assumed that the density function of all four types of service life follows a normal standard distribution, all having a mean service life of 50 years and a standard deviation of some 8 years. Then ASL will have a mean value of approx. 42 years with a standard deviation of 6 years. If no assumptions are made of statistically independencies and the density functions are unknown, the mean value for ASL will be approx. 36 years.
In the real world, the density functions of the underlying types of service life will be different from one another. Example: For normal distributions with \((TSL, FSL, ESL, AESL) = (60/9, 80/6, 50/12, 50/8)\), where \(X/Y\) expresses (mean value/standard deviation) in years, ASL can be calculated to approx. 44 years with a standard deviation of approx. 8 years. Not assuming anything about statistically independencies and the density functions, the mean value for ASL will be approx. 38 years.

3 SERVICE LIFE OF BUILDINGS

3.1 Building stock and demolition rate

The Danish building stock comprises some 660 mi sq.m. of floor area by the end of 2012 with an average age of 56 years, of which housing comprises 49 \% (average are 63 years) (Statistics Denmark, www.statistikbanken.dk/BYGB33).

From 1994 to 2012 approx. 120 mi sq.m. of floor area has been added while approx. 150 mi. sq.m. has been constructed in the same period, indicating substitution of approx. 30 mi sq.m. of existing floor area with new buildings corresponding to a demolition rate of 0,3\% on average in the period.

However, when assessing sustainability in terms of LCA and/or LCC for new constructions, conditions for the existing building stock are not essential, although it is obvious that the share of capacity for new construction decreases as maintenance of the ageing existing building stock will acquire a larger and larger share of the capacity of the construction industry.

Expectations on rehabilitation and demolition in the future are essential to the assessment of service life of buildings. For a demolition rate \(n\), expressed as the share of the existing building stock to be demolished every year, the average age \(A\) in years of the remaining building stock will develop asymptotic to \((N + (1-n)/n)\), \(N\) being an initial period in years for new buildings not to be demolished due to mortgage and legal reasons. For contemporary conditions in a Danish context, reasonable lower and upper estimates might be

- Lower estimate: \((n:N) = (0,005;30)\) \(\Rightarrow A = 229\) years
- Upper estimate: \((n:N) = (0,02;20)\) \(\Rightarrow A = 69\) years.

The single dominating figure in estimating the ageing of the building stock is by all means, the demolition rate. In average, the demolition rate in Denmark in the period 1994-2012 was 0,003-0,004. Unless the demolition rate is increased significantly in the future, new buildings may look forward to become very old; in average probably more than 200 years. If no new buildings were to be constructed and the capacity of the building industry was forwarded solely against demolition, it would correspond to a demolition rate of approx. 0,008. A higher demolition rate than this is hardly realistic except on specific areas where a backlog might be dominating, e.g. abandoned housing or industrial buildings with outdated facilities.

3.2 Functional service life of buildings

In the long run rehabilitation could be a more sustainable strategy than demolition and subsequent new construction since it implies huge volumes of reuse of building materials on site – at least under Dutch [20] and Finnish conditions [21]. Traditionally, more than 50-60 years of service life are seldom used for LCA/LCC for various reasons, e.g. [7]. However, a number of countries will probably increase the maximum average service life of buildings to 100 years or more for use in LCA and LCC; [4], [13], [19] and [22].
Based on these considerations and detailed analysis of the age and demolition rate for various usages, mean values for FSL for buildings as listed in Table 1 have been decided for a Danish context to be used in LCC and LCA.

### Table 1 Average FSL for buildings in Denmark.

<table>
<thead>
<tr>
<th>Average FSL [years]</th>
<th>Usage of Buildings</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>Farming buildings, secondary buildings, sheds, etc.</td>
</tr>
<tr>
<td>60</td>
<td>Facilities for transportation, garages, warehouses, sport, holiday homes</td>
</tr>
<tr>
<td>80</td>
<td>Factories, workshops, offices, retail, hotels, restaurants</td>
</tr>
<tr>
<td>100</td>
<td>Care centres, hospitals, schools, universities, exhibition facilities</td>
</tr>
<tr>
<td>120</td>
<td>Detached houses, blocks of flats, religious buildings, museums, theaters</td>
</tr>
<tr>
<td>100</td>
<td>All buildings, in total</td>
</tr>
</tbody>
</table>

### 4 ASSESSMENT OF ACTUAL SERVICE LIFE OF BUILDING COMPONENTS

#### 4.1 A simplified method for assessment

A simplified method for assessment of ASL of a given building component is suggested based on the considerations presented in previous sections:

1. Assess mean value and standard deviation for each of the four types of service life. The FSL service life cannot for any building component exceed the values in Table 1.
2. If an empirical basis for assessing the density function exist: Calculate the resulting ASL based on this.
3. If no empirical basis for assessing the density function exist: Calculate the resulting ASL without any prior assumptions.
4. The resulting ASL is rounded off according to specified rules; e.g. into a scale of fixed values: 120, 100, 80, 60, 50, 40, 35, 30, 25, 20, 15 and 10 years.

The type of service life with the lowest mean value may be used as reference value and then multiplied by a factor $k$ determined by the ratio of the three remaining types of service life in proportion to the least value as listed in Table 2. This is by a sensitivity analysis shown to be acceptable in light of the uncertainty/shortage of the underlying empirical data. If all 4 types of service life are equal, the lowest value in the range of $k$ is to be used. And if one type of service life has a much lower value than the other 3 types, the highest value in the range of $k$ is to be used.

### Table 2 Factor $k$ to be multiplied on least mean service life for determination of ASL.

<table>
<thead>
<tr>
<th>Standard deviation on types of service life</th>
<th>Range of factor $k$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Service life: Normal density function</td>
</tr>
<tr>
<td>Low</td>
<td>0.92-1.00</td>
</tr>
<tr>
<td>Medium</td>
<td>0.84-0.99</td>
</tr>
<tr>
<td>High</td>
<td>0.76-0.98</td>
</tr>
</tbody>
</table>
Example: The average service life for each of the 4 types is 50 years with normal density function and a medium standard deviation. The calculated ASL is then 50×0,84 = 42 years.

At present no Danish experiences or scientific evidence for assessment of economical or aesthetic service life exists, whereas both values are set to 1,0. ESL is embedded in assessment of the FSL. Likewise, the aesthetic service life is embedded in the assessment of TSL. ASL may then be approximated by a hyperbolic function

\[
\text{ASL} = \text{MIN} \times (1 - 3/(70 \times (\text{MAX}/\text{MIN})-60)) \tag{1}
\]

where \(\text{MIN} = \text{minimum}\{\text{FSL};\text{TSL}\}, \text{MAX} = \text{maximum}\{\text{FSL};\text{TSL}\}\), density functions and standard deviation neither known nor unknown (but somewhere in the middle) and parameters are calibrated so that \(\text{ASL} = 0,7 \times \text{MIN}\) for \(\text{MIN} = \text{MAX}\) and \(\text{ASL} = 0,98 \times \text{MIN}\) for \(\text{MAX} >> \text{MIN}\).

4.2 Scheme for actual service life

Average ASL of building components to be used in LCA and LCC in Denmark based on the presented simplified method for assessment is listed in [1]. The scheme is structured by a universal accepted hierarchal classification scheme of building components (e.g. basis, primary, secondary) vs construction materials (e.g. concrete, wood, steel). Calculated ASL is rounded into a fixed scale of values as listed in section 4.1.

Example from scheme: Internal walls (excl. basement): Assessment: FSL = 100 years:
- Masonry and concrete: \(\text{TSL} >> 100\) years \(\Rightarrow\) \(\text{ASL} = 100\) years \(\Rightarrow\) 100 years
- Gypsum on framework: \(\text{TSL} = 100\) years \(\Rightarrow\) \(\text{ASL} = 70\) years \(\Rightarrow\) 80 years

5 DISCUSSION

Assessment of the average FSL is by all means subject to great uncertainty since it deals with the question of how long building components or buildings as such are expected to have a function in the future. The assessment is hindered by the fact that the building stock is supporting a constantly and dynamically changing society and experiences on service life of previous building tradition are not necessarily applicable for future buildings.

However, the authors are confident that better assessments can be made by including into the question of service life of building components various non-technical matters and narrowing the assessment uncertainties by methods based on more detailed probabilistic modelling of the embedded parameters. Lots of research and modelling work needs to be done, and most importantly: sufficient empirical data is need in order to identify the most important parameters and calibrating models accordingly.

REFERENCES


ALKALINE HYDROLYSIS OF POLYMERS IN CONTACT WITH CEMENT BASED MATERIALS

Lars Wadsö (1) and Ola J. Karlsson (2)

(1) Building Materials, Lund University, Lund, Sweden; lars.wadso@byggtek.lth.se
(2) Physical Chemistry, Lund University, Lund, Sweden; ola.karlsson@fkem1.lu.se

Abstract

Hydrolysis of polymers is a common degradation mechanism under alkaline conditions such as found in contact with humid or wet concretes and mortars. Common building related products that are susceptible to alkaline hydrolysis are flooring adhesives, concrete admixtures, polymeric reinforcement materials and PVC plasticizers. We have developed a method based on isothermal calorimetry – the measurement of heat production rate at constant temperature – that can be used to quantify hydrolysis rates. The method relies on that the heat produced by hydrolysis is close to zero, but that the acid produced by the hydrolysis reaction is neutralized in the alkaline solution, and that the heat produced by this process is high and almost constant (per mol of reaction). With knowledge of this heat (the neutralization enthalpy) it is possible to directly measure the number of hydrolysis events taking place per second, i.e., the true kinetics of the process. Measurements can be made under different pH and temperature conditions, and the result can be used develop kinetic models of hydrolysis. We will in this paper exemplify this with data from calorimetric measurements on flooring adhesives, and discuss the use and limitations of kinetic models developed from such data.

1 INTRODUCTION

Hydrolysis is a common type of chemical reaction between organic substances and water. The most common type of hydrolysis is that of ester bonds, in which a water molecule cleaves the hydrolysed molecule at the ester bond, forming one alcohol and one carboxylic acid. This process is can be exemplified by the simple case of ethyl acetate hydrolysis, in which ethanol and acetic acid are formed.

The rate of hydrolysis increases both under acidic and under alkaline conditions. In the construction field, hydrolysis of polymers is a common degradation mechanism under alkaline conditions, such as found in contact with humid or wet concretes and mortars. Common building related products that are susceptible to alkaline hydrolysis are flooring adhesives, concrete admixtures, polymeric reinforcement materials and PVC plasticizers[1,2].

We have developed a method based on isothermal calorimetry – the measurement of thermal power – to quantify hydrolysis rate [2]. All processes (physical, chemical, biological) produce heat, which often can be measured with isothermal calorimetry. Generally, the
following two equations describe the relation between calorimetric measurements of thermal power \( P \) (W) and heat \( Q \) (J), and the rate \( \nu \) (mol/s) and extent of reaction \( \xi \) (mol/mol):

\[
\nu = \frac{P}{\Delta H} \quad (1)
\]

\[
\xi = \frac{Q}{\Delta H \cdot n_0} \quad (2)
\]

Here, \( \Delta H \) (J/mol) is the reaction enthalpy and \( n_0 \) (mol) is the amount of reacting substance at the start of the process. Note that the isothermal calorimeter continuously measures the thermal power, which can then be integrated between any two times during a measurement to yield the heat. Typically, the reaction starts at time zero and the result can be shown in two graphs: the thermal power as a function of time, and the heat produced as a function of time.

As the thermal power is proportional to the rate of the process, and the heat is proportional to how much that has reacted, isothermal calorimetry is a powerful method to study kinetics if the reaction rate times the enthalpy is high enough to be measured by the calorimeter used. In the case of hydrolysis, the enthalpy is close to zero so the thermal powers produced by the primary reaction are low, but the produced acid will react with the hydroxide ions and this secondary reaction (neutralization) has a high enthalpy, which is close to constant if a strong base like sodium hydroxide is used [2]. The enthalpy of neutralization is -55.81 kJ/mol and we found total reaction enthalpies of -57.3 kJ/mol for poly(vinyl acetate) and -57.9 kJ/mol for poly(lactic acid) [2] indicating that we can determine rates of alkaline ester hydrolysis of all polymers to within a few percent by measuring the produced thermal power [2]. We thus have two reactions: the hydrolysis reaction that we are interested to study the rate of, and the extremely rapid neutralization. The first is rate determining and the second produces the heat that is measured with an almost constant enthalpy. We will in this paper show some examples of results from measurements on polymer dispersions.

2 MATERIALS AND METHODS

Three commercial polymer dispersions commonly used in flooring adhesives were used in the present studies:

1. A vinylacetate/ethylen/vinylester-terpolymer
2. An acrylate/vinylacetate/ethylen-terplymer
3. A styren-acrylate-copolymer

All measurements were made on about 1 g of polymer dispersions added to about 16 mL of NaOH(aq). Measurements were made at 20, 30, 40 and 50 °C and the following concentrations of NaOH prepared from Fixanal (Merck) ampoules were used: 1.0 M, 0.5 M, 0.25 M and 0.10 M. All hydroxide solutions were pre-thermostated to the temperature of the measurement before the polymer was added. This significantly decreases the initial disturbance of the calorimeter.
The measurements were made in eight channel TAM Air isothermal calorimeters (Thermometric AB; now TA Instruments) using 16 mL water as reference. The results were evaluated by the following steps:

- Subtraction of baseline (offset) measured with ampoules with water.
- Multiplication with the calibration coefficient obtained from electrical calibrations.
- Division with the mass of polymer dispersion.
- Tian correction for the thermal inertia of the instruments (time constant 600 s).

3 RESULTS

To exemplify the use of the method we here present selected results. In Fig. 1 are seen results for the three dispersions at 40 °C, and in Fig. 2 the results for one dispersion at different temperatures are shown. It is seen that the hydrolysis of dispersion 3 follows different kinetics than the hydrolysis of dispersions 2 and 3, and that the kinetics are temperature dependent.

Figure 1. Thermal powers per mass of dispersion measured for the three dispersions hydrolysed at 40 °C and 1 M NaOH.
4 DISCUSSION

As noted in the introduction, there is proportionality between the thermal power and the rate of reaction. This means that we can calculate the rate of hydrolysis from the measured thermal power. As an example, the peak thermal power of dispersion 3 in Fig. 1 after about 40 h is about 0.75 milliwatt per gram dispersion. Using -57 kJ/mol as reaction enthalpy the 0.75 mW (=0.75 mJ/s) corresponds to a rate of about 13 nmol/s. Every second, 13 nmol hydrolysis events take place per gram dispersion.

If the whole peak seen for dispersion 3 in Fig. 1 is integrated, a value of about 380 J/g is found. If we assume that the hydrolysis has come to an end after 150 h, we can recalculate this heat into amount of hydrolysable ester bonds: 0.066 mol per gram dispersion.

As we can measure the reaction kinetics at different temperatures, we can calculate the activation energy of the rate determining hydrolysis reaction. This is made by plotting the thermal power (proportional to the rate) as a function of the heat (proportional to the extent of reaction), and using the thermal powers for different temperatures, but at the same heat, as input to the Arrhenius equation.

As the result from a calorimetric measurements directly shows the kinetics of the studied hydrolysis process, it is possible to use the calorimetric results to optimize kinetic models. In Fig. 1 it is seen that dispersions 1 and 2 possibly follow first order (exponential) kinetics, while dispersion 3 behaves more as if it was an auto-catalysed process that accelerates, peaks and then deaccelerates. Kinetic modelling based on data from isothermal calorimetry has been described by Beezer and co-workers in a series of papers; see for example references [4-5].
5. CONCLUSIONS

Isothermal calorimetry is an interesting method to study alkaline ester hydrolysis as the produced thermal power and heat are proportional to the reaction rate and the extent of reaction. The proportionality constant (the reaction enthalpy) is known to within a few percent.

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REFERENCES

Abstract

While researchers and scientists are providing tools to architects and engineers concerning the technical or economic service life prediction of building assemblies, the influence of evolving functional or socio-economic needs are often forgotten. Besides replacement of building components, residential buildings are also challenged to deal with plan alterations, building transformation or even renovation in order to keep up with changing comfort and thermal standards. This means that predicted service lives of building components may be reduced by these early interventions if no additional measures are taken in the way buildings are initially designed and assembled. Therefore, this paper deals with design strategies which extend the useful life of building components. It is analyzed to what extent transformable design strategies - in which reversible connection techniques are combined with reusable building materials - can prolong the use of buildings materials and thereby optimize environmental benefits during the total building life cycle.

The study reveals that this approach can result in major reductions of the environmental life cycle impacts when applied to relevant building layers. The Life Cycle Assessment (LCA) results show that transformable design strategies significantly reduce both material consumption and waste production for building layers with high functional turnovers. The results illustrate how for well-chosen practical applications, reversible assembly techniques and reusable building components support resource-efficient construction. Further development in the field of transformable building design consequently offers architects and engineers alternative solutions for today’s sustainable building practice with environmental benefits over a life cycle perspective.

1 INTRODUCTION

Our traditional building design is responsible for a vast amount of redundant construction and demolition waste in Europe - an amount that is still increasing as the number of building activities is increasing yearly. Our residential building stock cannot easily be adapted to changing needs in e.g. households, the variable factors of everyday life or new comfort standards due to the ‘static’ decisions made at the time of conception. This lack of integrated adaptability causes demolition of large building parts or entire buildings, once structural modifications can no longer be postponed. Consequently, there is a need for a complementary closed-loop approach in building design, enhancing the overall material and waste efficiency of buildings over their total life cycle. Reusing entire buildings and their components, rather
than demolishing them and creating new ones from raw materials is less energy intensive and often more economical [1].

The use of brick cavity walls has been popular in Belgium since its existence and its application has remained prevalent in the residential construction sector by a number of technical improvements made in its detailing ever since, e.g. thermal cavity insulation as a result of the oil crisis in the 70’s. Facing bricks have a high potential for reuse and are characterized by a long technical service life, but to guarantee their success as an environmentally beneficial building material, their entire life cycle must be taken into account [2]. Due to the high durability and long lifespan bricks can be reclaimed, and therefore their design and detailing should maximize the future potential and the overall benefits of multiple reuse. However, in recent years we have observed an undesirable tendency on behalf of mortar use in masonry structures. The switch of traditional recipe mortars, based on many years of experience, to performance based cement mortars focusing on compressive strength, has been disadvantageous for the reuse of bricks in cavity walls in the long term. Where high strength mortar has been used it can be far more difficult to reclaim any bricks, since salvaging them results in the bricks breaking rather than the mortar. Furthermore, bricks may be reclaimable with correct mortar use, but any earlier modification before the demolition phase is difficult due to the mortar that connects the bricks. The use of wet connection techniques impedes in situ dismantling (and reassembling) of building parts when e.g. thermal or aesthetic upgrade of facades would be requested during the life cycle.

To enlarge the reuse potential of building components (in this case bricks) transformable design strategies can be implemented. Transformable building design can help to achieve a prolonged usage of existing buildings and their composing materials, since it anticipates changes during the life cycle of buildings [1, 3, 4]. In these strategies reversible connection techniques are combined with reusable building materials – so that buildings can support changing future needs and consequently, a prolonged use of the same building materials optimizes the environmental benefits during the building life cycle. The aim of this paper is to identify what ‘transformable’ solutions are currently exist for residential brick facades and to evaluate which environmental life cycle benefits can be attributed to transformable design.

2 ASSESSMENT METHODOLOGY

2.1 LCA

Transformable building solutions typically have another distribution of environmental impacts over their total life cycle than conventional solutions. Solutions that anticipate disassembly and reuse introduce atypical assembly techniques and material choices, of which the financial cost and even the environmental impact can initially be higher than for traditional building products [1]. However, the environmental benefits of service life prolongation of building components can result in important reductions of the environmental impact of these solutions when assessed over a total life cycle of buildings.

To compare benefits and drawbacks of transformable building solutions to static solutions, besides the initial construction phase also the use, maintenance, adaptation, and end-of-life (EoL) stage of building components must be accounted for [1]. The environmental assessment method used for the evaluation in this paper is therefore based on an integrated method
including all environmental impacts during the building life cycle, i.e. Life Cycle Assessment (LCA). For the assessment of each building solution the production (building materials), construction (assembly works), use (maintenance, replacements, alterations), end-of-life (removal, waste treatment) and all transport phases are included. The ReCiPe environmental impact method is applied for its suitability in the West-European context. The Swiss data from the Ecoinvent database (version 2.2) are adapted to the Belgian context concerning the electricity mix and the transport processes.

2.2 Assessment scenarios

Buildings are complex artefacts with many uncertainties and long service lives compared to other daily products. It is difficult to predict when future building adaptations will be required, or to determine when a building will no longer be in use. Setting up a range of assessment scenarios is therefore a safe option in order to correctly interpret LCA results.

For these assessment scenarios estimations are needed regarding the building life span, but also the period after which functional alterations are requested and the moment in which building components need to be replaced in order to avoid obsoleteness of buildings. Service life scenarios can be determined at three levels: building, building element (e.g. facade, partitioning) and subcomponent level (e.g. brick, insulation). For this study, in a first scenario it is assumed that a residential building is in use for 60 years based on literature for the Belgian residential building stock [5]. Since the structural frame of a building has an estimated technical life span of 100 years, in a second assessment scenario a life span of buildings of 90 years is assumed.

Besides, functional alterations are expected in each individual building layer during the life cycle of buildings, i.e. the building skin, partitioning, and technical services. The functional service life of building facades is rather than technical factors also influenced by aesthetical and social aspects or comfort norms. A service life of 30 years is modelled based on observed needs for renovation in the residential building stock today. The urgent need for renovation of our (post-war) building stock can no longer be ignored today [6]. Due to rapidly evolving building standards and consumers’ needs, it is expected that, in analogy, new interventions will be needed in the future in the buildings we construct today.

Finally, a technical service life is modelled for each assembled subcomponent based on average reference values derived from literature in the building sector. The values for the estimated service life of the components (ESLC) are calculated using the Factor method outlined in ISO 15686-1 [7] using a typical life expectancy distribution.

2.3 End-of-life options

The connection type and durability are key indicators for the end-of-life options of buildings and their components undergoing change during their occupation [1]. Change can involve periodical upgrade of buildings, replacement of outdated or broken components, and eventually the removal of buildings.

Durable building elements that are assembled with reversible techniques, like bolts, can be reused after disassembly when modifications are made, while e.g. glued building components need to be removed from site and replaced with new building materials.

Secondly, modifications or replacements of building components assembled with non-reversible connections cause the removal of connected building elements - with remaining
technical service lives. Contrary, when replacing an individual building component featuring in demountable and reusable solutions, the surrounding building elements can simply be reused after disassembly and replacement.

Finally, the number of replacements is not only depending on the technical service life of building components, but also on the life span of the building (i.e. when the building is removed) and the functional turnover of each building layer - in this case the building facade. For example, when it is decided to upgrade the existing building envelope for a better performing one, building components need to be dismantled or removed before their technical service life has been reached. Also, some building materials may still have a long remaining technical service life even though a building is being demolished.

Based on these assumptions, the number of replacements of building components can be calculated for the selected building life scenarios. The environmental impacts of the required replacements over the total building life cycle are summed with the initial impact and the end-of-life impact of each facade solution.

3 EVALUATION

3.1 Brick cavity walls / rain screen facades

When we only consider the short-term environmental effects of a building facade using current environmental impact assessment methods and the materials we know today, brick cavity walls may seem suitable from an environmental point of view [1]. However, since brick walls are assembled with rigid mortars modifications are difficult to make and bricks can only be reclaimed after additional cleaning works (careful removal of the mortar) on off-site locations. There are several measures that could improve the general environmental profile for brick cavity walls regarding the maintenance phase, modifications, and the end-of-life phase of buildings (simple recuperation of bricks) [1,3,4], like using mortars which allow recuperation of bricks, introducing dry connections between bricks instead of mortar, and grouping bricks in preassembled panels.

Therefore, four alternative solutions are evaluated and compared in this paper with traditional cavity brick walls (Table 1):

- Type 1: cavity brick wall with lime-based mortars instead of cement mortars;
- Type 2: rain screen wall with dry connected facing bricks;
- Type 3: preassembled steel-back panels brick cladding with mortar connections;
- Type 4: preassembled panels with brick veneer and mortar connections.

The U-value of the building skin is chosen as the base for comparison - i.e. the functional unit - for different facade solutions on the market. An U-value of $U = 0.3 \text{W/m}^2\text{K}$ according to the current regulatory framework - is calculated for each wall category in order to make a correct comparison per square unit of the facade surface.
Table 1: Composition of four brick systems for the building envelope with a U-value of 0.3 W/m²K

<table>
<thead>
<tr>
<th>Category</th>
<th>Composition</th>
<th>Dimensions</th>
</tr>
</thead>
</table>
| Type 1: Cavity brick wall with lime mortar | interior wall – concrete blocks 14 cm  
glass wool insulation 12 cm  
cavity 3 cm  
clay bricks + lime mortar 9 cm |
| Type 2: Facing brick wall with dry connections | interior wall – concrete blocks 14 cm  
glass wool insulation 14 cm  
cavity 3 cm  
clay bricks + dry connections 9 cm |
| Type 3: Preassembled steel-backed brick cladding with lime mortar | interior wall – concrete blocks 14 cm  
glass wool insulation 14 cm  
steel backing tray  
clay brick veneer + mortar 4 cm |
| Type 4: Preassembled brick panels with dry connections | interior wall – concrete blocks 14 cm  
glass wool insulation 14 cm  
steel frame  
clay bricks + dry connections 9 cm |

The cavity brick wall type (Type 1) with lower-strength instead of high-strength mortar enables mortar to be easily removed from bricks after demolition, aiming for reuse instead of recycling. The rain screen walling system with grooved ceramic brick elements (Type 2) connected with reversible connections by means of steel clips instead of mortar speeds up the construction of conventional brickwork. In the third type - a preassembled rain screen walling system of steel-back panels - brick tiles with a profile are mechanically fixed to a steel galvanized receiver tray mounted to the framing system. The clipped bricks in the tray are finished with mortar. Finally, the last studied solution is a prefabricated rain screen walling system that connects bricks without use of mortar: bricks are crafted with notches in the factory so that they can be glided into steel carrier beams, which connect it to the supporting inner wall.

Table 2: Characteristics of studied brick facade solutions

<table>
<thead>
<tr>
<th>Facade categories</th>
<th>Dry connected components</th>
<th>On-site reuse of components</th>
<th>Off-site reuse of components</th>
<th>Preassembly of components</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reference: Cavity brick wall with cement mortar</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Type 1: Cavity brick wall with lime mortar</td>
<td></td>
<td></td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>Type 2: Facing brick wall with dry connections</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>Type 3: Preassembled steel-backed brick cladding with mortar</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Type 4: Preassembled brick panels with dry connections</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
</tbody>
</table>
Table 2 shows the characteristics of each solution in relation to strategies that prolong the component service life and simplify the (dis)assembly. The table shows if dry connection techniques are applied, if component reuse is possible at the building site/after waste treatment and if façade parts have been preassembled under controlled factory conditions - for the exterior facade leaf.

3.2 Results

Three environmental life cycle impact assessment scenarios of the five brick facade alternatives are compared to the initial environmental impacts at the time of construction (year 0) represented in Figure 1:

- LCA scenario 1: building life span = 60 years with facade maintenance/upgrade every 30 years, and cut-off of environmental benefits of recycling and reuse at building EoL;
- LCA scenario 2: building life span = 90 years with facade maintenance/upgrade every 30 years, and cut-off of environmental benefits of recycling and reuse at building EoL;
- LCA scenario 3: building life span = 60 years with facade maintenance/upgrade every 30 years, including environmental benefits of recycling and reuse of components that can be reclaimed at the building EoL.

![LCA scenarios 1&2: Environmental impact after 60 - 90 years](image)

Figure 1: The environmental impact assessment of five building envelopes with bricks shows that traditional brick cavity walls have a lower initial environmental impact than alternative evaluated facade systems, but that life cycle environmental benefits can be achieved with multiple reuse of components in transformable facade solutions (LCA scenario 1&2).

The results of the assessment show that traditional brick cavity walls have lower environmental impacts than alternative evaluated facade systems if we only look at the material impacts at the time of construction. Due to additional carrier components in the transformable brick facade solutions (Type 2-4) like steel clips, steel/aluminium ties, and steel-backing profiles the initial environmental increases compared to facade solutions with (non-reversible) mortar connections. Moreover, the preassembled brick panels (Type 3 and 4) carrying full bricks require large carrier beams, with a high initial environmental impact as a
result compared to the three in-situ assembled solutions. However, preassembly may have large beneficial effects on assembly speed, which reduces significantly the labour costs.

Yet, the reversible connection techniques and reusable building components in transformable solutions - causing a higher initial environmental impact – offer important benefits when we consider the total life cycle assessment scenarios. Figure 2 shows that in the life cycle scenarios - when facades need to be upgraded dismantled, maintained and (thermally) upgraded during their use – the components and connections in transformable solutions can be reused multiple times, thereby prolonging the technical service lives of components. Dry connected brick units (Type 2) consequently have lower life cycle environmental impacts than cavity brick walls using mortars: 15% lower for scenario 1 and 40% for scenario 2.

These assessment scenarios 1 and 2 consider a cut-off rule for the benefits of recycling and reuse of building components at the end-of-life phase. When considering these potential benefits, additional environmental impact reductions can be related to transformable building solutions. The environmental benefits of reuse and recycling can be calculated as avoided production of building materials in the assessment, like avoided production of gravel for the recycling of bricks, or avoided production of bricks in case bricks can be reclaimed at the end-of-life of buildings. Consequently, assessment scenario 3 shows that without cut-off approach, transformable solutions have environmental life cycle gains between 40-50% compared to traditional cavity brick walls (Figure 2).

**LCA scenarios 1&3:**

<table>
<thead>
<tr>
<th>Environmental impact with/without cut-off</th>
<th>Reference: Brick cavity wall with cement mortar</th>
<th>Type 1: Brick cavity wall with lime mortar</th>
<th>Type 2: Facing brick walls with dry connections</th>
<th>Type 3: Preassembled steel-backed brick cladding</th>
<th>Type 4: Preassembled brick panels with dry connections</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><img src="image" alt="Diagram showing environmental impact with/without cut-off" /></td>
<td><img src="image" alt="Diagram showing environmental impact with/without cut-off" /></td>
<td><img src="image" alt="Diagram showing environmental impact with/without cut-off" /></td>
<td><img src="image" alt="Diagram showing environmental impact with/without cut-off" /></td>
<td><img src="image" alt="Diagram showing environmental impact with/without cut-off" /></td>
</tr>
</tbody>
</table>

Figure 2: As a result of the reuse and recycling potential of building components like bricks and steel profiles in transformable facades, the life cycle environmental impacts of transformable solutions are significantly lower than traditional brick cavity walls.

Finally, in order to prolong the useful service life of building components strategies must be inserted that increase the quality of the products and of the execution. Preassembling building products clearly offer benefits in this context, since panels can be assembled off-site under controlled circumstances.

### 4 CONCLUSIONS

This study reveals that applying transformable design strategies can result in major reductions of the environmental life cycle impacts when applied in a realistic building life
cycle scenario. The Life Cycle Assessment (LCA) results show that transformable design strategies significantly reduce the material consumption and waste production for brick walls since they anticipate building upgrade and reuse at the end-of-life stage of buildings. Consequently, for building components with a high potential for reuse and with long component service life, like bricks, steel and aluminum components, insertion of transformable detailing principles can help reducing the environmental impacts of buildings in the long term.

Environmental aspects are only one of the pillars of sustainability. Financial calculations have to be considered in order to establish if reductions of life cycle impacts in environmental terms can also be achieved for the financial part. Besides, preassembly of building solutions can also reduce the financial benefits of transformable solutions compared to solutions that are assembled on site with wet connection techniques. Further development in the field of transformable building design consequently offers architects and engineers alternative solutions for today’s sustainable building practice with environmental and financial benefits over a life cycle perspective.

5 REFERENCES

EXPERIMENTAL STUDY ON CARBONATION AND CHLORIDE MIGRATION COEFFICIENTS OF SELF-COMPACTING CONCRETE (SCC) WITH BINARY AND TERNARY MIXTURES OF FLY ASH (FA) AND LIMESTONE FILLER (LF)

Pedro Raposeiro da Silva (1) and Jorge de Brito (2)

(1) Instituto Superior de Engenharia de Lisboa, Polytechnic Institute of Lisbon, Lisbon Portugal - silvapm@dec.isel.ipl.pt
(2) Instituto Superior Técnico / ICIST, University of Lisbon, Portugal - jb@civil.ist.utl.pt

Abstract
This paper’s goal is to present the results of an experimental study on the behaviour of SCC with binary and ternary mixtures incorporating high quantities of FA and LF regarding degradation mechanisms, namely carbonation and chloride migration.

Despite the requirements in terms of hardened state properties for SCC being the same as for conventional concrete (CC), the mix quantities used are different and that can substantially affect the SCC’s durability, namely regarding in terms of carbonation and chloride penetration. It is therefore relevant to expand the knowledge on the way the aggressive agents mentioned diffuse or penetrate SCC.

For that purpose, a total of 11 SCC mixes were produced: 1 with cement only (C); 3 with C+FA in 30%, 60% and 70% substitution; 3 with C+LF in 30%, 60% and 70% substitution; 4 with C+FA+LF in combinations of 10-20%, 20-10%, 20-40% and 40-20% substitution respectively.

The resistance against CO$_2$ penetration into the SCC was tested by an accelerated laboratory test procedure described in specification LNEC-E391 and in recommendation RILEM CPC-18. The resistance to chloride ions migration was tested resorting to non-steady-state migration tests, according to specification LNEC-E463 based on NT-BUILD-492.

The results obtained indicate that the properties studied are strongly conditioned by the type and quantity of additions used. The use of ternary mixtures also proves to be extremely favourable, confirming a beneficial effect of the synergy between these additions.

Keywords: Self-compacting concrete; Durability; Carbonation; Chloride migration; Fly ash; Limestone filler.
1 INTRODUCTION

According to report ACI 201.2R [1], the durability of concrete is defined by its capacity to resist the different types of degradation mechanisms, both of concrete itself and the reinforcements embedded in it. Reinforced concrete should maintain all the project requirements, namely its functionality, safety and aesthetics, without maintenance costs higher than initially predicted.

There are several factors that may alter the service time of a reinforced concrete structure and they do not depend exclusively on its intrinsic properties. There are numerous conditions, both of environmental exposure and in service, which may lead to the deterioration of reinforced concrete. In the reinforced concrete’s deterioration process mentioned, it is possible to identify a set of degradation mechanisms that condition the behaviour of concrete and the set that causes reinforcement’s corrosion. The specific concrete’s deterioration processes can be subdivided in mechanical, physical, chemical and biological processes.

The mentioned concrete’s deterioration processes, despite not acting directly on the reinforcement, have an indirect action on them because, as they deteriorate concrete, they allow an easier and faster penetration of the reinforcement’s degradation agents. As for the specific corrosion processes of the reinforcement, they occur essentially by carbonation and by chloride penetration that, despite not attacking directly the cover concrete, act on the reinforcement’s passive protection layer by decreasing its alkalinity.

Regarding SCC’s carbonation, it is possible to find, in the publications consulted, different results. While some authors mention, for example, not having found significant differences in the carbonation depth between SCC and conventional concrete (CC) with the same mechanical resistance and for the same exposure period, other authors present penetration values, for SCC, up to 40% below those of equivalent CC [2]. Like the remaining durability properties of SCC, the results available about chloride penetration are scarce and sometimes somehow contradictory in what concerns essentially the factors (in terms of SCC composition) that influence the referred chloride penetration [2; 3].

Therefore, this paper intends to evaluate the influence of the use of FA and LF on the degradation mechanisms carbonation and chlorides diffusion coefficient in binary and ternary SCC mixes at three ages (28, 91 and 182 days) resorting to specification LNEC-E391 and recommendation RILEM CPC-18 for the carbonation test and according to NT Build 492 and specification LNEC-E463 for the rapid chloride migration test.

2 EXPERIMENTAL PROGRAMME

2.1 Materials and mix proportions

The following materials were used: one type of cement complying with NP EN 197-1 (cement type I-42.5 R with specific gravity of 3.14; two mineral additions: fly ash (FA) complying with NP EN 450-1 and NP EN 450-2 with specific gravity of 2.30 and limestone filler (LF) complying with specification LNEC-E466 with specific gravity of 2.72; two limestone coarse aggregates complying with NP EN 12620, gravel 1 with specific gravity of 2.59, \( D_{\text{max}} \) of 11 mm and water absorption of 1.46% and gravel 2 with specific gravity of 2.64, \( D_{\text{max}} \) of 20 mm and water absorption of 0.78%; two siliceous sands complying with NP EN 12620, one coarse (0/4) with specific gravity of 2.55, fineness modulus of 3.70 and water absorption of 1.10% and one fine (0/1) with specific gravity of 2.58, fineness modulus of 2.03
and water absorption of 0.70%; a third-generation high-range water-reducing admixture (S_p) complying with NP EN 934-1 and NP EN 934-2 (a modified polycarboxylic liquid admixture with a density of 1.07) and tap water complying with NP EN 1008.

With the goal of scoping all variants of contents used in the mixes and the corresponding analysis of the binary and ternary mixes of FA and LF, 11 SCC mixes were produced according to the NP EN 206-9. These data is shown in Table 1.

Table 1: Mix proportions and basic properties of SCC

<table>
<thead>
<tr>
<th>Mixes</th>
<th>SCC1.100C</th>
<th>SCC2.30LF</th>
<th>SCC2.60LF</th>
<th>SCC2.70LF</th>
<th>SCC3.30FA</th>
<th>SCC3.60FA</th>
<th>SCC3.70FA</th>
<th>SCC4.10FA20LF</th>
<th>SCC4.20FA10LF</th>
<th>SCC5.20FA40LF</th>
<th>SCC5.40FA20LF</th>
<th>SCC5.40FA20LF</th>
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<tbody>
<tr>
<td>CEM I 42.5 R [kg/m^3]</td>
<td>707</td>
<td>512</td>
<td>297</td>
<td>222</td>
<td>503</td>
<td>290</td>
<td>218</td>
<td>506</td>
<td>506</td>
<td>297</td>
<td>293</td>
<td></td>
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<tr>
<td>Fly ash [kg/m^3]</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>158</td>
<td>318</td>
<td>373</td>
<td>53</td>
<td>106</td>
<td>109</td>
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<tr>
<td>Limestone filler [kg/m^3]</td>
<td>---</td>
<td>190</td>
<td>386</td>
<td>449</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>125</td>
<td>63</td>
<td>257</td>
<td>127</td>
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<tr>
<td>Superplasticizer [kg/m^3]</td>
<td>7</td>
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<td>3</td>
<td>3</td>
<td>5</td>
<td>4</td>
<td>3</td>
<td>5</td>
<td>5</td>
<td>3</td>
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<td>189</td>
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<td>168</td>
<td>170</td>
<td>183</td>
<td>180</td>
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<td>180</td>
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<td>168</td>
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<td>Fine aggregate 01 [kg/m^3]</td>
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<td>457</td>
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<td>443</td>
<td>447</td>
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<td>Fine aggregate 04 [kg/m^3]</td>
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<td>301</td>
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<td>Corse aggregate 1 [kg/m^3]</td>
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<tr>
<td>Corse aggregate 2 [kg/m^3]</td>
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<tr>
<td>W/C [-]</td>
<td>0.27</td>
<td>0.34</td>
<td>0.57</td>
<td>0.76</td>
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<td>0.62</td>
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<td>0.34</td>
<td>0.57</td>
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<td>770</td>
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<td>780</td>
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<td>690</td>
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<td>V-funnel [s]</td>
<td>9.3</td>
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<td>9.1</td>
<td>9.9</td>
<td>7.3</td>
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<td>8.6</td>
<td>9.3</td>
<td>10.8</td>
<td>9.1</td>
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<tr>
<td>L-box [-]</td>
<td>0.91</td>
<td>0.89</td>
<td>0.85</td>
<td>0.82</td>
<td>0.84</td>
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<tr>
<td>f_cm,28d [MPa]</td>
<td>83.6</td>
<td>70.1</td>
<td>42.3</td>
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<td>70.0</td>
<td>42.8</td>
<td>32.6</td>
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<td>62.5</td>
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<td>59.9</td>
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<td>71.1</td>
<td>74.7</td>
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</tbody>
</table>

In order to evaluate only the change in the unit substitution ratios of cement by mineral additions (f_ad by volume), the following conditions were taken into account: the volumetric ratio between mortar and coarse aggregates’ content (V_m/V_g=2.625), as well as the absolute volumes of coarse aggregate (V_p=0.268 m^3/m^3) and mortar (V_m=0.702 m^3/m^3), were kept constant; the volumetric ratio between the total powder content, cement and mineral additions, and fine aggregates in the mix (V_p/V_s=0.80) was kept constant; the volumetric ratio between water and fine material content in the mix (V_w/V_p), as well as the percentile ratio in mass between the high-range water reducing admixture (S_p) and the fine material content (S_p/p%), varied depending on the need for water and S_p of each mix in order to obtain the self-compacity parameters according to the works of Nepomuceno and Oliveira [4] and Silva et al. [5].

2.2 Test methods and sample preparation

The specification LNEC-E391 and the recommendation RILEM CPC-18 were used to determine the resistance to carbonation. For each SCC reference, a 100 mm diameter and
approximately 50 mm thickness sample was tested, in each of the four ages (7, 28, 91 and 182 days of exposure in the CO$_2$ chamber). All the moulds tested were subjected to a process of humid cure by immersion in water at 20 ± 2°C until 14 days prior to the start of the test, i.e., the entry into the carbonation chamber. At that time (14 days), the moulds were sectioned and its tops were protected with an insulating coating. Then, the moulds were conditioned at 20 ± 2°C and 50% of RH, until entering the carbonation chamber (28 days). At that time, the moulds were placed in a chamber with 5±1% of CO$_2$, 60±5% of RH and a temperature of 23±3°C. With 7, 28, 91 and 182 days of exposure to the conditions mentioned, the moulds were removed from the chamber, sectioned and, using a colorimetric method (with phenolphthalein at 0.1%), a measure of the carbonation depth was made.

The test procedure used to determine the chloride diffusion coefficient was the one described in NT Build 492 and specification LNEC-E463. For that purpose, three cylindrical moulds with 100 mm diameter and 50 mm height were used, for each mix and age studied. After the adequate time of wet curing (climate room at 20 °C ± 2 °C and more than 90 % RH), the moulds were pre-conditioned in vacuum and immersed in a saturated calcium hydroxide (Ca(OH)$_2$) solution in distilled water. Afterwards, the moulds were connected to a rubber sleeve in order to contain the anodic solution of sodium hydroxide in distilled water (NaOH 0.3 N), without chlorides. The rubber sleeve, with the mould properly sealed according to NT Build 492 and specification LNEC-E463, was inserted in the sodium chloride catholyte solution (at 10% in NaCl mass in tap water). Then, through the stainless steel plaques on the mould’s tops, an electric potential (according to the specification in point 6.4.4 of NT Build 492 or LNEC-E463) was applied, which forced the transportation by migration of the chloride ions through the mould. After a given test period, the mould was broken by being axially split and the rectangular sections obtained were sprayed with a silver nitrate solution (AgNO$_3$ 0.1 N). From the visible white silver chloride precipitation, the penetration depth was measured. Based on the penetration depth measurement performed as well as in other parameters, it was possible to calculate the chloride ($D_{nssm}$) diffusion coefficient in non-stationary regime from the simplified equation (1):

$$D_{nssm} = \frac{0.0239 \left( \frac{273 + T}{L} \right)}{\left( U - 2 \right) t} \left( \frac{273 + T}{L} \right) \left( x_d - 0.0238 \frac{273 + T}{L} x_d \right)$$

$$D_{nssm} = \text{non-steady-state migration coefficient (x 10}^{-12} \text{ m}^2/\text{s}; U = \text{absolute value of the applied voltage (V); } T = \text{average value of the initial and final temperatures in the anolyte solution (°C); } L = \text{thickness of the specimen (mm); } x_d = \text{average value of the penetration depths (mm); } t = \text{test duration (hours).}$$

3 TEST RESULTS AND DISCUSSION

3.1 Carbonation test

In a first analysis of the results on carbonation depth showed in Table 2, one can conclude that, for the SCC mixes with values of $f_{ad}$ up to 30%, no carbonation effect is observed during 6 months of exposure in the accelerated carbonation chamber. This happens independently of the type of additions used, that is, in the binary mixes with LF (SCC2), FA (SCC3) and $f_{ad}$ of 30%, in the ternary mixes with global $f_{ad}$ also of 30% and obviously in the SCC1 mixes (with cement only), being practically impenetrable to carbonation at the ages studied. For those mixes, no measurable carbonation was detected, at any of the ages studied and consequently
the coefficient were set at a minimum value of 1 mm/year$^{0.5}$.

Table 2: Depth of carbonation and the corresponding coefficients for all the mixes

<table>
<thead>
<tr>
<th>Mix</th>
<th>$d_{km,7d}$</th>
<th>$S$ [mm]</th>
<th>$d_{km,28d}$</th>
<th>$S$ [mm]</th>
<th>$d_{km,91d}$</th>
<th>$S$ [mm]</th>
<th>$d_{km,182d}$</th>
<th>$S$ [mm]</th>
<th>$K_c$ [mm/year$^{0.5}$]</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>SCC1.100C</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>SCC2.30LF</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>SCC2.60LF</td>
<td>5 0.58</td>
<td>9 0.50</td>
<td>13 0.58</td>
<td>16 0.96</td>
<td>48 0.50</td>
<td>64 0.97</td>
<td>22.57</td>
<td>0.97</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>SCC2.70LF</td>
<td>10 0.50</td>
<td>19 0.50</td>
<td>28 1.00</td>
<td>44 0.97</td>
<td>64 0.97</td>
<td>0.97</td>
<td>-</td>
<td></td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>SCC3.30FA</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>SCC3.60FA</td>
<td>5 0.58</td>
<td>10 0.50</td>
<td>11 1.15</td>
<td>16 1.26</td>
<td>32 2.63</td>
<td>40.89</td>
<td>20.78</td>
<td>0.91</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>SCC3.70FA</td>
<td>9 0.58</td>
<td>14 0.82</td>
<td>18 0.50</td>
<td>32 2.63</td>
<td>48 0.97</td>
<td>0.94</td>
<td>-</td>
<td></td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>SCC4.10FA20LF</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>SCC4.20FA10LF</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>&lt;1</td>
<td>-</td>
<td>1.00</td>
<td>-</td>
</tr>
<tr>
<td>SCC5.20FA40LF</td>
<td>6 0.50</td>
<td>13 0.96</td>
<td>23 1.50</td>
<td>26 2.71</td>
<td>37.96</td>
<td>0.96</td>
<td>-</td>
<td></td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>SCC5.40FA20LF</td>
<td>5 0.50</td>
<td>12 1.15</td>
<td>21 2.22</td>
<td>24 0.82</td>
<td>35.53</td>
<td>0.97</td>
<td>-</td>
<td></td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

The remaining mixes show a significantly distinct behaviour, with carbonation right at 7 days of exposure in the accelerated carbonation chamber. Analysing the values shown in Table 2, it is possible to see that the binary mixes with $f_{ad}$ of 60%, independently of the addition used, are those that show greater resistance to carbonation, with average depths, after 6 months of exposure, between 16 mm and 20 mm. As for the binary mixes with $f_{ad}$ of 70%, one can see greater penetrations than those with $f_{ad}$ of 60% and with a greater increase between 3 and 6 months of exposure in the accelerated carbonation chamber. Nevertheless, these mixes show a clear difference in the resistance to carbonation between the SCC2 (with LF) and the SCC3 (with FA). In the case of the first, with LF (SCC2), the depth varies between 47 and 48 mm and in the seconds, with FA (SCC3), varies between 31 and 32 mm, all at 6 months of exposure.

The ternary mixes with global $f_{ad}$ of 60% show a resistance to carbonation slightly lower comparing to the binary mixes with the same $f_{ad}$. Those mixes (SCC5) obtain a carbonation depth, after 6 months of exposure, between 24 mm and 26 mm, that is, 6 mm to 7 mm greater than the corresponding binary mixes.

The carbonation of concrete can also be quantified by the corresponding coefficient, by analysing the graphs of the depth of carbonation versus time, by the expression $x=kt^{1\over 2}$ ($x=kt^{1\over n}$ with $n=2$). By analysing the graphs shown in Figure 1, one can find that the variation of the carbonation depth as time elapses is adequately characterised by the expression mentioned, showing reasonable correlation coefficients, always greater than 0.9.

As found by other authors, e.g. Neville [6], Bertolini et al. [7] or Siddique [8], it is possible to conclude, in our work, for the mixes with W/C ratio lower than approximately 0.36 (namely, SCC1, W/C=0.27; SCC2.30LF, W/C=0.34; SCC3.30FA and SCC4 W/C=0.27), that the carbonation was negligible, considering an exposure of up to 6 months.

From the analysis of the results obtained, it is possible to state that, in the case of the SCC produced with a lower W/C ratio, in general terms, its greater compacity creates an almost impenetrable barrier to CO$_2$, significantly delaying its penetration. To this high compacity mixes is usually associated a low porosity accessible to water (essentially capillary pores), which represents the main route for CO$_2$ penetration. As confirmed in Silva and de Brito [9; 10] with the results of water absorption by immersion and capillary absorption, these are the
mixes (SCC1, SCC2.30LF, SCC3.30FA and SCC4) with the lowest accessible porosity values and also the lowest capillary coefficients.

![Figure 1: Depths of carbonation as function of the √t for all the mixes](image)

### 3.2 Chloride migration test

In a first analysis of the results obtained, chlorides penetration resistance is strongly conditioned by the use of mineral additions in the SCC production and by their type. This can be observed in the values for SCC3 with FA, which are significantly lower when compared to those for SCC2 with LF. These results confirm the ones obtained, for example, by Zhu and Bartos [11] and Audenaert et al. [12]. Comparing our results with those of the authors mentioned, one should highlight that the use of FA may result in a higher resistance to chlorides penetration when compared to the concrete produced only with C or to concrete produced with C + LF. Zhu and Bartos [11] state that the increase of resistance to chloride penetration due to the incorporation of FA observed may be the result of the chloride ions passage by diffusion being more difficult or even blocked, since the FA particles, which are rounder, significantly contribute to a higher compacity of the particles, both of the SCC paste matrix and of the ITZ itself around the coarser aggregates.

Regarding the $D_{nssm}$ variation with age and the $f_{ad}$ value, it is observed that those differences are minimal and decrease significantly with age, i.e. for all the values of $f_{ad}$ the variation from 91 days to 182 days is negligible.

The same is not true for the SCC3 with FA. For these mixes, a small decrease is observed in the value of $D_{nssm}$ with the increase of $f_{ad}$ to 60% at 91 days. That value tends to stabilize, mainly for values of $f_{ad}$ of 60% at 182 days. For $f_{ad}$ of 70 %, the value of $D_{nssm}$ tends to increase despite being still significantly below the one obtained for the SCC2 with LF.

Interpreting the data in Figure 2, there is a consistent coherence when compared to the variation of the $D_{nssm}$ with the tests age as well as with the $f_{ad}$ used. One should also mention the coherence with the results obtained by the other authors mentioned. The best behaviour is that of the mix with 30% of FA (SCC3.30FA) at all ages when compared to all the mixes produced (except for SCC2.60FA which at 91 days shows a slightly lower $D_{nssm}$).

From the analysis of Figure 2, it is observed that the ternary mixes show a very satisfactory behaviour when compared to the remaining mixes. One should highlight the behaviour shown at the first ages by SCC5.40FA20LF with a $D_{nssm}$ lower by 27.6% than SCC3.60FA (both with total $f_{ad}$ of 60%). That difference fades at as age increases, nevertheless maintaining
small values of the migration coefficients for both SCC. The ternary mix SCC4.20FA10LF (with total $f_{ad}$ of 30%) shows, at 28 days, a value for $D_{nssm}$ very close to that of SCC3.30FA, with a difference between them of approximately 3.3%. At 182 days, the difference mentioned increases to approximately 50%, nevertheless still remaining the lowest value of the ternary mixes at that age.

Figure 2: Results of the non-steady-state migration coefficient

4 CONCLUSIONS

This study showed that for SCC mixes with $f_{ad}$ values of up to 30%, one cannot observe any carbonation effect, for 6 months of exposure in the accelerated carbonation chamber. This occurs independently of the type of additions used, i.e. in the binary mixes with LF (SCC2), FA (SCC3) and $f_{ad}$ of 30%, in the ternary mixes with the same global $f_{ad}$ of and obviously in the SCC1 mixes (cement only), revealing to be practically impenetrable to carbonation at the ages studied.

The variations in carbonation depth with the value of the W/C ratio observed in this work agree with what was found by other authors, namely that, for the mixes with W/C ratios lower than approximately 0.36, carbonation was negligible, for an exposure period of up to 6 months.

The chloride migration coefficient is strongly influenced by the amount and type of additions used, as well as by the test age of concrete. The use of FA in partial substitution of C reduces the chloride migration coefficient essentially due to the improvement of the concrete’s permeability. If, on the one hand, the use of FA causes an increase in the concrete’s porosity in the hardened state at younger ages, it also causes a reduction in the average pores diameter with consequences such as a decrease in the concrete’s permeability and a denser ITZ, making it less susceptible to the penetration of chlorides.

The ternary mixes show very satisfactory results when compared to the remaining mixes. The behaviour of the mix with $f_{ad}$ of 20% FA and 10% of LF should be highlighted. The incorporation of FA in the ternary mixes turned to be very beneficial to the behaviour of those mixes concerning the penetration of chlorides.
ACKNOWLEDGEMENTS

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REFERENCES


RESISTANT-BASED MOISTURE CONTENT MEASUREMENTS ON WATER REPELLENT TREATED MORTAR AND BRICK

Kathrin A. Sachse (1), Klaus Littmann (1), Ragna L. Fege (1) and Xenia Oremek (1)

(1) Faculty of Architecture and Landscape Sciences, Leibniz University Hannover, Hannover – sachse@ibw.uni-hannover.de; littmann@ibw.uni-hannover.de

Abstract
In the context of this study the suitability of an electrical resistance based moisture content measurement method, which is known to guarantee reliable values for moisture contents of wood, were tested for selected mineral building materials with and without water repellent treatment. For this purpose specimens of cement-mortar and solid brick were produced. The water repellent treatment of the mortar was carried out by internal hydrophobation and surface treatment; the bricks were hydrophobated by surface treatment only. The specimens were provided with electrodes. For this purpose the measuring point at the tip of the electrodes was glued conductively into a borehole in the material. The remaining outer part of the electrodes was sealed with electrically isolating glue in order to establish a conductive connection in the middle of the specimens only. After storage in different climates the electrical resistance of the specimens was recorded using data logging devices. Based on gravimetrically measured moisture contents resistance characteristics for each material were established. A satisfying accuracy resulting from the comparison between gravimetrically measured and arithmetically developed material moisture contents confirmed the suitability of electrical resistance based moisture content measurements for selected treated and untreated mineral materials.

1 INTRODUCTION
Because of their pore structure mineral materials and water interact with each other. The absorbed water from the environment – whether liquid or vaporous - as well as the added water during the production process of cement bound materials results in a moisture content, whose quantification is essential at performance tests, such as equilibrium moisture content or workability of screeds. The determination of moisture content can be carried out with a multitude of measuring methods, which are divided into direct and indirect measuring methods [1]. At direct measurements the water has to be removed from the material and afterwards to be determined gravimetrically, volumetrically or by chemical analysis. Measuring methods such as the oven dry method and the calcium carbide method according to DIN 18560-4 [2] belong to this category. Latter method leads to lower values for moisture content in cement bound materials in comparison to the oven dry method. During the drying process partly chemical bonded water is also removed from the cement bound material [3]. A disadvantage of the oven dry and the calcium-carbide method for water content measurements is that some disruptive sampling is necessary, with which a repetition of measurements at the same sample is impossible.
Indirect measurement methods consult another quantity, which allows a conclusion to be drawn about the water content. Typical measuring systems are hygrometric and electrical methods, since these methods lead to destructions on a very low level because of electrodes and measuring probes in boreholes [4]. Belonging to the usual electrical methods the resistance-based moisture content measurement determines the electrolyte resistance, which correlates significantly with water content in the construction.

Electric moisture content measurements are regarded as being acknowledged and practicable with its satisfying results in wood technology. Brischke et al. [5] suggested a resistance-based wood moisture content measurement, which determines the electrical resistance by means of glued electrodes and data logging devices. The values of electrical resistance were used to create resistance characteristics by means of a regression function, which allows a temperature-depending calculation [6]. The aim of this study was to investigate the suitability of the resistance-based method suggested by Brischke et. al [5] for moisture content measurements of untreated and water repellent treated mortar and brick.

2 MATERIALS AND METHODS

2.1 Preparation and conditioning of specimens

In total, 240 specimens were prepared from cement-mortar and solid brick. Therefore slabs of cement mortar (CEM II/B-S 32,5 R) were produced following EN 480-13 [7] with w/c-ratio of 0.6 and 0.7. During the production one slab of each water-cement-ratio was provided with a powdery internal hydrophobation (SEAL 200; ELOTEX, Germany). After storage of the slabs at 100 % RH for 28 d the specimens were sawn in dimensions of 30 x 30 x 50 mm³. To guarantee homogeneous material for the specimens without margin effects the shuttering edges of the slabs were cut away beforehand. The solid brick specimens (hand mould brick, Wienerberger, Germany) were sawn in analogy to those of cement mortar. Half of the specimens of cement-mortar each w/c-ratio and solid bricks were treated with a silane based hydrophobic impregnation (Protectosil®BH-N; Evonik industries, Germany). After submersion in the water repellent agent for 120 min the specimens were stored for 5 d at 20 °C.

Two holes of 4 mm diameter, with a depth of 17 mm and a distance between both holes of 30 mm were drilled into each specimen for installation of measuring electrodes. These were made of polyamide-coated stainless steel cables with a core diameter of 1.2 mm. At the bottom of the holes 0.1 ml of a conductive glue was injected without contaminating its walls. The conductive glue was made from 2K-epoxy resin, ethanol and graphite powder. At the tip of the electrode, the first 5 mm of the plastic coating was removed and the electrode was put into the glue. After 24 h hardening, isolating glue, consisting of the same 2K-epoxy resin and a suspensing agent, was filled into the remaining holes.

In a first series of experiments from a total of 30 replicates per batch, 5 replicates were dedicated to each moisture conditioning regime. For conditioning, specimens were stored in ventilated miniature climate chambers at 20 °C over saturated salt solutions and demineralised water until reaching moisture equilibrium according to EN ISO 12571 [8]. The target relative humidities according to the salt solutions are shown in Table 1. To obtain exact sorption isotherms, the actual relative humidity at moisture equilibrium content was measured by means of an Almemo 2590 device (Ahlborn, Germany).
Table 1: Media for conditioning and target relative humidity

<table>
<thead>
<tr>
<th>media for conditioning</th>
<th>target RH at 20 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>sodium bromide</td>
<td>60 %</td>
</tr>
<tr>
<td>sodium chloride</td>
<td>75 %</td>
</tr>
<tr>
<td>potassium chloride</td>
<td>85 %</td>
</tr>
<tr>
<td>potassium nitrate</td>
<td>94 %</td>
</tr>
<tr>
<td>potassium sulfate</td>
<td>97 %</td>
</tr>
<tr>
<td>demineralised water</td>
<td>100 %</td>
</tr>
</tbody>
</table>

In a second series of experiments the specimens of untreated mortar each w/c-ratio were conditioned additionally at moisture contents above the hygroscopic range. Therefore the specimens were vacuum-pressure impregnated with distilled water (4 kPa for 20 min \(\rightarrow\) 750 kPa for 30 min), kept submerged for further 24 h, and afterwards dried down to the respective target moisture contents of 5, 6, 7, 8, 9, 10 % at room temperature (20 °C). As soon as the specimens reached their target weight, they were tightly packed into polyethylene bags and stored at 5 °C for 7 d to guarantee a homogenous moisture distribution in each specimen.

2.2 Electrical resistance measurements and gravimetric moisture content measurements

The electrical resistance was measured by data logging devices (Materialfox; Scantronik, Zorneding, Germany), which were equipped with three ports. The measuring ranged from \(2 \times 10^4 \Omega\) to \(5 \times 10^8 \Omega\) and a sampling interval of 5 s was chosen. The measuring principle is based on the discharged-time-measurement method. First, a capacitor was charged through a very small ohmic resistance and then discharged through the material to be measured. Based on the time needed for discharging, electrical resistance can be calculated. Directly after measuring the electrical resistance, the specimens were weighed again to determine gravimetric moisture content according to Eq. 1.

\[
u_{\text{grav}} = \frac{(m_u - m_0)}{m_0} \cdot 100 \%
\]

\(u_{\text{grav}}\); \(m_u\); \(m_0\)  
gravimetric moisture content [%]; mass specimen wet [g]; mass specimen oven dried

Based on the corresponding pairs of electrical resistance R and gravimetrically determined moisture content u a linear regression function for each material was calculated by least square procedure. The linear function following Altmann [9] shown in Eq. 2 was used as base function to display the hygroscopic range of material moisture content. The material specific variables a and b were determined with the aid of Microsoft Solver.

\[
u (R) = a + b \cdot R
\]

\(u\); \(R\); \(a,b\)  
electrical moisture content; electrical resistance [10 lg Ω]; material specific variables
3  RESULTS AND DISCUSSION

3.1  Sorption isotherms

The relation between relative humidity and gravimetric moisture content showed the typical behaviour of a sorption isotherm (Figure 1).

![Sorption isotherms at 20 °C for solid brick and cement mortar, treated and untreated](image)

**Figure 1:** Sorption isotherms at 20 °C for solid brick and cement mortar, treated and untreated

The sorption isotherms of mortar, whether treated or untreated, with w/c-ratio 0.7 resulted in expectedly higher values in comparison with those of w/c-ratio 0.6. The internal hydrophobation had no considerable influence on the degree of adsorption as shown by similar behaviour of the sorption isotherms for untreated and treated cement mortar with internal hydrophobation. Similar results were achieved by Wittmann *et al.* [10] with a 2 wt % silan emulsion for high water-cement-ratios. At a lower w/c-ratio of 0.4 the isotherms differed significantly at higher relative humidity, which were attributed by Wittmann *et al.* [10] to the suppression of capillary condensation in the pores. The sorption isotherms of cement mortar with hydrophobic impregnation adopted lower values for moisture contents. The water repellent mortar absorbs less water than the untreated mortar, in which the difference is more pronounced at high relative humidity [11]. Earlier studies by Johannson *et al.* [12] verified lower degrees of adsorption for hydrophobated concrete, in which the moisture content inside the mortar at the time of the hydrophobic treatment had a significant effect on the sorption isotherms at higher relative humidity, because the hydrophobic agent cannot enter pores that are filled with water. This influence of a hydrophobic impregnation on the degree of adsorption was also confirmed for other porous materials e.g. ceramic brick and calcium silicate by Carmeliet [13].
3.2 Resistance characteristics

Resistance characteristics show the relationship by means of a graph between logarithmic values for resistance and gravimetric moisture content. The higher the moisture content the lower the values for electrical resistance are contained [14].

For mortar of each w/c-ratio treated with the internal hydrophobation resistance characteristics were determined for the hygroscopic range shown in Figure 2 and Figure 3. The resistance characteristics served with an acceptable coefficient of determination as a regression function of the recorded measuring data. The comparison of both resistance characteristics revealed, that the electrical resistance of mortar treated with internal hydrophobation with w/c-ratio 0.6 showed lower values as those with w/c-ratio 0.7 at equal moisture contents.

The value pairs of the mortars treated with hydrophobic impregnation manifested itself in a scatter plot in an interval between 84.5 and 86.0 10 $lg \Omega$. Similar effects were achieved for solid brick, as a result of its very low water adsorption. On that account resistance characteristics could not be established for mortar treated with hydrophobic impregnation and for treated and untreated solid brick.

![Graph showing resistance characteristic](image)

Figure 2: Resistance characteristic for mortar treated with internal hydrophobation (w/c-ratio 0.6) at 20 °C for the hygroscopic range
In contrast, the resistance characteristics of untreated mortars obtained significantly higher values of electrical resistance in comparison with the mortars treated with internal hydrophobation. In Figure 4 and Figure 5 the resistance characteristics of the untreated mortars are shown, in which the measuring data above the hygroscopic range were taken into account additionally.

Figure 4: Resistance characteristic for untreated mortar (w/c-ratio 0.6) at 20 °C for the whole moisture range
The different values of electrical resistance at equal moisture content of the untreated mortars in comparison with those of mortars treated with internal hydrophobation pointed to a higher amount of electrical conductive ions, which were added through the internal hydrophobation.

4 CONCLUSIONS

The results lead to the conclusion, that the introduced measuring method, which had been well established for wood based materials, can be generally transferred onto mortar - untreated and treated with internal hydrophobation. The benefit of the method lies in its minor-destructive, especially economical and time-saving execution. The gluing of electrodes requires little equipment and the measurement of electrical resistance occurs with low expenditure. Furthermore a purposeful position allows a selective determination of moisture content in building components. The analysis of the resistance characteristics of the untreated mortar showed, that these have to be material specific for each w/c-ratio. Further studies on various types of cement as well as different formulations of mortar, such as w/c-ratio, are necessary to ensure a reliable measuring method. Additional well-known influence factors like temperature and chloride ingress should be taken into consideration.

Nevertheless, the method is suited especially for long-term moisture measurements und monitoring - e.g. on screeds, cement-bonded façades in each case, since a calibration of the measuring system with solely created resistance characteristics for materials of interest in laboratory is possible.
ACKNOWLEDGEMENTS

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REFERENCES

DURABILITY OF ROOF UNDERLAYS EXPOSED TO LONG TIME EXPOSURE UNDER IN-USE CONDITIONS

Erik Brandt (1) and Ernst Jan de Place Hansen (1)

(1) Danish Building Research Institute, Aalborg University, Copenhagen

Abstract

In Denmark water tightness of roof underlays is of great importance as the underlay serves as the final barrier against water ingress in roof constructions. A popular trend is pitched roofs with ceilings parallel to the roof surface leaving no or very poor accessibility for inspection and repair of the roof underlay.

Roof underlays used in Denmark are to a large extent based on flexible polymer sheets or fibre boards with a coating on the upper side. The underlays are not supported by a rigid underlay.

The durability of the roof underlay is crucial in order to maintain the water tightness for the entire service life of the roof covering. Especially taking into consideration that there is a frequent use of clay tiles and similar where wind-driven rain has fairly easy access to the roof underlay. Also, some clay tiles allow a relatively high amount of solar radiation to pass through the corner joints thereby allowing degradation due to UV light.

The paper presents results from testing of water tightness of different types of roof underlays including a wood fibre board and a gypsum board. Results are mainly from test specimens of nine different roof underlays which were exposed to in-use conditions in a test house at the Danish Building Research Institute. Specimens at the test house were exposed to the south and to the north respectively. These specimens have been exposed for about 18 years. The results from the test house are supplemented by results from a small number of test specimens taken out from buildings after some years of service.

The results of the investigations are that a major part of roof underlays commercially available in the 1990ies degrade considerably in-use thereby losing its water tightness.

1 INTRODUCTION

For quite some years it has in Denmark been common practice to use roof underlays (also designated sarking felt or underlayment) for sloping roofs with roof coverings of tiles or slates.

The purpose of the roof underlay is primarily to act as a barrier against water penetrating through the roof covering itself. In Denmark some types of roof coverings – especially clay pantiles with small overlapping joints, and fibre cement slates – are fairly open to water especially in the form of wind-driven rain. Further, some clay pantiles allow a relatively high amount of solar radiation to pass through the corner joints. All tile or slate roof coverings will allow drifting snow to pass unless special solutions e.g. mechanically tightened joints are used.

Roof underlays are also used for metal roof coverings in order to catch drips from condensation on the backside of the roof covering.
The roof underlay must not jeopardize the natural moisture transportation by diffusion through the roof construction. Traditionally this has been achieved by using roof constructions with a ventilation gap between the insulation and the roof underlay. In such cases vapour tight materials perform very well as long as the ventilation space has sufficiently large openings to the surroundings.

About 20 years ago a new type of materials was introduced to the market as alternatives to the existing roof tile underlays - not only were they watertight but they were also vapour permeable. A relatively large part of these materials is based on flexible polymer sheets fastened to the trusses, i.e. not supported by a rigid underlay but also wood fibre boards and gypsum boards were used as roof underlays.

These new roof underlays - which are very open to water vapour diffusion - are used in a different way than previously as they are placed directly on the insulation without the traditional ventilation gap between insulation and roof tile underlay. As the ventilation gap is omitted such roof constructions can be made thinner but consequently do not have the possibility of removing moisture by ventilation. Moisture penetrating from the interior of the building must consequently be removed by diffusion through the roof underlay instead of by ventilation under the roof underlay. The materials used for the roof underlay must be water vapour permeable to such an extent that moisture is not accumulated inside the construction.

A popular trend in contemporary Danish architecture is pitched roofs with ceilings parallel to the roof surface leaving no or very poor accessibility for inspection of the roof underlay. For use in such constructions roof underlays with long service lives are required – if possible the service life should be the same as for the roof covering.

In earlier studies, [1] and [2], the most important properties for roof underlays were found to be:

- **Tightness against precipitation** - this property is especially required during the construction period until the primary roof has been laid.
- **Tightness against water** - this property covers standing water as well as water running on the surface. When well-constructed no ponding should occur on the roof underlay.
- **No tent effect** - tent effect refers to the well-known fact that touching of the inside of a tent during rain may cause penetration of water. For underlayment laid directly on wood and insulation, no tent effect should occur, as it would impair the water tightness.
- **Water vapour permeability** - for unventilated constructions it is evident that moisture from the interior of the building can only escape through the underlayment by diffusion. Consequently the material shall be very permeable to diffusion of water vapour.
- **Moisture accumulating properties** - are a supplementary asset if all other requirements to the roof are fulfilled. It allows the up-take and accumulation of moisture during periods with high exposure. The moisture is allowed to be removed during other periods.

### 2 RESULTS FROM TEST HOUSE

To investigate the behaviour of unventilated roofs a new roof construction was made on the test house at the Danish Building Research Institute (SBi) in 1995. The roof has a slope of 40° (1:1.2) and consists of 11 pairs of elements, each pair with an element oriented towards north and south respectively.
Each element is 1 m wide and has a height of 240 mm. 10 pairs are unventilated and the last one - acting as a reference - is ventilated. The elements are made with two timber members as sides and with a gypsum board as interior surface. There is no vapour barrier but the gypsum board is painted to achieve a desired water vapour permeability. The elements are totally filled with mineral wool as thermal insulation. On the outside the roof underlay is placed directly on top of the insulation material. The roof underlay is fastened to the rafters and a counterbatten of 22 mm is attached to the rafters over the underlayment. The roof is finished with battens and roof tiles.

The climate in the test house is controlled in winter time to 23 °C (73 °F) and 60 % RH, i.e. a very high humidity level compared to the expected 30 % RH or less under winter conditions. The 60 % RH is chosen to make the exposure as harsh as possible simulating the worst possible in-door conditions. The performance of the tested roof underlays as regards their ability to let moisture from the interior of the building pass has been reported earlier [1] and [2]. The results show that membranes with a high permeability to water vapour can function as a roof underlay, i.e. the amount of water penetrating from the interior is removed by diffusion without causing accumulation of humidity in the construction.

For the present investigation 9 of the roof underlays have been removed from the test house and have been tested as regards tent effect and water tightness (under a head of water). Only the ventilated roof tile underlay and an underlay of plywood with a liquid applied moisture barrier were not tested. The nine roof underlays were tested separately for the specimens facing south and north respectively.

The specimens taken out were very dirty and all looked deteriorated to some degree. Examples are shown in figure 1. The specimens had clear marks of the battens. For one set of specimens small parts of the protecting membrane had fallen off, see figure 2. The wood fibre board and the gypsum board had no stains or discolourations from water on the back.
Figure 2: Examples of test specimens (roof underlays) removed from the test house (a, b, c and d). It is clearly seen where the battens have touched the roof underlay. (c): The roof underlay in (c) has damages due to contact with the battens. (d): Close up of damages in (c).

2.1 Testing of tent effect

Testing of tent effect comprises normally an accelerated ageing followed by a test of water tightness of the product when placed in contact with the substrate. For this investigation no accelerated ageing was performed as the intention was to find out how the roof tile underlays were deteriorated under in service conditions.

During the test of water tightness a flat aluminium "tray" is used to collect any water penetrating the underlayment during the water exposure, see figure 3. In the lower end of the tray a piece of mineral wool and in the upper end a piece of plywood is placed. Both materials are slightly higher than the aluminium tray. The underlayment is mounted on a wooden frame which fits around the aluminium tray. When mounted the frame is placed around the tray causing the underlayment to rest around the mineral wool and the plywood respectively. The underlayment is held in place by the weight of the frame and the underlayment themselves.

This situation is intended to simulate the conditions in a real roof. Water is sprayed from a nozzle over the entire surface of the specimen for 6 hours. The water pressure in front of the nozzle is very low simulating a fine to medium rain. The test method was in the late 1990ies elaborated into a Nordtest test method, NT BUILD 488, "Roof tile underlays: Water tightness..."
- Tent effect” (may be downloaded from www.nordicinnovation.org). The result using this test method is now part of the necessary documentation needed to obtain an “approval” of roof tile underlays in Denmark. The requirement used is that a maximum of 15g of water must pass during the 6 hours exposure to water.

![Diagram of testing setup](image)

**Figure 3:** Testing of tent effect. The roof underlay is placed over a substrate partly of plywood partly of insulation material. Any water passing through the underlayment during exposure to water spray from the nozzle is accumulated in the aluminium tray and weighed.

<table>
<thead>
<tr>
<th>Material no.</th>
<th>Type of roof underlay</th>
<th>North facing [g water passed during the exposure to water]</th>
<th>South facing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Flexible sheet</td>
<td>762</td>
<td>685</td>
</tr>
<tr>
<td>2</td>
<td>Flexible sheet</td>
<td>9</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>Flexible sheet</td>
<td>124</td>
<td>42</td>
</tr>
<tr>
<td>4</td>
<td>Flexible sheet</td>
<td>120</td>
<td>181</td>
</tr>
<tr>
<td>5</td>
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<td>3221</td>
<td>2080</td>
</tr>
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<td>6</td>
<td>Flexible sheet</td>
<td>120</td>
<td>171</td>
</tr>
<tr>
<td>7</td>
<td>Flexible sheet</td>
<td>841</td>
<td>1821</td>
</tr>
<tr>
<td>8</td>
<td>Wood fibre board</td>
<td>128</td>
<td>264</td>
</tr>
<tr>
<td>9</td>
<td>Gypsum board</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

**Table 1.** Results of the testing of tent effect expressed as grams penetrated through the roof underlay after 6 hours water spray. The requirement normally used in Denmark is that a maximum of 15g of water must pass during the exposure to water.

2.1.1 Test material

The materials used for the testing were 7 different types of flexible sheets, 1 wood fibre board (oil impregnated) and 1 gypsum board (with surface impregnation). The materials were
all intended for use as unventilated roof underlays and commercially available on the time they were mounted in the test house.

Only one specimen of each type was available for the test because the test specimens are rather large. The results of the tests are shown in table 1.

Only one of the flexible sheets passed the test. The wood fibre board becomes moist during the test and water penetrates. The gypsum boards failed, as the carton on the upper side had deteriorated before the test and further lost adhesion to the gypsum core during the initial period of testing.

3 TENT TESTING OF SAMPLES TAKEN OUT FROM EXISTING BUILDINGS

For some years samples taken out from existing buildings have been tested, typically because problems were experienced with the roof underlay. A project was started together with a manufacturer to investigate the problem in a more systematic way. Unfortunately only one sample was taken out before the manufacturer decided to stop the project.

More than 20 roof underlays taken out from existing buildings have been tested and only one passed the test. This shows that there is a problem with lack of water tightness either because the products used have never been watertight or because they have deteriorated under service conditions. Many of the products tested have very poor performance as regards tent effect and are unlikely to provide protection against penetration of water.

The one product that passed the test was taken out from a leaking roof. In that particular roof the insulation layer in the roof was thicker than the nominal value (but still within the tolerances for thickness of the insulation). This results in a deformation of the roof underlay so the water cannot run off freely underneath the battens, see figure 4. Instead the water follow the slope of the roof underlay towards the trusses which means that water on the roof is accumulated at the counterbatten where it can penetrate through the holes from the nails. This means that focus not only should be on the material itself but also on the design and workmanship.

Figure 4: Due to insulation material thicker than the trusses the roof underlay is pressed up against the battens in the middle. The water cannot run off freely and instead follow the slope of the roof underlay towards the trusses where it accumulates and results in water penetration at the nails.

4 TESTING OF WATER TIGHTNESS

As a supplement to the testing of tent effect the specimens from the test house were also tested for water tightness under a head of water, in the same way as used in previous studies [3].
Tests of water tightness were performed in a plexiglass/acrylic glass cylinder with a 200 mm head of water. A filter paper was placed under the tested material to simulate tent-effect. Water tightness between test material and container is obtained by a rubber sealing supplemented with silicone grease. The container is pressed against the lower plate with bolts in the four corners, see figure 5. The weight of the filter paper was determined before and after 60 minutes of testing. The tests were considered failed if the water uptake exceeded 0.5 g. The results of the test are shown in table 2.

![Container](image)

**Figure 5.** Test set-up for water tightness. The test material is placed on the lower plexiglass/acrylic glass plate. Water tightness against the specimen is secured with a rubber seal and the container being pressed against the lower plate with bolts in the four corners. Water is poured into the cylinder until a water level of 200 mm above the test material.

Table 2. Results of test of water tightness. The ratio between the number of failed and tested specimens after exposure to 200 mm head of water for one hour. The gypsum board was not tested as the carton was deteriorated and partly without adhesion to the gypsum core.

<table>
<thead>
<tr>
<th>Specimen number</th>
<th>Material</th>
<th>Failed/tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Flexible sheet</td>
<td>3/3</td>
</tr>
<tr>
<td>2</td>
<td>Flexible sheet</td>
<td>0/3</td>
</tr>
<tr>
<td>3</td>
<td>Flexible sheet</td>
<td>3/3</td>
</tr>
<tr>
<td>4</td>
<td>Flexible sheet</td>
<td>3/3</td>
</tr>
<tr>
<td>5</td>
<td>Flexible sheet</td>
<td>3/3</td>
</tr>
<tr>
<td>6</td>
<td>Flexible sheet</td>
<td>3/3</td>
</tr>
<tr>
<td>7</td>
<td>Flexible sheet</td>
<td>3/3</td>
</tr>
<tr>
<td>8</td>
<td>Wood fibre board</td>
<td>3/3</td>
</tr>
<tr>
<td>9</td>
<td>Gypsum board</td>
<td>-</td>
</tr>
</tbody>
</table>
The results of these tests are similar to the testing of tent effect, i.e. a very large rate of materials failed.

A small investigation of the wood fibre board showed, that a pond of water (roughly 1 mm thick) placed on the surface was absorbed in the fibre board within a few minutes. Only after repeating the ponding four times small stains appeared on the back, i.e. the fibre board is able to absorb a fair amount of water before penetration to the back.

5 DISCUSSION

A prerequisite for the introduction of new materials without in-use experience is that the performance properties are documented and that the properties fit with the performance requirements for the use including the ability to withstand the exposure to the expected environment. Ideally such information should be given by the manufacturer but unfortunately information is often not available as was the case for the products tested in this investigation.

The results from the tests where 8 out of 9 materials fail after only 18 years of service are very disappointing and could mean increasing problems with leaking roofs in the years to come. These results are in accordance with the findings in a study carried out by Atelier Dek [3] where also big problems with service life were revealed. The need for better methods of accelerated ageing has also been discussed within CEN/TC 254 WG 9/10 [4], apparently because there is agreement about the need for better methods. Fortunately many of the products marketed in the 1990ies are no longer on the market and hopefully the new products have better performance than the tested ones.

The roof underlays of oil impregnated wood fibre boards and surface impregnated gypsum boards are no longer on the market. However, it is interesting to notice that neither of these had stains or discolourations on the back. This is possibly due to relatively small amounts of water penetrating through the roof covering itself and the fact that both are able to absorb rather large amounts of water.

The present results are in accordance with earlier findings [3] where it was concluded that the methods used for long term ageing of roof underlays are not sufficient to predict the service life of roof underlays. At least not with the type of roofing materials used in Denmark.

From building surveys and damage investigations a number of problems have been encountered apart from the problems with the materials themselves. The major problems seen so far are: 1) Mould growth on the back of the roof underlay due to vapour barriers that are not sufficiently airtight in order to avoid convection of moist indoor air to the roof construction. 2) Insulation layers pressing the roof underlay against the batten – as described earlier in this paper. 3) Insufficient designed details which allow penetration of water.

It is proposed that manufacturers should not only give guidance on what the products can be used for but should also provide well documented information on the expected service life of the product under the intended use. Besides manufacturers and/or suppliers should provide guidance for installation including information on all necessary details.

The building research bodies should help in making accelerated test methods simulating the in-use exposure in order to give reliable prediction of service lives.

6 REFERENCES

Abstract

Pore connectivity determines transport properties of concrete, including the moisture transport. The drying rate of concrete is a process that strongly depends on the transport properties of concrete. This process may be dominated by the evaporation rate near the surface of concrete or the diffusion of moisture through concrete. The relationship between the rates of both processes is dependent on the temperature and vapor pressure. If concrete samples are oven dried, the evaporation rate is much faster than the rate of moisture diffusion, and the transport mechanism may therefore be effectively evaluated.

In this paper, the capability of the drying rate to be used as a descriptor of the transport properties of concrete is evaluated. As a first step, the proposal is made with a prescriptive perspective. The representativeness of the method that determines the drying rate of concrete samples under controlled conditions is studied. In addition, a relationship is established between the drying rate and the properties of concrete usually used as design parameters for concrete durability in aggressive environments.

1 INTRODUCTION

Aggressive environments compel to design concrete considering criteria in relation with durability, whether from a prescriptive or performance-based perspective. Prescriptive design for durability is based on conformity of values for some parameters of concrete such as the compressive strength, water/binder ratio (w/b), sorptivity, resistivity, permeability, and others. Performance-based design for durability is sustained by the relationship between durability and parameters of concrete whose values are defined to comply with a definite life span of the structure, such as thickness of cover concrete and its properties.

There are some advantages of using transport properties for prescriptive design instead of other properties of concrete. Parrot (9) obtained data that suggests that OPC concrete at depths higher than 20 mm may be considered self-curing for 28 days or even longer, as cover concrete (the outer 20 mm) offers protection from the environment that allows relative
humidity to be over 95%. This means that the drying rate (DR) is a key parameter in relation with the sensibility to curing of concrete. The results presented in (9) imply that the compressive strength of young concrete determined from cores will not be sensitively affected by the lack of curing treatment. On the other hand, pore connectivity of cover concrete is affected by early drying and its transport properties are determinant for durability.

Pore connectivity controls transport properties in concrete, including moisture transport. Moisture transport may occur in the form of water or vapor. The relationship between liquid and gaseous phases of water will depend on the moisture content, temperature, pressure and geometry of the pore structure. Vapor diffuse from high to low moisture content zones. Water also moves through concrete by capillary suction if concrete surface is in contact with a source of water.

A similar transport process occurs for the egress of water. During drying, menisci locate near the surface at the evaporation front, and the capillary flow compensates the loss of moisture when steady conditions have been achieved. Then, the relationship between the capillary suction rate, or sorptivity, and the evaporation rate at the concrete surfaces that are exposed to the environment will define the moisture content of concrete in steady-state conditions. In a specimen, for instance, if the DR is higher than the sorptivity, concrete will not reach saturation and the flow rate will be controlled by sorptivity.

Given the empirical application of sorptivity as a parameter to ensure concrete durability (1), there is not a given inconvenience for adopting the same approach by using the DR. In (11), a sound relationship between sorptivity and DR, both calculated according to IRAM Standard 1871 (2), is presented. This relationship is phenomenologically sustained by the similar driving forces for transport involved in both mechanisms. In fact, the progressions of these two moisture transport mechanisms have been repeatedly modeled with the square root of time (4, 8, 16-17).

In the case of the sorptivity test applied to cylindrical samples such as those considered in (2), there is water ingress through one concrete surface and water egress (although minimum) through the other. This egress is negligible as regards the change in weight of the sample. On the other hand, oven-drying produces water loss through two concrete surfaces simultaneously (the lateral cylindrical surface is waterproofed in (2) to avoid multidimensional transport). If the concrete sample is originally saturated, the initial saturation gradient will be the same for any kind of concrete, and, as the moisture gradient or water content is a function of concrete porosity, the results will indicate the moisture transport performance of the evaluated concrete.

The mass balance in Equation 1 (3) aids for the determination of the distribution of moisture content in concrete. This equation considers a first term for diffusion and a second term for sorptivity. The period required in the test method for capillary suction make it possible to consider the mass involved in the diffusive term on the second level, and assume the simplified hypothesis that the whole mass gain of the sample being tested is the result of capillary suction (wet front hypothesis) (4). This leads to the Darcy, Laplace and Kelvin-Laplace equations and their deductions that allow calculating the capillary ascension L in a capillary tube of radii r (5), Equation 2, where \( \gamma_w \), \( \mu \) and \( \theta \) are the density and viscosity of the liquid and the solid-liquid contact angle, respectively. As it can be noted, this parameter is a function of the square root of time. In contrast, diffusion is the dominant term for drying, and if there is an initial moisture concentration \( h(x,0) = h_0 \) in concrete (e.g., the equivalent to the
saturation state), the solution to Equation 1 is the Equation 3. A simplified version of Equation 3 can be obtained analyzing certain moisture content \((h_o)\). Then the different terms can be grouped into only one coefficient, \(s_a\), Equation 4 (considering a negligible final moisture content), where \(\text{erf}^{-1}\) is defined according to Equation 5. Finally, Equation 6 is obtained, where \(x_{ha}\) is the corresponding depth to \(h_o\). This expression has been widely used and it is applicable to the experimental method analyzed through one front interpretation (colorimetric method for chloride ingress, carbonation front). As said, Equations (2) and (6) are both linear functions of the square root of time. In this sense, Dias (12) experimentally found, however, that the duration over which linearity is manifested is much less for drying than for sorption.

\[
\frac{\partial w}{\partial t} = \frac{\partial}{\partial x}\left(D_w(w)\frac{\partial w}{\partial x_m}\right) - \frac{S_p^2}{2 \rho_t} \frac{\partial w}{\partial x_m} \tag{1}
\]

\[
L = \left(\frac{r \gamma \cos \theta}{2 \mu}\right)^{1/2} \cdot \sqrt{t} \tag{2}
\]

\[
h_{xj} = h_o + (h_s - h_o) \left(1 - \text{erf}\left(\frac{x}{2\sqrt{Dt}}\right)\right) \tag{3}
\]

\[
2\sqrt{D} \text{erf}^{-1}\left(\frac{1 - h_o}{h_s}\right) = s_a \tag{4}
\]

\[
x = \text{erf}(\text{erf}^{-1}(x)) \tag{5}
\]

\[
x_{ha} = s_a \sqrt{t} \tag{6}
\]

According to previous investigations (12-14), different drying regimes will have a different impact on concrete microstructure and therefore will alter in different ways the results of the drying test. Dias (12) claims that the desorption ratio, defined as the weight loss from a saturated condition (i.e. from the end of curing by water immersion) to the start of the sorptivity test, divided by the weight of the saturated specimen, explains to a large extent the variations in sorptivity. Hence, whatever the concrete grade, specimen preconditioning, specimen size or specimen coating, the ensuing sorptivity may be estimated from the desorption ratio.

Regarding the quantification of the drying process to describe the pore connectivity of concrete, much research has been done on the effect of oven-drying on the pore structure of concrete to assess the potential damage that this preconditioning procedure may cause on samples that will be tested in a different way after it. However, limited considerations about the drying procedure as a test method itself have been made. Moisture diffusion is due to a moisture gradient within concrete and water evaporation from the concrete surface. When concrete samples are oven dried, the evaporation rate is much faster than moisture diffusion, and the process is controlled by the flow rate of moisture through concrete. Then, the DR can be determined under controlled conditions so as to obtain repetitive results in relation with pore connectivity. This standard procedure should include a fixed temperature at which drying...
carried out. Higher drying temperature would lead to shorter test time, but this may be objected considering the damage that high temperature may cause on samples.

Then, from a prescriptive point of view, the concrete DR under controlled conditions is an index related to concrete porosity and connectivity. In support of this, Bahador and Jong (8) presented results showing that the DR is sensitive to technological parameters such as curing period, w/b, mineral addition type and percentage of replacement. However, they argue that artificially high temperatures can modify the pore structure of concrete and, thus, concrete DR. It is important to emphasize that the measurement procedure of the index DR should be done under controlled conditions so as it can be reproduced, repeated if necessary and used for comparisons. In this sense, the most convenient temperature would be the highest one that assures minimal affectation of the pore structure of concrete. Moreover, if drying is not a conditioning procedure but a test method itself, oven-drying temperature is not very much a key parameter. This is because any alteration of the sample will have a limited effect on the DR itself, i.e. extended oven-drying results in a higher total amount of removed water but not in any modification of the DR, which is controlled by the flow at the near the wet front. In any case, very high temperatures do not seem convenient either. A high temperature gradient would cause fast drying and a steep moisture gradient leading to an early steady-state transport regime.

In comparison with other methods to assess concrete transport properties, Sanjuán and Muñoz-Martialay (10) found that values of air permeability under steady-state conditions had a large variation coefficient, whereas the variation among the results from the oven-drying procedure was lower. From these data, it may be derived that the preconditioning procedure increases the variation of results of the subsequent test (such as air permeability or sorptivity), as the variation of these tests is the sum of the intrinsic variation in each test and the variation in the preconditioning. Then, one of the advantages of using the oven-drying procedure as a characterizing test for concrete is the absence of alterations due to preconditioning procedures after curing.

Following, DRs of different types of concrete are quantified and contrasted to other concrete properties that are usually applied for prescriptive design for durability. This preliminary evaluation of the potentiality of DR aims to value its usefulness for prescriptive design, but it does not imply that further research allows using it as a parameter for performance-based design for durability.

2 EXPERIMENTAL

Seventeen concretes were analyzed to compare their DR with their sorptivity, porosity, compressive strength and resistivity. The proportions of materials used for concrete production are shown in Table 1. The w/b ratio varied between 0.35 and 0.60, the numbers in the designations indicate the w/b multiplied by 100. In all mixes, a water reducing admixture was used to obtain similar fresh consistency levels. The different types of concrete were made using Ordinary Portland Cement (O35, O40, O40b, O45, O45b, O50, O60, O45C, and A45x) and Blended Portland Cement (C40, C45 and C60); siliceous sand, granitic crushed stone and rounded gravel (only in O45C). Six batches with a different water-reducing admixture in each one were produce with the A45x proportion. Porosity was computed from water absorption under immersion at 24h according to ASTM C1754. Compressive strength was determined in accordance with ASTM C 39 on 10x20cm cylindrical specimens.
DR and sorptivity were tested on concrete samples according to IRAM Standard 1871 (2). Samples were discs of 5cm height sawed from cylindrical specimens of 10x20cm, corresponding to the sector between 3 and 8cm from the base. The lateral cylindrical surfaces of the samples were waterproofed in order to ensure one-dimensional transport. These samples were firstly water saturated, and saturated resistivity was measured applying a 13VAC potential. Then, they were placed in oven for drying, with the temperature was set at 50±2ºC, and their loss on drying were determined at intervals of 24h. The drying procedure was finished when this loss was lower than 0.1% w/w in a 24h period. For sorptivity, only one face of these dried samples (the one corresponding to the plane at 3cm from the base of the original cylindrical specimens) was put in contact with water, 3mm depth, and weight gain was determined at intervals 0.5, 1, 2, 3, 4, 5, 6, 24h, and from then on, every 24h up to a weight gain lower than 0.1% w/w in a 24h period.

As an example, oven-drying and sorptivity results for O35, O40, O45 and O50 are shown in Figure 1. The losses on oven-drying per unit area at different exposure times were calculated considering the moisture egress through both circulars faces of the samples. The sorptivity is only calculated (and generated) through the wet face. Sorptivities and DRs were computed by linear regression of the changes in weight of the whole group of samples as a function of the square root of time. The detailed procedure can be found in (2).

Table 2 shows the correlation coefficients of the regressions. It can be noticed that linearity is similar for drying and sorptivity curves. The mean value for the drying process is 0.97 (s = 0.03) and for capillary suction 0.98 (s = 0.01). This supports the theoretical assumptions involved in Equations (2) and (6).
The comparison between values of the DR and the other properties of concrete are compared in Figures 2 to 5. There, the A45x mixes are represented by solid triangles (▲); C45, C60 and O60 mixes are represented by empty squares (■), and the rest of the mixes by solid squares (■).

Figure 2 shows the relationship between the DR and the compressive strength at 28 days. It can be seen that the higher the compressive strength the lower the DR. This is consequent with both the compressive strength and the DR being closely related to w/b. A consistent trend is noted for the relationship between the DR and the porosity in Figure 3, where it can be seen that the higher the porosity, the higher the DR. Therefore, results in Figures 2 and 3 show a clear relation between the porosity, directly related to w/b, and the DR index.

Figure 1: Results for O35, O40, O45 and O50 concretes: (a) loss on drying; (b) sorptivity.

Table 2: Linear correlation coefficients with the square-root of time.

<table>
<thead>
<tr>
<th>Series</th>
<th>DR r</th>
<th>Sorptivity r</th>
<th>DR r</th>
<th>Sorptivity r</th>
<th>DR r</th>
<th>Sorptivity r</th>
</tr>
</thead>
<tbody>
<tr>
<td>O35</td>
<td>0.97</td>
<td>0.95</td>
<td>O40b</td>
<td>0.98</td>
<td>0.97</td>
<td>0.99</td>
</tr>
<tr>
<td>O40</td>
<td>0.95</td>
<td>0.98</td>
<td>C40</td>
<td>0.99</td>
<td>0.99</td>
<td>0.97</td>
</tr>
<tr>
<td>O45</td>
<td>0.98</td>
<td>0.98</td>
<td>O60</td>
<td>0.98</td>
<td>0.99</td>
<td>0.92</td>
</tr>
<tr>
<td>O50</td>
<td>0.99</td>
<td>0.99</td>
<td>C60</td>
<td>0.97</td>
<td>0.98</td>
<td></td>
</tr>
</tbody>
</table>

Figure 2: DR vs. compressive strength at 28d.  
Figure 3: DR vs. porosity.
Figure 4 shows the relationship between the DR and the saturated resistivity. The resistivity is a transport index that describes the porosity of concrete, the higher the resistivity the lower the porosity. Resistivity is a quality index of concrete (15) and, hence, the DR is closely related to it.

The relation between the DR and the sorptivity is shown in Figure 5. The correlation indicates a sound relation between both parameters. It should be noted, however, that the found relation is not linear. One point to take into account is that, as previously mentioned, in the sorptivity test there is water ingress through one concrete surface and water egress (although minimum) through the other. This egress is negligible as regards the change in weight of the sample. On the other hand, oven-drying produces simultaneous water loss through two concrete surfaces at a similar rate. It can also be seen that as sorptivity increases, DR also increases but at a lower relative rate. It seems that sorptivity is more sensitive than the rest of properties to a certain parameter. The three mixes that behaved differently from the group are C45, O60 and C60, i.e., the three mixes with the higher capillarity (C60 and O60 for having the highest w/b, and C45 for having being produced with a BPC that led to lower performance of concrete than the OPC). This could indicate that a given rise in the sorptivity does not necessarily imply the consequent relative change in other properties of hardened concrete. This same distinction has already been noticed for another transport property such as chloride diffusion in (7). In this sense, it must be taken into account that concrete mixtures evaluated in this study contained low paste content, because of the use of a water-reducing admixture. Considering this, it should be noted that if the volume of paste was higher the relation between the two parameters could be different. This is a matter for future research.

3 CONCLUSIONS

Moisture transport in concrete is a complex mechanism that needs to be better understood in relation with concrete durability. Then, the drying rate is considered a valid alternative to indirectly describe concrete durability. A good relation between the DR and the sorptivity has been found.

Drying rate as a transport index has two main advantages: it requires a short test time in comparison with other tests, such as the sorptivity test; and it is consistently linked to other transport indexes, such as resistivity, porosity and sorptivity. This representativeness of the drying test method is supported by the close relation between the drying process and the pore connectivity of concrete.
The results presented in this work show good correlation between drying rate and compressive strength, porosity, saturated resistivity and sorptivity. Thus, given the simplicity of this test, the drying index is presented as a convenient transport index.

4 REFERENCES

DEVELOPMENT OF A METHODOLOGICAL PROCEDURE FOR THE DURABILITY EVALUATION OF EXISTING EXTERIOR FINISHINGS

Silvia Erba\(^{(1)}\), Bruno Daniotti\(^{(2)}\) and Elisabetta Rosina\(^{(2)}\)

(1) ABC Department, Politecnico di Milano, Milano - silvia.erba@polimi.it
(2) ABC Department, Politecnico di Milano, Milano - bruno.daniotti@polimi.it; elisabetta.rosina@polimi.it

Abstract
The aim of the research is the development of a methodology to improve the precision of the degradation survey phase, assessing durability and environmental impacts in the context of sustainable constructions. The method concentrates on the evaluation of the residual service life of external finishing systems correlating the level of damage (carried out through NDT and GIS application), to residual performances. The research of a methodological formulation, to be applied on historical and new (existing) buildings, has the aim to elaborate strategies to increase the service life of the components minimizing the environmental impacts inside the building process. The study promotes maintenance planning or preventive preservation plans, in order to reduce the risk factors.

The procedure has a direct experimental application within the research project “Città Studi: Sustainable Campus”, intended to transform the university area in Milan into an exemplar campus with respect to life quality and environmental sustainability. The paper reports the application of the investigation procedures on some buildings inside Leonardo Campus. On the base of the results, the researchers are performing different lab tests for the evaluation of the adsorption properties and durability of mortars, in view of a validation of the behaviour obtained through software simulation.

Keywords: durability, diagnostic, mortars, sustainability, IR Thermography, planned conservation
1 INTRODUCTION

A precise evaluation of the present state of degradation of buildings components, is fundamental to define the residual service life and therefore to study the possibilities of improving the actual performances and to limit the degradation growth. Within the standards framework, the ISO 15686 – Buildings and constructed assets. Service life planning – defines service life as the period of time after installation during which a building or its parts meets or exceeds the performances requirements upper or equal to the accepted limits [1] [2]. Dealing with existing buildings, the researchers have to take into account the residual service life, defined as the service life remaining after a certain moment of consideration. To assess residual service life of an inspected building or component is important to know its history, i.e. data on the original performance values, information on the installation, maintenance, trend of deterioration, etc., which can involve several difficulties to be obtained. During the process of assessment of the residual service life of the existing building/component, the researchers have to ensure that the performance levels will be maintained equal or higher to accepted limits, taking into account sustainability aspects and maintenance plans [3]. In the case of historical buildings, life-cycle assessment offers a holistic method for comparing the costs of renovation against maintenance, showing the economic advantages of conserving existing finishing, and supports also the planned conservation strategy [4].

Nevertheless the assessment phase is underestimated and often brings to errors of identification and, therefore, inappropriate approaches of intervention. The aim of the research is to propose a valid methodology for exterior finishes analysis, based on the use of ND techniques in order to improve the correctness of the results and promote more effective and targeted interventions of maintenance or planned conservation. By changing the boundary conditions and reducing the risk factors or by flanking and reinforcing the fragile elements is possible to slow down the degradation growth and elongate the permanence of the element guaranteeing a proper level of performance. The methodology proposed is based on the study and comparison of the results obtained through three different approaches: visual inspections, non-destructive surveys (IR thermography and hammering) and laboratory tests (emissivity and heat conduction tests). Buildings inside Campus Leonardo of Politecnico di Milano served as study cases within the project “Città Studi, Campus Sostenibile”. The research project has the aim to revitalize and transform the university area Città Studi into an exemplar campus with respect to life quality and environmental sustainability, involving the scientists employed at Politecnico and at State University of Milan on the study, analysis, survey, assessment, management, repair, enhancement of the university’s campus placed in the eastern area of Milano. The façades of the buildings under study have two different types of finishing (cladding system with ceramic tiles or plaster) and at present, after 50 years of usage, have many degradations of materials and systems, such as detachments, missing tiles, discoloration, exfoliations due to the action of atmospheric agents and the loss of adherence between the support and the tile, or among the layers of the support, and the absence of a correct maintenance [5] [6]. Studying the results obtained through the application of the mentioned investigation procedure, the researchers focused on the main cause of degradation: the water absorption in the mortar layer. Therefore they have addressed the following step of the research to the characterization of the components with regard to water absorption, by laboratory tests and software simulation. The paper describes the two main phases of the research, the obtained results, and the possible developments regarding future interventions.

2.1 Inspections, diagnostic and laboratory tests: the methodological approach for the definition of the state of degradation

The methodological procedure applied comes from the use of three different approaches to assess the state of degradation of components belonging to the external façades of existing buildings. To have more precise results, the researchers flanked the most common visual inspections with non-destructive surveys, especially IR Thermography, and validated the results by hammering. In addition, they performed laboratory tests for the evaluation of the heat transmission and emissivity [7] [8]. They carried out the thermographic survey at transient conditions, and developed the thermal analysis using alternative methodologies, based on the use of ArcGIS. After the identification of the reference temperatures and the characteristic gradient through IRT Analyzer, they optimized the phase of defects mapping through ArcGIS, which improves the geometric localization on the façades. The use of this innovative technology allowed to obtain more accurate results avoiding manual and punctual analysis of single areas [9]. They acquired thermal images after heating the façade by solar irradiation for 2 hours. This time window allows to obtain the best contrast between delaminated areas and adherent ones, considering the factors that play a prominent role in the heat diffusion in a component, such as its thickness and thermal characteristics, the supposed depth of the defect, the environmental conditions (temperature of the air, wind speed, humidity), and the season (consequently the inclination of solar rays) [10]. In the final step the researchers have validated the results obtained with the thermographic survey through hammering tests and a precise comparison with photos of the façades, in order to avoid misidentifications. The procedure provides plausible results with a probability of real presence of the pathology that can reach 80%. The reliability of the diagnosis does not reach 100% because at the state of the art there are still unknown aspects due to uncertainties regarding heat transfer methods and boundary conditions. The researchers have defined three levels of probability: high (80%), medium (50%) and low (30%) because different aspects (change of thermal and optical characteristics, such as discolorations or the reflectance of the surface, and the orientation) can influence the results and therefore decrease the reliability of the results.

2.2 The results: anomalies and their probable causes classification

The phase of assessment of the degradation state of the façades has provided information about the pathologies detected and their possible causes [11]. The façades with external cladding system made of ceramic tiles present detachments, missing elements, superficial alteration, crazing, stains and graffiti, superficial deposits, joints degradations, cracking, crusts, splitting and biological patina. The façades with a plaster finishing show chromatic alteration, efflorescence, stains and graffiti, superficial alteration, superficial deposits, crusts, exfoliations, erosion, disgregation, detachments, cracking and blowings. The anomalies have been classified according to the associated gravity (possible danger and therefore urgency of the repair) [12]: the most important damage tallied is the detachment, since it represents a danger for people (in particular in a public building as campus universities) and it implies the necessity of a timely intervention to guarantee safety [13]. The detachment is expressed in
terms of lack of adhesion and the corresponding degradation level is linked to the entity, the probability of correct diagnosis and the superficial extension (localized or diffuse) of the pathology. A large extension is connected to the possibility of fractures which can imply the collapse of the element: this is associated to a high degradation level. These three level of damage have been defined in order to quickly and intuitively localize the areas more affected by the pathology and therefore to plan possible interventions to flank and reinforce the fragile elements. From the relationship between degradation and possible causes, the researchers agreed that in the majority of cases the critical point is given by the water infiltration that causes loss of adherence. Consequently the research has continued testing the absorption properties of different kinds of mortars, both for plaster and for the embedment of ceramic tiles, to obtain more precise results for the materials characterization. They will use the outcomes as input data to simulate the heat and moisture transfer in a modeled wall considering a specific climatic context.

3 SECOND PHASE OF THE RESEARCH: EXPERIMENTAL METHODOLOGY FOR THE CHARACTERIZATION OF MORTARS

3.1 The description of the mortars types

Figure 1: Composition of the mortars samples
The researchers chose and tested six different plaster lime mortars (n°1-6 in fig.1) and two cement mortars (n°7-8 in fig.1) for embedding. Table 1 reports the composition of the mortars. They evaluated accurately the water content in the mixture to obtain the correct porosity of the material and an homogeneous degree of workability of the mixture.

Table 1: Types of mortars

<table>
<thead>
<tr>
<th></th>
<th>Binder:</th>
<th>Aggregate:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>aerial lime putty</td>
<td>pozzolan, fine sand and sieve sand 0,4</td>
</tr>
<tr>
<td>2</td>
<td>aerial lime putty</td>
<td>cocciopesto, fine sand and sieved sand 0,4</td>
</tr>
<tr>
<td>3</td>
<td>natural hydraulic lime NHL 3,5</td>
<td>sieved sand 0,4 and natural coarse sand</td>
</tr>
<tr>
<td>4</td>
<td>natural hydraulic lime NHL 3,5</td>
<td>natural coarse sand</td>
</tr>
<tr>
<td>5</td>
<td>natural hydraulic lime NHL 3,5</td>
<td>cocciopesto, sieved sand 0,4 and brick fragments</td>
</tr>
<tr>
<td>6</td>
<td>1 part of putty and 0,3 parts of NHL 3,5</td>
<td>coarse fragments of Zandobbio limestone, “spolverino” (main dolomia of Carenno)</td>
</tr>
<tr>
<td>7</td>
<td>CEM I OPC Portland cement</td>
<td>Ticino sand</td>
</tr>
<tr>
<td>8</td>
<td>CEM 4 Pozzolanic cement with low amount of pozzolan</td>
<td>Ticino sand</td>
</tr>
</tbody>
</table>

3.2 Test to determine the hygroscopic sorption properties

The researchers executed the adsorption test in the climatic chamber, based on the UNI EN ISO 12571, for the evaluation of the hygrothermal behaviour of building materials and products [14]. They evaluated the properties of hygroscopic adsorption, that regulate the transfer of water vapour between air and a porous material up to reach the equilibrium state, according to the method described in the standard. For each material they realized 3 samples with dimensions 5x5x2 cm, and mass around 100 g each. This test is based on the principle that if a porous material is placed in an environment, characterized by a certain relative humidity (RH) and temperature (T) after a while it will reach a state of equilibrium with the humidity of the ambient air, exchanging water [15] [16]. Whilst maintaining a constant temperature, in the specific case fixed to 20°C, the 24 specimens have been placed consecutively in a series of test environments, inside the climatic chamber, characterized by relative humidity increasing in stages, respectively 30%, 50%, 70%, 90%. The moisture content is determined when equilibrium with each environment is reached. Equilibrium with the environment is established by weighing the specimens until constant mass is reached. After establishing the moisture content at each relative humidity, the adsorption curve for the different specimens has been drawn. In the following the graph of the adsorption curve is reported for all the mortar types as mean value between the 3 samples analyzed. It establishes
a relationship between the moisture content mass by volume at equilibrium with the environment and the relative humidity of the ambient air, at the specified temperature of 20°C. Experimentally the researchers couldn’t obtain the sorption curve values at high relative humidities on the base of the adsorption test in the climatic chamber and the curves stop at 90% RH. The missing values will be completed in the following part of the research, through total absorption tests in saline solutions.

![Graph showing relative humidity-moisture content mass by volume](image)

Figure 2: Relative humidity-moisture content mass by volume

From the graph it’s possible to notice that the behavior of all the mortars is similar and undergoes a raising in correspondence of the 50% R.H. It is also clearly visible the difference between the lime mortars and the cement ones: the first ones present a moisture content (mass by volume) at 90% around 20 kg/m\(^3\), the second ones around 65 kg/m\(^3\).

The specific results will be carefully analyzed and used as input data for the assessment of moisture transfer in a wall modeling, considering a specific climatic context. The researchers are collecting the necessary materials data set through different laboratory tests on the base of the UNI EN 15026 [16].

4 CONCLUSIONS

− The described research presents a valid methodology for assessing the degradation of components belonging to the external envelope of existing buildings, especially detachments and delaminations of the external layers.
− The analysis has been developed through the performance limits method, which connects the estimated service life of component to its performances. As an example, in the specific case of the cladding system, the performance requirement is expressed in
terms of the adhesion level of the cladding system and therefore the performance decay (and consequently the degradation growth) is evaluated in terms of tiles detachments.

- The research aims to formulate interventions of maintenance or planned conservation, with the aim to intervene by changing the boundary conditions and therefore removing the risk factors or by flanking and reinforcing the fragile element (e.g. possible waterproof protective coatings for the mortar [17]) so that to slow down the degradation growth and elongate the permanence of the element guaranteeing a proper level of performance.

- Nowadays tests are in progress for the determination of water absorption of the mortar samples with/without water-repellent treatments by capillarity, water vapour transmission, and hygroscopic adsorption (points of the curve over 90%) [18] [19] [20] [21]. Durability tests (accelerated tests) on the mortars samples with/without protective coatings will follow [22] [23].

- The next step of the research will be based on the use of the results obtained in laboratory for the development of time based simulations through the use of software, such as WUFI for the determination of moisture and heat transfer, and for the evaluation of the degradation probability.

ACKNOWLEDGEMENTS

Thanks to Dr. Antonio Sansonetti (ICVBC, CNR Milano Bicocca) for the collaboration and consultancy to the experimental phase.

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STOCHASTIC, DETERMINISTIC, STATISTICAL AND ARTIFICIAL INTELLIGENCE BASED MODELS TO PREDICT THE SERVICE LIFE OF RENDERED FACADES

Ana Silva (1), Pedro L. Gaspar (2) and Jorge de Brito (1)

(1) Instituto Superior Técnico, Civil Engineering, Architecture and Georresources Department, Lisbon - anasilva931@msn.com; jb@civil.ist.utl.pt
(2) Faculty of Architecture - University of Lisbon, Lisbon - pmsgaspar@fa.utl.pt

Abstract

Service life prediction has a primary role in today’s context, allowing a more rational use of construction elements, reducing the costs associated with rehabilitation procedures. In the literature, the most common methods used to estimate the service life of buildings and its components can be classified as deterministic, probabilistic and engineering (a symbiosis of the previous two). In this study, the application of deterministic (graphical method), stochastic (logistic regression and Markov chains), statistic (multiple linear and non-linear regression techniques) and artificial intelligence based models (artificial neural networks) is proposed to predict the service life of rendered facades. Rendered facades are one of the most common types of claddings applied in Portugal. However, the predominance of this type of coating is related with the low investment applied in this solution, which often implies an unacceptable degradation of the building heritage. A comparative study is performed to analysis the applicability of each method proposed. This analysis relates the ease of application with the effectiveness of the proposed models. The results obtained through the different methods applied in this study are coherent from an empirical point of view. Furthermore, the results obtained are consistent with other studies performed in this field of knowledge.

Keywords: Service life prediction; deterministic; stochastic; statistic; artificial intelligence based models; rendered facades.

1 INTRODUCTION

The durability of constructions is essential to the quality of urban spaces. Presently, the degradation of buildings and their components has become a complex problem from the economic, cultural and environmental points of view. The increasing scarcity of funding for the maintenance and rehabilitation of infrastructures requires a more rational approach to decision-making, in terms of inspection, maintenance and rehabilitation (Paulo et al., 2013).
For this, an efficient evaluation of the service life must be considered, taking into account the properties of materials, the environmental conditions of exposure, the workmanship, the service conditions and the maintenance planning (ISO 15686-1:2000).

Service life [SL] is generally estimated based on knowledge on the material and its deterioration state, using as indicators given measurable properties. According to various authors (Hovde, 2004), (Lacasse & Sjöström, 2004) there are three basic methods of service life prediction: the deterministic methods, the probabilistic methods and the engineering methods. The first are based on the study of the degradation factors that affect the elements under analysis, on the understanding of the mechanisms involved, and on their quantification translated into degradation functions. Probabilistic methods, usually based on matrices or probabilistic calculus, define the likelihood of a change of the state of an element occurring with the objective of overcoming the uncertainty related to the degradation evolution and the unpredictability of the in-service conditions. Finally, engineering methods are often based on dose/response functions that model the performance of building materials for a given set of degradation agents.

2 METHODOLOGY

In order to compare the results obtained for the estimated service life of rendered facades through each of the three basic methods, a sample of 100 rendered facades was chosen. The sample analyzed is composed of 100 cases studies located in Lisbon, whose degradation state had been previously determined through in situ visual inspections. The results were obtained through the application of a numerical index that expresses the global degradation of the coatings analyzed. This index is obtained as the ratio between the extent of the façade degradation - weighted according to the degradation level and the severity of defects - and a reference area, equivalent to the maximum theoretical extent of the degradation for the facade in question (Gaspar and de Brito, 2011):

\[ S_w = \frac{\sum (A_n \times k_n \times k_{a,n})}{A \times k} \tag{1} \]

where \( S_w \) is the weighted severity of degradation of the facade (%); \( A_n \) is the area of coating affected by a defect \( n \); \( k_n \) is the multiplying factor for defect \( n \), as a function of its condition (between 0 and 4); \( k_{a,n} \) is the weighting coefficient corresponding to the relative importance of each defect \( (k_{a,n} \in \mathbb{R}^+) \) (based on the cost of repair of defects); \( k \) is the weighting factor equal to the highest degradation level in the façade (4, in the case of renderings); \( A \) is the total area of the cladding. The degradation condition of rendered facades was thus characterized and classified according to a range of discrete levels from A (no visible degradation) to E (widespread deterioration, requiring an immediate corrective action), Table 1. These data - along with the building, environmental, use and maintenance characteristics - was then fed into each of the three methodologies and the results obtained were compared.

3 DETERMINISTIC MODELS

Deterministic models are usually straightforward mathematical operations or matrices, obtained through empirical evidence (ageing tests, real life assessment, laboratorial testing), easy to apply and to be understood by all actors in the building cluster. Not surprisingly, these methods are those which have produced the most practical results serving as the basis for the international standard for the durability of construction (ISO 15686:2000). However, most deterministic methods tend to regard SL as an absolute value, independent of the degradation
processes (or the transitions from a degradation condition to the next one), therefore neglecting all the variability associated with them. In the present study, overall degradation of the facade was quantified using a graphical procedure in which a function was adjusted to the scatter of points of the sample (average degradation curve), whose abscissa measure the variable “age” and whose ordinates measure the variable “severity”. In theory, by establishing the degradation curves of a given construction element it is possible to predict the end of its service life, having defined a maximum acceptable degradation level. In this study the average degradation curve for the 100 case studies was obtained through simple regression by adjusting a 2nd order polynomial line to the cloud of points obtained from the field work (Figure 1). This degradation path corresponds to the occurrence of physical and chemical phenomena whose action is felt slowly at first but whose degradation potential grows with time, i.e. the greater the degradation level the higher the probability it will increase, and at a faster pace (Shohet et al., 1999).

Table 1. Proposed classification of degradation condition of renderings

<table>
<thead>
<tr>
<th>Condition level</th>
<th>Example</th>
<th>Physical and visual assessment</th>
<th>Severity of degradation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level A</td>
<td><img src="example1.png" alt="Example" /></td>
<td>Complete mortar surface with no deterioration. Surface even and uniform. No visible cracking or cracking ≤ 0.1 mm. Uniform colour and no dirt. No detachment of elements.</td>
<td>&lt; 1%</td>
</tr>
<tr>
<td>Level B</td>
<td><img src="example2.png" alt="Example" /></td>
<td>Non-uniform mortar surface with likelihood of hollow localized areas determined by percussion, but no signs of detachment. Small cracking (0.25 mm to 1.0 mm) in localized areas. Changed in the general colour of the surface. Eventual presence of microorganisms.</td>
<td>1 to 5%</td>
</tr>
<tr>
<td>Level C</td>
<td><img src="example3.png" alt="Example" /></td>
<td>Localized detachments or perforations of the mortar. Hollow sound when tapped. Detachments only in the socle. Easily visible cracking (1.0 mm to 2.0 mm). Dark patches of damp and dirt, often with microorganisms and algae.</td>
<td>5 to 15%</td>
</tr>
<tr>
<td>Level D</td>
<td><img src="example4.png" alt="Example" /></td>
<td>Incomplete mortar surface due to detachments and falling of mortar patches. Wide or extensive cracking (≥ 2 mm). Very dark patches with probable presence of algae.</td>
<td>15 to 30%</td>
</tr>
<tr>
<td>Level E</td>
<td><img src="example5.png" alt="Example" /></td>
<td>Incomplete mortar surface due to detachments and falling of mortar patches. Wide or extensive cracking (≥ 2 mm). Very dark patches with probable presence of algae.</td>
<td>&gt; 30%</td>
</tr>
</tbody>
</table>

Figure 1. Development of rendered facades degradation over time (average degradation curve)

The analysis of the square of the Pearson product correlation coefficient ($R^2$) reveals that the regression curve has a relatively high value ($R^2 = 0.876$). In this case 87.6% of the variability of the degradation (dependent variable) is explained by the model, i.e. 89% of the variability of $y$ (degradation) is explained by $x$ (age of the painting) and 12.4% is due to other factors. Assuming that level D corresponds to the end of service life of rendered facades, it is possible to determine graphically their reference service life, through the intersection of the degradation curve with the horizontal line that represents the minimum performance level. The value obtained is 18 years.
4 STOCHASTIC MODELS

Due to the high dependence on time and the uncertainty associated with the performance of buildings, it is often necessary to use stochastic models to predict the SL of building components. Within these methods, Markov chains can be used to emulate the evolution of the degradation state of constructions, defining the probability of a future state based only on the present condition, independently of previous deterioration history (Neves et al., 2006), modelling the probability of transitions between given states (Moricous and Lounis, 2005). For the sample studied, Figure 2 shows the mean longevity in each degradation state, $T_i$. The results show that transitions between condition states of facades occur faster in less deteriorated facades (changes from level A to level B occur in only 2.5 years). Renders remain for a longer period of time in higher degradation levels (condition C and D). The transition between levels associated with higher degradation states imply the presence of a larger number of defects and/or more hazardous defects, including simultaneous occurrence of defects and synergies between them.

![Figure 2. Mean longevity in each degradation state (Markov chains)](image1)

Figure 2 illustrate the probabilistic distribution of the degradation condition over time. As expected, the probability of renders being in level A decreases rapidly in time, becoming lower than 2% at year 10. The probability of renders being in level B initially increases and reaches a peak at around year 3 (probability of 43.5%) and then steadily decreases as the probability of higher levels of degradation increases. Probability for levels C and D displays skewed distribution curves, with rapid increases and peaks at year 10 ($P = 51.4\%$) and year 19 ($P = 38.5\%$) for levels C and D respectively. Finally, the probability of level E steadily increases with age as expected, i.e. it is practically nil before year 9, and above 75% after year 38.

![Figure 3. Probabilistic distribution of the degradation of renders according to the age (using Markov chains)](image2)

The mean relative error obtained for the estimated number of cases belonging to each degradation condition is relatively low: the mean relative error for all states is 7.55% and all results are lower than 16%. Taking into account all the variability associated with the degradation phenomena it is considered that the model is suitable and able to correctly classify the cases analysed. The overall sample reaches the maximum probability (38.5%) of belonging to level D (which marks the end of service life) at year 19. The probability of the renderings being in Level D or E (i.e. unacceptable) reaches 50% at year 14.
Logistic regression is another stochastic method that allows establishing an empirical relation between variables, thus producing a probabilistic analysis of the degradation condition of rendered façades, as a function of age. As expected, the probability of a render to belong to level A decreases over time and is practically nil at year 5. The younger coatings have a greater probability of belonging to the lower degradation condition classes. The probability of level B increases initially reaching a peak at around 5 years (probability of 43%) and then decreasing. As for level C the maximum probability (74.2%) is reached at year 10. For level D the peak is reached at around year 19 (probability of 78.3%). Finally, the probability of level E increases with age and after year 30 it is higher than 70%. After year 20 the probability of levels A, B and C of degradation is practically nil. The time intervals when the probability of belonging to a given level is similar to that of the next level are also the moments where the probability of transition from one level of degradation to the next is greatest. As time goes by, the probability of the rendered façades reaching the end of their SL increases. After year 17 the probability is higher than 50% and after year 21 it is higher than 90%. Therefore and unless there is some intervention, there is then an increasing probability that rendered façades are significantly degraded after reaching a certain stage of degradation.

5 STATISTIC MODELS

Regression analysis is one of the statistical techniques used most often to study the influence of a dependent variable, in comparison with other independent variables, which are responsible for the performance of the dependent variable. The main objective of this type of analysis is to explain a given reality and try to anticipate the role of a dependent variable as a function of the independent variables. Using this method, it was concluded that the severity of degradation depends on the age of the facades, the orientation of facades, on render type, the exposure to damp, the facade protection level and the level of protection in the balconies. In expression (2) is presented the mathematical equation which expresses the severity of degradation in function of the six explanatory variables. These six variables are able to explain 90.5% of the variability of the degradation and the remaining 9.5% can be explained by factors extrinsic to the analysis. The average estimated SL thus obtained is 15.7 years, with a standard deviation of 3.02 years.

\[ S_w = 0.013 \cdot A - 0.166 \cdot O - 0.088 \cdot B - 0.2 \cdot R - 0.109 \cdot D - 0.152 \cdot P + 0.757 \]  \hspace{1cm} (2)

where \( S_w \) is the weighted severity of degradation of the facade (%); A the age of the rendered facades; O the facades orientation; B the level of protection in the balconies; R the render type; D the exposure to damp; P the facade protection level.

Simple and multiple linear regressions are the most known and most commonly functions used in SL prediction. However, in most cases, the degradation phenomena, whether they are physical, chemical or biological, can be better represented by other functions. In this study various non-linear models to estimate the degradation of rendered facades were analysed. Table 2 shows the results obtained by the various functions used.

Table 2. Estimated service life by different non-linear models (regression analysis)

<table>
<thead>
<tr>
<th>Estimated service life</th>
<th>Polynomial model</th>
<th>Gompertz curve</th>
<th>von Bertalanffy curve</th>
<th>Richards curve</th>
<th>Weibull curve</th>
<th>Brody curve</th>
<th>Exponential model</th>
<th>Potential model</th>
</tr>
</thead>
<tbody>
<tr>
<td>( R^2 )</td>
<td>0.904</td>
<td>0.872</td>
<td>0.872</td>
<td>0.872</td>
<td>0.902</td>
<td>0.905</td>
<td>0.875</td>
<td>0.900</td>
</tr>
</tbody>
</table>
6 ARTIFICIAL INTELLIGENCE BASED MODELS

Artificial neural networks (ANNs) have been successfully used to solve complex problems in various application fields (Silva et al., 2013). The commonest type of ANN is the multilayer perceptron (MLP). An MLP is a network with three or more layers of neurons: an input layer, one or more intermediate (hidden) layers and an output layer. In this study, MLPs were used to develop a model to estimate the degradation severity of rendered facades. In the model obtained, the degradation severity was a function of the render type, the exposure to damp, the facade protection level and the render age, as seen in expressions (3) and (4). Coefficients $h_0$ to $h_4$ and $c_{i0}$ to $c_{i11}$ are presented in Table 3.

$$S_i = h_0 + \sum_{i=1}^{4} h_i H_i$$

(3)

$$H_i = \tanh \left( c_{i0} + \sum_{n=1}^{11} c_{in} V_n \right)$$

(4)

Where $V_1$ represents the age of the render, $V_2$ marble agglomerate, $V_3$ monomass, $V_4$ cement and lime mortar, $V_5$ cementitious mortar, $V_6$ a favourable exposure, $V_7$ an unfavourable exposure, $V_8$ a normal exposure, $V_9$ a poor facade protection, $V_{10}$ a good facade protection and $V_{11}$ an average facade protection.

Table 3. Coefficients for the ANN-based model

<table>
<thead>
<tr>
<th>$i$</th>
<th>$h_i$</th>
<th>$c_{i0}$</th>
<th>$c_{i1}$</th>
<th>$c_{i2}$</th>
<th>$c_{i3}$</th>
<th>$c_{i4}$</th>
<th>$c_{i5}$</th>
<th>$c_{i6}$</th>
<th>$c_{i7}$</th>
<th>$c_{i8}$</th>
<th>$c_{i9}$</th>
<th>$c_{i10}$</th>
<th>$c_{i11}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>5.52E-1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>-1.63E-3</td>
<td>6.59E-1</td>
<td>6.82E-3</td>
<td>-1.45E-2</td>
<td>-3.66E-1</td>
<td>5.27E-1</td>
<td>-4.39E-1</td>
<td>-4.68E-1</td>
<td>7.27E-1</td>
<td>-5.81E-1</td>
<td>-2.95E-1</td>
<td>-7.40E-1</td>
<td>-2.01E-1</td>
</tr>
<tr>
<td>2</td>
<td>2.45E-1</td>
<td>-6.857</td>
<td>6.03E-2</td>
<td>2.119</td>
<td>1.695</td>
<td>2.129</td>
<td>-1.065</td>
<td>-1.183</td>
<td>1.730</td>
<td>1.219</td>
<td>6.10E-1</td>
<td>-4.12E-2</td>
<td>1.188</td>
</tr>
<tr>
<td>3</td>
<td>1.31E-1</td>
<td>-2.096</td>
<td>2.59E-2</td>
<td>1.167E-1</td>
<td>-3.69E-3</td>
<td>5.26E-1</td>
<td>-8.96E-2</td>
<td>6.88E-1</td>
<td>1.05E-2</td>
<td>3.33E-1</td>
<td>4.10E-2</td>
<td>1.138</td>
<td>5.28E-1</td>
</tr>
<tr>
<td>4</td>
<td>-2.68E-1</td>
<td>1.012</td>
<td>-5.56E-2</td>
<td>4.30E-3</td>
<td>2.145E-1</td>
<td>2.58E-2</td>
<td>1.54E-1</td>
<td>6.40E-2</td>
<td>-3.28E-1</td>
<td>-1.45E-1</td>
<td>-4.41E-1</td>
<td>4.58E-1</td>
<td>3.639E-2</td>
</tr>
</tbody>
</table>

The average estimated SL obtained using ANN based methods is 17.5 years, with a standard deviation of 2.74 years.

7 RESULTS AND DISCUSSION

The deterministic model (graphical method) is a simple and expedient method. This method is easy to apply and gives information on the performance loss over time. However, it only provides an absolute value of the estimated service life of rendered facades.

Stochastic models consider the rate of transition between degradation states to predict the behaviour of the rendered facades and estimate the time after which they will be unable to meet the performance requirements for which they were designed. With this methods it is possible to: i) predict the probability of occurrence of each of various degradation conditions in each case study and define an equivalent probabilistic model; ii) evaluate the probability of transition from each condition level (i.e. degradation state) to the next one; and iii) translate the data collected in the field work into statistical distributions allowing probabilistic analyses of the degradation phenomena.

Statistic methods (multiple linear and non-linear regressions), albeit more complex, allow evaluating the influence of the different variables considered as factors for degradation severity, thus leading to a ‘wider scope’ analysis.

Through artificial neural networks based methods empirical knowledge can be acquired from a learning data set relative to a given problem. These methods are thus capable of
learning and generalizing based on experience and examples, an ability that is extremely important since it means that complex problems can be solved, which are difficult to solve either analytically or numerically. However, these methods are usually highly complex. In fact one does not intuitively grasp how a network functions; additionally, they use thousands of synaptic weights that are not subject to a logical/intuitive interpretation.

Besides the prediction ability of each method, the ease with which each one can be used was also assessed. Neural networks are the most complex, but lead to the lowest errors. Statistic methods are relatively simple and lead to good results. Deterministic models are the most simple but the less accurate. Stochastic models give a probabilistic analysis of the degradation phenomena and are relatively simple to apply.

The size of the sample is paramount when establishing SL prediction models. In fact the more complex the behaviour in terms of the reality one intends to model, the more data are needed. The main disadvantage of all the methods studied is that they react to data modification, so that their ability to generalize increases, in principle, with the size of available data. All the methods can be easily complemented with more data over time.

Finally, it is found that the results for the models proposed are logical from a physical point of view, and match reality found in the field work. Furthermore these results agree with those of studies performed by another authors. Shohet and Paciuk (2004) took two distinct levels of requisites: for the most stringent one the service life of rendered facades was estimated as 15 years (with a range between 12 and 19 years); for a lower level of requirement the service life rose to 23 years (with a range of 19-27 years). Figure 5 shows the results obtained using the various methods proposed. The mean estimated service life obtained is 18.5 years (with a range of 15.7-22 years); this value is indicated with a line in Figure 4. These values are coherent with the values in the literature. The graphic method, the simplest, gives an estimated service life of 18 years, relatively close to the mean value from the various methods. Therefore, it seems reasonable to accept that this method may be applied in current applications, as the planning of maintenance actions, leaving the more complex methods as a reference for more detailed investigations about the service life of rendered facades.

![Figure 4](image-url)

**Figure 4. Results obtained using the different methods proposed**

8 CONCLUSIONS

This study intended to provide a comparison of distinct SL prediction models. The models proposed to estimate the service life of rendered facades are useful tools and are relatively simple that balance cost and speed, enabling its practical application to buildings. The study of service life prediction contributes to a more rational management of the maintenance of
buildings. The models proposed can be employed in various scopes of service life prediction and maintenance of constructions and are therefore capable of providing indications relative to a complex phenomenon such as cladding degradation, giving some information on the synergy between the degradation agents and the way they influence the degradation levels. Furthermore, the stochastic models make it possible to evaluate the probability of transition from one condition level (i.e. degradation state) to the next. The methodology tested in this study on rendered facades may be applied to other facade coatings or other construction elements.

All the models proposed were found to be valid and to adjust to the reality they are intended to model. However, their main disadvantage is that they react to data changes. In fact the quality of the models is directly related to the amount of data available. Future studies may lead to improvements of the accuracy of the models proposed, with the acquisition of new and more abundant data. Furthermore, as new data are added new variables may be included in the models if they are found to be better at explaining the degradation of this type of coating. A greater complexity of the service life prediction models does not always lead to better results. In most cases, highly complex models produce a decrease in the errors obtained between the predicted and the observed service lives of rendered facades. However, errors obtained by the most complex model (ANN) are relatively close to the errors obtained with more simple models (multiple regression analysis). Often it seems more reasonable to sacrifice the accuracy of the model in exchange for greater applicability and simplicity.

ACKNOWLEDGEMENTS

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REFERENCES


INFLUENCE OF LIMESTONE POWDER ON THE SETTING AND HYDRATION OF CEMENT PASTE IN THE FRESH STATE

Yury A. Villagrán-Zaccardi (1,2), Darío D. Falcone (1,3) and Damián E. Benito (1)

(1) LEMIT, La Plata, Argentina, yuryvillagran@conicet.gov.ar
(2) CONICET, CCT-La Plata, La Plata, Argentina
(3) CICPBA, La Plata, Argentina

Abstract
Limestone powder influences the hydration of Portland cement. Limestone particles act mainly as nucleation sites for the precipitation of hydration products, and they thus contribute to the development of the process.

In practical terms, the progress of cement hydration during the fresh state is evaluated by the setting times. Initial and final setting times are commonly associated with specific stages of the hydration process. In this sense, it may be important to note that the process of hydration can differ from static to dynamic conditions. A continuous removal of hydration products from the surface of the hydrating particles contributes to the acceleration of the process by increasing the exposure of anhydrous surfaces. Then, hydration during continuous agitation in a concrete mixer may differ from hydration during the setting test.

In this paper, influences of the limestone powder content in fresh cement paste are evaluated. Mixes were tested for setting times and hydration degree at the first hours of hydration. Results from static and dynamic conditions are presented. From the results, the effect of limestone powder on the hydration process is analyzed, with particular consideration of the differences between static and dynamic conditions in which hydration is allowed.

1 INTRODUCTION
Limestone powder contributes to the development of cement hydration mainly increasing the nucleation sites for the precipitation of hydration products. However limestone cannot be considered an inert addition, as it leads to the formation of some calcium carboalbite hydrate and modifies AFm and AFt phases [1-2], the reaction of limestone has minor effects on strength and pore connectivity. Then, its main action is in the fresh state and early ages.

However, limestone powder also indirectly affects durability at later ages by means of the effect it may show on the fresh state. For instance, limestone cements, despite the higher fineness they may have, generally demand less water than pure cements [3]. This is mainly
attributed to the different particle size distribution of the fines, which range is widened with the inclusion of limestone powder in comparison with pure cements. Then, concrete made with limestone cements may show less bleeding than concrete with pure cements for the same consistency level [4-5]. Bleeding is relevant for structures that may suffer physical abrasion, where it must be limited, and structures that may suffer plastic cracking, where it helps to prevents drying at the surface.

Concrete placing and compacting is another issue in concrete construction regarding durability. The time open for these tasks is determinant for the results that the utilization of adequately proportioned concrete may have in practice. This is, properly proportioned concrete may lead to inadequate performance if enough time for its application on-site is not allowed.

In this sense, the effect that limestone may show on the fresh state, and specifically on setting and hydration, is relevant for durability of reinforced concrete structures, with particular concern for those constructions that require long transport periods of fresh concrete.

In practical terms, the progress of cement hydration during the fresh state is evaluated by the setting times. However, setting defined by the Vicat needle does not correspond to particular changes in structure or forces within the fresh cement paste [6]. The evolution of the structure of the paste occurs during the first minutes after the end of mixing, when it spontaneously evolves within a few minutes to a structured state, with the formation of a single aggregate from all particles in the mix. Then, it is important to keep in mind that setting times of cement paste are technological parameters that have been defined by convention. This is, setting times cannot be linked to any particular change in the paste due to the evolution of hydration. Setting is understood in terms of coalescing C-S-H coated cement particles [7], and it is therefore mainly due to physical mechanisms that are not related with specific chemical changes. To link setting and demeanour changes in the fresh mix, alternative parameters to the penetration of the Vicat needle have been proposed, such as yield stress [8] or conductivity [9]. However, these techniques are still being developed, and their practical application to commercial cements is not possible.

It is important to note that the process of hydration can differ from static to dynamic conditions. Contacts between particles resulting from particle interactions are considered preferential sites for C-S-H nucleation [10], and their increase in number shortens the induction period. Continuous mixing prevents to some extent these contacts, and it may therefore affect the induction period. A continuous removal of hydration products from the surface of the hydrating particles contributes to the acceleration of the process by increasing the exposure of anhydrous surfaces. Hydration during continuous agitation in a concrete mixer may differ from hydration during the setting test. Limestone powder, on the other hand, provides nucleation sites, and it contributes to shorten the induction period.

Some studies presented results that supports the idea that limestone may decrease setting times [11-12], whereas results from others show little to null influence of limestone on setting [3]. In this regard, the fineness of limestone powder is a key factor [13], and the influence of limestone filler, coarse addition usually added into self-compacting concrete, is usually low, whereas limestone-blended cements (where limestone particles are usually finer than clinker particles) show differences from non-blended cement as regards setting.

In this paper, influences of the limestone powder content in fresh cement paste are assessed. Mixes were tested for setting times and hydration degree at the first hours of hydration. Results from static and dynamic conditions are presented. From the results, the
effect of limestone powder on the hydration process is analyzed, with particular consideration of the differences between static and dynamic conditions in which hydration is allowed.

2 MATERIALS AND METHODS

Cement pastes were prepared using Ordinary Portland Cement and variable amounts of limestone powder, which properties are presented in Table 1. Pastes with 0, 10, 20 and 30% w/w of cement replaced by limestone were studied. For each blend, water demand for standard consistency was determined.

Standard setting times were determined in accordance with IRAM 1619:2002 [14]. The procedure in this standard is similar to that in ASTM C 191:2008, with a difference in the determination of the initial setting time, which is considered when the needle penetrates up to 4±1mm form the bottom plate. In addition, pastes were prepared with the same proportions for 0 and 20% w/w of replacement, and the mixing was prolonged. During this prolonged mixing, the mixing batch was covered with a wet cloth to prevent water loss by evaporation. Samples for reference, and after 0.5, 1 and 1.5h of mixing, were obtained and tested for setting times and hydration. Hydration was assessed by loss on ignition and heat of hydration.

For the loss on ignition determinations, free water was removed by solvent exchange. Fresh samples were mixed and rinsed three times with acetone. Then, triplicate samples were put in filter papers into crucibles, and put in oven at 105°C. Then, the initial dry weights were noted. Finally, these samples were calcined at 950°C and weighted. Non-evaporable water contents were computed from the difference between the loss on ignition of the paste and deducing the loss on ignition of anhydrous cement and limestone.

For the measurements of the released heat of hydration, about 200g of fresh sample of each paste was put in a semi-adiabatic calorimeter, and the temperature raise was monitored during the first 24h.

Table 1: Properties of cement and limestone powder.

<table>
<thead>
<tr>
<th>Propiedades</th>
<th>OPC</th>
<th>Limestone</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blaine specific surface (m²/kg)</td>
<td>303</td>
<td>556</td>
</tr>
<tr>
<td>Retained on 75μm sieve (% w/w)</td>
<td>3.90</td>
<td>1.60</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
<td>3.13</td>
<td>2.75</td>
</tr>
<tr>
<td>Cementing index (%)</td>
<td>-</td>
<td>0.74</td>
</tr>
<tr>
<td>Compressive strength 2d / 28d (MPa)</td>
<td>19.2 / 40.0</td>
<td>-</td>
</tr>
<tr>
<td>Chemical analysis (% w/w)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>2.33</td>
<td>36.01</td>
</tr>
<tr>
<td>Insoluble residue</td>
<td>0.55</td>
<td>6.65</td>
</tr>
<tr>
<td>SO₃</td>
<td>1.47</td>
<td>0.21</td>
</tr>
<tr>
<td>MgO</td>
<td>2.03</td>
<td>0.76</td>
</tr>
<tr>
<td>SiO₂</td>
<td>19.9</td>
<td>11.58</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>4.02</td>
<td>0.68</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>2.95</td>
<td>1.82</td>
</tr>
<tr>
<td>CaO</td>
<td>66.6</td>
<td>45.82</td>
</tr>
</tbody>
</table>
3 RESULTS AND DISCUSSION

Setting times for the different replacement percentages of cement by limestone are presented in Figure 1. It can be noted that limestone powder shortened setting times in accordance with the percentage of replacement. For these pastes, water demand was 0.27, 0.255, 0.25 and 0.25 w/w for 0, 10, 20 and 30% of limestone content, respectively. The reduction of the water demand is explained by the lower hydraulicity of the blend derived from the dilution effect by the replacement of cement by limestone.

In Figure 2, setting times for the different mixing times for pastes with 0 and 20% of limestone content are presented. In increasing order, the obtained setting times were those for 0.5, 1, 1.5 and 0h of mixing time. Initial setting occurred approximately 2h after the end of mixing in the 0% paste, no matter it was mixed for 1 or 1.5h (initial setting times: 3:05 and 3:50 for 1 and 1.5h, respectively), and after 1.5h in the 20% paste (2:34 and 3:00 for 1 and 1.5h, respectively). This time spans were of approximately 2.5 (3:05) and 2h (2:25) for 0 and 20% pastes, respectively. Then, setting times are shortened after 0.5 and 1h of mixing, but this decrease is compensated for 1.5h of mixing, and similar setting time to those of the reference mix were obtained for the longest mixing time. Then, at least two opposite mechanisms are inferred. First, continuous mixing would have allowed faster hydration as a consequence of anhydrous particles more exposed to water due to the cleaning caused by dynamic friction. The effect of this mechanism on setting decreased with time and showed its maximum for 0.5h of mixing time. Second, the measurement of setting times by Vicat needle penetration is a static method that requires the development of physical forces among particles. This development would have been prevented during mixing, but would have been rapidly caused when mixing stopped. The rate of this development seems not fast enough to avoid a net delaying after prolonged mixing for 0.5 and 1h. However, even if hydration was further accelerated by mixing beyond 0.5h, this acceleration was not fully reflected in the setting times due to the lack of the sufficient development of physical forces among particles, and moreover, it was fully compensated for 1.5h of mixing time.

Figure 1: Vicat setting times for different replacement percentages of cement by limestone.
Nevertheless, it should be noted that final setting times were shortened for 1.5h of mixing in comparison with reference setting times, for both 0 and 20% pastes. This difference is still lower than those for 0.5 and 1h of mixing. In consequence, the span between initial and final setting times decreased with prolonged mixing time, from 1:40 and 1:00 to 1:00 and 0:30 for 0 and 20% pastes, respectively, regardless they were mixed for 0.5, 1 or 1.5h. This supports the hypothesis of two competing mechanisms, and implies that the acceleration of hydration due to prolonged mixing would not be able to shorten setting below the time required for the development physical bounding among particles.

From the comparison between 0 and 20% pastes with prolonged mixing times in Figure 2, it seems that limestone not only shortens setting times as shown in Figure 1, but also helps the development of physical forces among particles at static conditions before the end of mixing for all the mixing times tested.

Figures 3 and 4 show the rate of heat evolution of paste samples in the semi-adiabatic calorimeter. These results include not the full amount of heat of hydration for the samples that were mixed for 0.5, 1 and 1.5h, as part of the heat of hydration was released during the prolonged mixing. In spite of that, the aim of this evaluation was to compare the rate of heat evolution of the pastes, and this is feasible with the presented data.
In correlation with results from the setting tests, the peak of heat occurs earlier in 20% pastes in comparison with 0% pastes. Again, it is demonstrated how limestone powder contributes to accelerate hydration.

Moreover, the dilution effect of limestone can be noted when comparing the values for the lower maximum peaks in 20% pastes (Figure 4) in comparison with 0% pastes (Figure 3). This agrees with studies of similar raw materials from other researchers [15]. The replacement of cement by limestone decreases the hydraulicity and thermal power of the blend, and the heat of hydration decreases.

The results of heat of hydration also support the findings of earlier setting times for the pastes mixed for 0.5 and 1h, followed by pastes with 1.5h of prolonged mixing, and reference pastes. Again, it seems that converse mechanisms act during prolonged mixing. Pastes mixed for 0.5 and 1h showed earlier hydration than reference pastes, which agrees with findings in [16]. But progressive mixing did not increase this difference in pastes mixed for 1.5h. The structure of the fresh paste is not reflected by the heat of hydration, and it is likely that a different reason than that argued for the ambiguity in setting times can be argued.

The action of at least two mutually competing mechanisms can be again suggested for the different times for the maximum peak of heat for pastes mixed for 0, 0.5, 1 and 1.5h. The mechanism in favour of hydration would be the cleaning of hydration products from the surface of anhydrous particles and the absence of a diffusive layer that controls the hydration rate. In opposition, the increase in the amount of hydration products would proportionally increase the binding forces among hydrating particles. The mixing energy would be sufficient to initially break this binding, but, this breaking would require increasing energy as hydration progresses. Then, it seems that with 0.5 and 1h of prolonged mixing, aggregation of particles is prevented by the applied mixing energy, but with increased mixing time, this aggregation may have been favoured by the higher amount of hydration products, and hydration would be comparatively retarded due to the presence of the diffusive layer in aggregates of particles. Further research on this issue would help to clarify these concepts.

Finally, the contents of non-evaporable water of the pastes are presented in Table 2. Here, losses on ignition from cement and limestone have been deducted from the loss on ignition of the paste. It is again noted an increase in the hydration degree with the limestone content.
must be noted that the non-evaporable water content referred to the hydraulic material (cement) are even higher for pastes with limestone, as the presented values are referred to the sample weight of paste.

Finally, synergy between limestone and prolonged mixing is noted. It can be derived that mixes containing limestone are more sensitive to prolonged mixing than non-blended mixes, as demonstrated by the results of setting times, heat of hydration and non-evaporable water contents.

Table 2: Non-evaporable water contents with dynamic hydration (g/100g).

<table>
<thead>
<tr>
<th>Limestone content</th>
<th>Mixing time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.0</td>
</tr>
<tr>
<td>0%</td>
<td>0.12</td>
</tr>
<tr>
<td>20%</td>
<td>0.33</td>
</tr>
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</table>

4 CONCLUSIONS

Setting and hydration of cement pastes with and without limestone powder, and with and without prolonged mixing have been evaluated. Setting times, heat of hydration and non-evaporable water contents have been determined. From the results, the following conclusions are drawn:

- Limestone powder accelerates hydration, and this is intensified with prolonged mixing time.
- Opposite effects of prolonged mixing are derived. It is presumed that the cleaning of anhydrous surfaces produced by continuous mixing competes with another mechanism that caused lower hydration rate when pastes were mixed for 1.5h in comparison with pastes mixed for 0.5 and 1h. The nature of this mechanism is not totally clear at this stage. More research is required in this sense.
- Mixes containing limestone may show shorter working times than non-blended mixes. This could be more critical if long transport distance or mixing time is required. Then, some difficulties during placing may arise, with a logical negative impact on the performance of hardened concrete as regards strength and durability.

REFERENCES


IN-SITU MEASUREMENTS OF MOISTURE CONTENT AND TEMPERATURE IN WOODEN CONSTRUCTIONS

Karin Sandberg(1), Simon Dahlquist(1), Anna Pousette(1)

(1) SP Technical Research Institute of Sweden, Wood Technology, Skellefteå – karin.sandberg@sp.se, simon.dahlquist@sp.se, anna.pousette@sp.se.

Abstract
Service-life assessment and life-cycle cost are becoming increasingly important in investment in and planning of construction work. The objective of the present work was to show results on moisture conditions in glued laminated timber (glulam) beams of pine (Pinus sylvestris L.) and spruce (Picea abies (L). Karst.) and glulam columns of spruce to demonstrate differences that can be ascribed to different coating types and colours and the influence of the varying positional orientation (north/south), up or down at columns and to measurement depth.

To be able to measure a large amount of data, a wireless system was chosen. For every point, temperature and moisture content were registered together with climate data. To keep track of the measurements, they were registered in a database. The glulam beams were 315 mm high, 140 mm, 215 mm or 90 mm wide and 2 m long. The columns had 100- x 100-mm cross-sections. The specimens were placed in Bygdsiljum, near Skellefteå, in the northeast of Sweden and in Borås in the southwest of Sweden.

1 INTRODUCTION

Long life and low life-cycle costs are increasingly in demand in planning and construction. The ability of a structure to resist degradation by weather exposure is therefore essential for any outdoor structures. The durability of wooden structures depends on the wood material, impregnation, surface treatment, moisture content, temperature, mechanical impact and maintenance [1]. Extending the life of existing structures can be economical, but only if it is more profitable to repair and maintain than to demolish and rebuild. It is not always easy to determine the extent of decay, the rate of degradation and the remaining life for various structures and environments [2], [3].

Today there are a number of on-going projects to develop models for service-life prediction on timber structures. Developments in this area have led to different approaches for modelling service life, such as Scheffer Index [4] and the factor method [5] for predicting the service life of components and structures.
Dose-response models [6], [7] were developed to deal with the long exposure time in field trials.

The degradation of wood is a complex problem dependent on many variables, such as water, oxygen, temperature, digestible substrate, PH range and chemical growth factors (nitrogen compounds, vitamins, etc. [8]. The major risk factor for timber exposed to weather is water, since water serves a variety of functions in the degradation process. Water also affects dimensional change through shrinking and swelling, which in turn influence crack formation. Most of all, water is a prerequisite for decay and sap stain fungi, since they need free water (above fibre saturation point) in the wood cells in order to grow.

The transport of water in wood can be divided into the two main categories of hygroscopic and capillary water transport. Hygroscopic transport is by diffusion. When the relative humidity is close to 100%, the cell walls of wood are saturated with moisture, a condition called the fibre saturation point (FSP). In this state, a moisture content of 28%–30% is generally assumed [9].

1.1 Objectives

The objective of the present work was to show results on moisture conditions in glued laminated timber (glulam) beams of Scots pine (*Pinus sylvestris*) and Norway spruce (*Picea abies*) and in glulam columns of spruce to demonstrate differences that can be ascribed to different coating types and colours and the influence of the varying positional orientation (north/south), up or down at columns and to measurement depth.

WoodBuild was the name of a Swedish research programme (2008–2012) with the goal of developing engineering tools based on models that can be used in the planning and design of timber structures with regard to improved service life. The purpose of the full-scale test was to verify models for prediction of wetting periods on wood surfaces and of moisture conditions dependent on climate loads, finishes, wood processing and design.

2 MATERIAL AND METHODS

2.1 Field test

The 2-m-long glulam beams and columns of impregnated pine (*Pinus sylvestris*) and spruce (*Picea abies*) were placed on the testing ground in Bygdsiljum (Lat. 64°20′57″N, Long. 20°30′14″E) in the county of Skellefteå in northeast Sweden in August–September 2007 and are part of a larger test described in [10]. A testing ground with beams and columns in the county of Borås was established in 2008 [10].

For continuous measurements of moisture content (MC), relative humidity (RH) and temperature (measured every hour), beams and columns were chosen from the field and the measurement started in January 2009. The columns were fastened to a concrete foundation. Climate, temperature and humidity at the test site were measured with a weather station at the test field.

Continuous measurements of MC, RH and T were performed at the middle of the glulam beams on the south side, north side and underneath and on the top (two meters’ height) and bottom of columns on the south side. Top surfaces and beam ends were covered with sheet metal. The measurements were made at the two depths: 5 mm and 15 mm below the surface. The wireless Hygrotrac system is described in Sandberg *et al. 2011* [11].
The beams and the columns were documented with tomography through the cross-section and scanning of the side surfaces before they were placed on the test site.

**Glulam Beams**

Table 1 shows the characteristics of the glulam beams. The surface treatments were: Oil (raw linseed oil with aliphatic naphtha as thinner). Paint system 1: priming oil and two layers of water-based, alkyd oil-acrylic latex hybrid opaque glazing paint. All surfaces were treated with water-based priming oil, biocide Class 3, before painting. Paint system 2: priming oil + primer + two layers of topcoat. All surfaces were treated with solvent-based priming oil, biocide Class 3, before painting. The primer was solvent-based, semi gloss modified alkyd oil. The painting was done at the glulam manufacturer to a dry-film thickness of 60 µm.

Table 1: Characteristics of glulam beams 2 meters long. MB type of beams; number of the beam group 1–7; name of the beam H Bygdsiljum and B in the name indicates Borås, level 1, level 2 and level 3 above the ground.

<table>
<thead>
<tr>
<th>Name/type</th>
<th>Material/dimensions (width x height) [mm]</th>
<th>Surface treatment/Thinner</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>MB1H59-1</td>
<td>Pine, pressure treated/140 x 315</td>
<td>Oil/solvent based</td>
<td>-</td>
</tr>
<tr>
<td>MB2H63-1</td>
<td>Pine, pressure treated/140 x 315</td>
<td>Paint system 1/water based</td>
<td>White</td>
</tr>
<tr>
<td>MB3H69-1</td>
<td>Pine, pressure treated/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4H73-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4H74-2</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4H75-3</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4H76-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 2/ solvent based</td>
<td>Red</td>
</tr>
<tr>
<td>MB6H81-1</td>
<td>Spruce/90 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB7H86-1</td>
<td>Spruce/215 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4B06-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4B07-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4B08-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4B09-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
<tr>
<td>MB4B10-1</td>
<td>Spruce/140 x 315</td>
<td>Paint system 1/ water based</td>
<td>Red</td>
</tr>
</tbody>
</table>

**Columns**

The columns were painted with the same system 1 that was used for the beams (see Table 2).

Table 2: Columns height two 2 meter from group S7, from test field Bygdsiljum (H) and Borås (B).

<table>
<thead>
<tr>
<th>Name/ type</th>
<th>Number</th>
<th>Wood/ Dimension (mm2)</th>
<th>Surface treatment</th>
<th>Colour</th>
</tr>
</thead>
<tbody>
<tr>
<td>S7H51-H55</td>
<td>5</td>
<td>Solid spruce / 100 x 100</td>
<td>Paint system 1</td>
<td>Red</td>
</tr>
<tr>
<td>S7B6-B10</td>
<td>5</td>
<td>Solid spruce / 100 x 100</td>
<td>Paint system 1</td>
<td>Red</td>
</tr>
<tr>
<td>S7B1-B5</td>
<td>5</td>
<td>Solid spruce / 100 x 100</td>
<td>Paint system 1</td>
<td>White</td>
</tr>
</tbody>
</table>
3 RESULT AND DISCUSSION

The measurements were made at two depths: 5 mm and 15 mm below the surface. Continuous measurements of MC, RH and T were performed in the middle of the glulam beams, on the north and south surfaces and on the underside and on the top and bottom on the south sides of the columns. Measurements were performed from 2009 until 2012.

The solar radiation on the surface has an effect on temperature and through that on the MC measurements. Therefore, data used for comparisons were measurements made at night from 01:00 to 03:00 to avoid influence from the sun.

Figure 1 shows an example of graphs of MC and temperature for beam H76 during outdoor exposure for four years. The climate conditions varied during the years. Autumn 2008 and summer-autumn 2012 were rainy. The environment for the equipment in Bygdålum was harsh, with temperatures between -35°C and +35°C and with both rain and snow.

There were periods of missing data on all specimens due to various causes. For example, a thunderstorm caused an interruption in the supply of electricity. During two winters, the logger stopped working when it was colder than -25°C. There was also another kind of missing data where sensors had quit working and gave intermittent data as result. In Borås there was a considerable amount of missing data due to malfunctions in the wireless Internet connection. Some sensors and batteries were replaced during the test period. MC below 8% can be considered incorrect measurements.

The purpose of the full-scale test is to verify models for prediction of wetting periods on wood surfaces and of moisture conditions dependent on climate loads.
Over time, the MC measurements showed fluctuations that can be difficult to explain. For example, there were very high MC values in December in a winter climate and temperatures below minus. It was found that for a couple of days, the temperature was above the freezing point, and probably the sun melted the snow or frost on the surface for a few hours during those days. After that, the weather turned cold again. Some sensors registered MC above 26% during a long period when temperature was around or below zero. Unexpectedly high MC was found during this period. A possible reason might be measuring failure due to condensation or snow on the device.

Above fibre saturation point (FSP), there are values that showed some dissolution, but the algorithm that calculates the MC is not calibrated for high MC.

MC measurements at the underside of a beam can be stable around 20% (see Figure 2) but can increase to 30% or more in autumn (see Figure 3).

![Figure 2: MC measured at 15 mm depth at underside of beam H63 (white) in Bygdsiljum](image)

![Figure 3: MC measured at underside of beam B6 in Borås.](image)

A crack that appears close to a sensor can affect the measurements. The crack size differs due to the surrounding weather, and whether or not water penetrates into a crack depends on the climate, wind and surface treatment [12]. If the crack breaks the paint coating, the paint gives limited protection and water can more easily enter into the wood. There are different kinds of cracks in glulam beams (see Figure 4). They can be long and extend along almost the whole beam. Large (wide) cracks may follow the glue line (not delamination) [13]. There may be very tiny cracks or a few close to the end grain.
The oiled H59 beam has a lot of cracks. The oiled surface gives limited protection and the surface gets wet and dry very fast and the large movements in the surface increase the amount of cracks. Cracks occurred around measured point that may have influenced the measurements (see Figure 4 and Figure 5).

Figure 4: Photo of beam H59 north side on the top and south side below and H86 red beam.

Figure 5: MC variations in the impregnated and oil-treated H59 beam measured at 5 mm depth on the north side follows climate changes rather quickly. Below, MC measured at underside of beam H59.
In glulam beams, there will be cracks that influence the measurements in several ways. Firstly, cracks influence measurement correctness if they occur close to a measurement point. Secondly, a crack can break through the protective coating on the wood. Thirdly, a crack can be an entrance point for water to get into the wood.

There were differences in the amounts of cracks between test groups. The impregnated beams and the oiled beams in particular, had more MC fluctuations compared to painted surfaces.

In general, MC depends on the depth of measurement. The MC variation is less at 15 mm depth than at 5 mm but cracks might change that relation. Precipitation of resin around resin pocket occurs after some time and might also influence the measurements.

Risk for decay is greatest when the MC is above FSP and depends on temperature and the wetting time. If wood dries fast, there is a shorter wetting time and thus a shorter time with optimal conditions for fungi to grow. In what way water penetrates into wood and dries out is a complex problem and differs between heartwood and sapwood [14], [15], [16].

In a painted facade, the highest MC seldom reaches above 20%, and investigations show that facades treated in this way have acceptably long service life [17] if the design is correct. Facades are less exposed and more sheltered and have fewer cracks than glulam beams in this test.

4 CONCLUSION

In general, MC differences can be ascribed to different coating types and colours and to the influence of the varying positional orientations (north/south), up or down at columns and measurement depths but also number of cracks and position.

The purpose of the full-scale test is to verify models for prediction of wetting periods on wood surfaces and of moisture conditions dependent on climate loads, finishes, wood processing and design. Large variations in MC between beam types and over the years generate complex MC models.

ACKNOWLEDGEMENTS

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REFERENCES


EXPERIMENTAL EVALUATION OF PERFORMANCES OF DIFFERENT SOLUTIONS OF FINISHING FOR MASONRY BUILDINGS FOR DETERMINING DURABILITY

Maurizio Nicolella(1), Fabio Iucolano(2)

(1) Department of Civil, Construction and Environmental Engineering, University of Naples Federico II, Naples – maurizio.nicolella@unina.it
(2) Department of Chemical, Materials and Industrial Engineering, University of Naples Federico II, Naples – iucolano@unina.it

Abstract
The components of the building envelopes have to provide performances of different types: it is supposed to resist to the weather agents, to have a good water vapor permeability in order to prevent some degradation phenomena, and to inhibit the capillary rising dampness. But it must also maintain all these features over the time, in other words it must have a high durability. The designers, when they have to choose finishing solutions, are put in front of very complex evaluations, with the aim of finding the most appropriate one, given the specific context stress factors.

To provide a useful guide for those frequent situations, two Departments of University of Naples Federico II conducted combined tests in the laboratories and on the field, on four types of finishing solutions (plaster + painting) for tuff masonry, which is typical of Southern Italy. In the DICMAPI laboratory the specimens were subjected to tests of water absorption by capillarity, tests of water vapor permeability, tests of water absorption at a low pressure, and tests of accelerated aging in a QUV machine.

The field assessments were carried out on 500 sampled buildings in the city of Naples, over 10 years of monitoring, showing the different behavior of the four types of selected specimens: for example, the characteristics of the finishing solutions of macroporous plaster + silicate paint and cement plaster + quartz paint have remained almost the same, without significant performance decay. The results could represent a good starting point for the creation of a useful handbook for designers of recovery interventions.

Keywords: durability, performances, laboratory test, masonry, plasters, paints.

1. INTRODUCTION
The most part of materials and building components provide multiple performances: for example, finishing materials used for perimetral walls are mainly characterized by esthetical
needs, but also by necessities of protection towards the inside and, according to the latest laws about energy saving, often by needs of thermal insulation. Considering this multiplicity of performances they have to carry out, their context of stress is pretty various as well: sun, wind, dampness from different sources (rain, capillary rising dampness, condensation), and also mechanical stress, such as thermal variations, and phenomena of contraction.

Designers often find it hard to choose the products that suit the most a specific case, since the performances the components are needed for, may require totally opposite characteristics. In addition to this, the producers’ data sheets usually refer to the reactions of the single components themselves, rather than to their behavior when they are used in a “system” of materials, for example wall + plaster + finishing + paint.

So, while it is easy to get information about the water vapor permeability of a painting product, it is much more difficult to get it for an ensemble made up by tuff + plaster + skimming mortar + paint. And there is also the necessity to gather information about the combined action of rain and wind, and the effects of UV rays, dampness condensation, etc.

Then, it seemed particularly useful to carry out an experimental research on the ensemble of a building envelope, that requires the evaluation of reciprocal influences that should be carefully examined for the analysis of the behavior of the whole system, rather than on the behavior of a single component used in the external façade, and on the stress context that can be predicted for it. Then, it ultimately seemed appropriate to inspect on the permanence of the characteristic performances during the time, through durability tests carried out with two different approaches:

- tests on the field, monitoring the real behavior of the components, during ten years, when used in buildings with finishing solutions that will be further explained;
- tests of accelerated aging in the laboratory with a QUV machine.

2. CRITERIA OF CHOICE OF SPECIMENS AND STRESS AGENTS

Choosing the typologies of specimens to use for the tests, it was decided to select the finishing solutions that are most used in the designing procedure of the recovering interventions, for buildings with tuff masonry, in the specific context of the city of Naples. For this reason, we availed ourselves of the collaboration with the society S.I.Re.Na. – created by the City of Naples in order to promote the recovery of the historical center – that has collected of a database of more than 1200 interventions realized in the decade 2002-2012. According to the statistical data, it resulted that the most used solutions were by far:

a. plaster made up with lime mortar + lime paint (hereinafter called “B”);

b. plaster made up with “common” mortar (lime + plaster) + siloxane paint (“Gs”);

In order to complete the extrapolation of the range of possibilities, two more solutions were chosen. These two appear to have opposite problems in relation to dampness:

c. plaster made up with cement mortar + quartz paint (“Gc”);

d. macroporous plaster + breathable paint (“G”).

The specimens were made up in the following modes:

- dimensions of yellow tuff specimens: 5 x 5 cm x (5+1) cm - 16,0 x 4,0 x 2,0 +/- 0,5 cm. only for the test in the QUV machine;
- finishing of the sides: plaster +paint on one side, bituminous liquid sheath on the other (fig. 1a).
Among the different performances that characterize the finishing of a tuff masonry, the following were mainly considered: (i) the faculty to resist decay because of the action of phenomena of capillary rising damp, (ii) the faculty of perspiration, (iii) the faculty to resist the action of rain, sun and condensation, (iv) the reaction of the material to these stress agents during the time.

So the following tests were prepared for the four different types of specimens: capillary water absorption, water vapor permeability, water absorption under low pressure, accelerated aging in a QUV machine, in addition to the monitoring on the field, executed on about 500 buildings with the aim of evaluating the in-use behavior for the four solutions.

3. EXPERIMENTAL

3.1 Capillary water absorption

The amount of absorbed water was measured according to the European Standard EN 15801 which specifies a method for determining the water absorption by capillarity of porous inorganic materials. The method may be applied to porous inorganic materials either untreated or subjected to any treatment or ageing. Three specimens for every typology, parallelepiped shaped, were dried in an oven at 40°C until to obtain a constant mass. Subsequently, the specimens were placed on a multilayer of 5 mm of filter paper, soaked in deionized water, maintaining the water level constant during the test (see figure 1b). Starting from the placement on the multilayer, the samples were weighed at fixed times and the amount of absorbed water \( A \) was calculated using the equation: 

\[
A = \left( \frac{m_i - m_0}{S} \right) \times 1000,
\]

where \( m_i \) is the specimen weight (g) at time \( t_i \), \( \sqrt{s} \), \( m_0 \) is the dry specimen weight (g) and \( S \) is the specimen surface (cm\(^2\)) lying on the wet multilayer.

The capillary absorption coefficient (CA, expressed in mg·cm\(^{-2}\)·s\(^{-1/2}\)) was also calculated at the end of the test for all the samples, using the equation: 

\[
CA = \left( \frac{A_{30} - A_0}{\sqrt{t_{30}}} \right)^{1/2},
\]

where \( A_{30} \) is the amount of water absorbed by the specimen per area unit after 30 min, \( A_0 \) is the intercept of the straight line obtained in the linear graph with the y-axis and \( \sqrt{t_{30}} \) is the square root of the time (s) after 30 minutes.

3.2 Water vapor permeability

The permeability to water vapor is defined as the amount of water vapor flowing in the time unit and in the surface unit through a porous sample. It is expressed as the ratio between the weight variation of the whole system in 24 h and the area of the sample surface, under a constant difference of the vapor pressure of water (expressed in Kg/m\(^2\)·s·Pa at 20°C), according to the European Standard EN 1015-19. The permeability test was carried out on three samples for each series, placed in Plexiglas vessels containing a saturated solution of potassium nitrate (KNO\(_3\)), capable of maintaining, at \( T = 20 \) °C, a constant relative humidity \( H_R \), equal to 93.2% (see figure 1c). Then the vessels were sealed and placed in a climatic chamber at \( T = 20 \) °C and \( H_R = 50\% \). The steam flow through the samples was then evaluated by measuring, at time intervals of 24 h, the mass variation of the containers. The test may be considered completed when, by plotting the values of the mass as a function of time, three points are arranged on a straight line, that is, when the mass changes are constants for at least three successive weightings. The data so obtained allowed to calculate, in accordance with the European Standard mentioned above, the water vapor permeability (WVP).
3.3 Water absorption under low pressure (pipe method)

This test is useful to simulate the effect of water absorbed for rain and/or for moisture condensation on different types of finishing. The measurement of the absorbed water allows to evaluate surface modifications, to characterize the effect of a treatment of impregnation altering the surface permeability or to characterize the effect of a natural weathering. The water absorption was measured with the pipe method according to the RILEM II.4 standard and using the apparatus illustrated in figure 1d. The test is performed by applying a water column on the material under examination and measuring every 5 minutes the volume of water absorbed (directly by reading on the graduated glass tube), for a total time of 60 minutes. Based on the data collected during the test, it is possible to calculate the degree of absorption of water at low pressure (WLP), obtained from the ratio between the difference of the water volume (ml) absorbed at the final time ($Q_{tf}$) and at 5 minutes ($Q_{t5}$), and the surface area (cm$^2$) of contact of the cell (S), as follows: $WLP = (Q_{tf} - Q_{t5})/S$.

![Figure 1](image)

Figure 1 - (a) four typologies of manufactured specimens; (b) capillary water absorption of a specimens placed on a wet multilayer; (c) scheme of apparatus for water vapor permeability test: (1) sample, (2) test vessel rectangular shaped, (3) putty, (4) air interspace ($\approx$ 10 mm), (5) saturated solution of potassium nitrate (KNO$_3$); (d) apparatus of water absorbed at low pressures (dimensions in cm).

3.4 Accelerated aging in a QUV machine

The enrollment of the test and the elaboration of the results were carried out taking account of the following standard: UNI 9922, UNI EN ISO 4628-1, UNI EN ISO 4618-2, UNI EN ISO 4628-1, using a machine with fluorescent QUV lamps. The QUV accelerated weathering tester reproduces the damage caused by sunlight, rain and dew. In a few days, the QUV tester can reproduce the damage that occurs over years outdoors. Differently from xenon arc test
chambers, it doesn’t reproduce the whole sun radiation, but only its damaging effects with a wavelength between 300 nm and 400 nm. It is based on the principle that the long-lasting materials are mainly affected by UV rays with short waves when used outdoors, and this is the primary cause of their atmospheric aging. To simulate outdoor weathering, the QUV tester exposes materials to alternating cycles of UV light and moisture at controlled, elevated temperatures. It simulates the effects of sunlight using special fluorescent UV lamps, and simulates dew and rain with condensing humidity and/or water spray. Before subjecting the specimens to the accelerated aging test, a chromatic characterization was carried out, realizing a number of color measurements between 5 and 7, thanks to the use of a spectrophotometer, which were then elaborated with the software Spectramatic NX. The testing cycle (see figure 2) was repeated for 32 times, exposing the specimens to 128 hours of radiation and 128 of condensation, for a total of 256 of exposition, and it was structured in a pattern described in tab.1. At the end of the testing cycles, the evaluation of the decay of the surface was realized using three tables, according to the UNI EN ISO 4628-1 standard. It classifies the number and dimension of defects found, and the intensity of variations, as shown in tab 2. Concerning the intensity of the chromatic variation, it was evaluated by comparing (using the spectrophotometer) the colorimetric surveys \((L^*, u^*, v^*)\) of the stressed specimens with those of the unstressed ones. The color difference, in fact, can be calculated using the Euclidean distance of the \((L^*, u^*, v^*)\) co-ordinates. In colorimetry, the CIE 1976 \((L^*, u^*, v^*)\) color space, commonly known as CIELUV, is a color space adopted by the International Commission on Illumination (CIE) in 1976, as a simple-to-compute transformation of the 1931 CIE XYZ color space, but which attempted to a greater perceptual uniformity.

Table 1 - Chart of the accelerated aging cycle in a QUV machine

<table>
<thead>
<tr>
<th>SUB-CYCLES</th>
<th>DURATION</th>
<th>TEMPERATURE</th>
<th>SUNLIGHT RADIATION</th>
<th>CONDENSATION</th>
</tr>
</thead>
<tbody>
<tr>
<td>SUNLIGHT RADIATION</td>
<td>4 hrs.</td>
<td>60°C</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>CONDENSATION</td>
<td>4 hrs.</td>
<td>50°C</td>
<td>No</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Table 2 - Chart of classification to indicate the defects and to measure the variations

<table>
<thead>
<tr>
<th>Class.</th>
<th>Number of defects</th>
<th>Dimension of defects</th>
<th>Intensity of variations</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>none, that is to say no detectable</td>
<td>not visible at a 10x zoom</td>
<td>unchanged, no detectable variations</td>
</tr>
<tr>
<td>1</td>
<td>very few, a small, scarcely significant number of defects</td>
<td>only visible at a 10x zoom</td>
<td>very slight, a barely detectable variation</td>
</tr>
<tr>
<td>2</td>
<td>few, a small but significant number</td>
<td>barely visible at common sight</td>
<td>slight, a clearly detectable variation</td>
</tr>
<tr>
<td>3</td>
<td>moderate number of defects</td>
<td>clearly visible at common sight (to 0.5 mm)</td>
<td>moderate, a variation that can be detected very clearly</td>
</tr>
<tr>
<td>4</td>
<td>considerable number of defects</td>
<td>from 0.5 mm to 5 mm</td>
<td>considerable, a determined variation</td>
</tr>
<tr>
<td>5</td>
<td>dense disposition of defects</td>
<td>bigger than 5 mm</td>
<td>greatly marked variation</td>
</tr>
</tbody>
</table>

Figure 2 - The specimens subjected to accelerated aging in a QUV machine.
4. RESULTS AND DISCUSSION

Figure 3 shows a comparison between the 4 types of capillary absorption curves (each curve represents an average value of three experimental tests carried out for each type of specimen), together with a table which reports the average values of capillary absorption coefficients CA, calculated taking into account the linear portion of the curves. The trend of the curves shows that in a first period (about 30-40 min) water absorption is substantially constant, while varying significantly when the rising water begins to affect the finishing layers, characterized by porosity (and hence absorption of water) very different from each other. Figure 4 reports a comparison between the 4 typologies of water vapor permeability curves (each curve represents an average value of three experimental tests carried out for each type of specimen), together with a table in which are reported the average values of the water vapor permeability (W_{VP}) and the resistance to the steam diffusion (\(\mu\)), together with some porosimetric characteristics of the four different finishing (porosity and average pore diameter). The trend of these curves shows that there are two different behaviors, closely related to the different slopes of the lines: “Gc” and “Gs” specimens exhibit a lower slope than “B” and “G” specimens, meaning that they present a lower W_{VP} and therefore a greater diffusion resistance \(\mu\). These results are strongly correlated to the porosimetric data. In fact “Gc” and “Gs” specimens are manufactured with cement-based plasters, very compact, with as much compact paintings, while “B” and “G” are both manufactured with plaster and paintings characterized by higher porosity. Finally, figure 5 shows the curves related to the water absorption at low pressures (each curve represents an average value of three experimental tests carried out for each type of specimen), together with a table which summarizes the values of the W_{LP}. The behavior of these curves is substantially determined by the type of painting, more or less impermeable to water. Among the paints used in the present work, the lime paint (specimens “B”) is definitely the most permeable to water, and this leads to W_{LP} values about 4 times higher than other materials. On the contrary, the small differences between the other three samples are related to the different substrates. It is interesting to note that, among the solutions evaluated in the present work, the typology “G” shows the higher water vapor permeability (\(\mu = 6.40\)) coupled with a low permeability to rainwater (W_{LP} = 0.56).

<table>
<thead>
<tr>
<th>Materials typology</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>Mean value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gc</td>
<td>18.3</td>
<td>15.4</td>
<td>17.8</td>
<td>17.2</td>
</tr>
<tr>
<td>Gs</td>
<td>21.1</td>
<td>22.0</td>
<td>19.8</td>
<td>21.0</td>
</tr>
<tr>
<td>B</td>
<td>21.9</td>
<td>22.9</td>
<td>19.2</td>
<td>21.3</td>
</tr>
<tr>
<td>G</td>
<td>20.6</td>
<td>19.2</td>
<td>19.9</td>
<td>19.9</td>
</tr>
</tbody>
</table>

Figure 3 - Capillary water absorption curves of the manufactured specimens
Concerning the QUV tests, “G” and the “Gc” specimens were characterized by a general lack of decay, showing a good resistance to the accelerated aging cycles that were carried over. The “B” specimens have shown a slight superficial decay in the shape of a very thin fading, and of just visible cracks, and for this reason this decay involves both the paint, and the plaster. Finally, the “Gs” specimens show a significant decay, mainly characterized by a slight fading of the pictorical layer, and in particular by chalking, and clearly visible cracks.

The color difference, in the last column of the following table 4 (referred only to one of the series of specimens), is obtained with the formula $\Delta E = (\Delta L^2 + \Delta u^2 + \Delta v^2)^{1/2}$.

Table 4 - Influence of the accelerated aging test on the color variation.

<table>
<thead>
<tr>
<th>SERIES</th>
<th>PLASTER</th>
<th>PAINT</th>
<th>n° of samples</th>
<th>$\Delta E$</th>
<th>Mean $\Delta E$</th>
<th>Colour fading rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>Lime</td>
<td>Lime</td>
<td>1</td>
<td>2.09</td>
<td>0.82</td>
<td>1.85</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2</td>
<td>0.90</td>
<td>0.74</td>
<td>COLOUR FADING 1</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3</td>
<td>0.93</td>
<td>0.73</td>
<td></td>
</tr>
<tr>
<td>G</td>
<td>Macroporous</td>
<td>Breathable</td>
<td>1</td>
<td>0.90</td>
<td>0.74</td>
<td>COLOUR FADING 0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2</td>
<td>0.93</td>
<td>0.73</td>
<td></td>
</tr>
<tr>
<td>Gc</td>
<td>Cement</td>
<td>Quartz</td>
<td>1</td>
<td>1.68</td>
<td>1.33</td>
<td>COLOUR FADING 0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2</td>
<td>1.54</td>
<td>1.33</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3</td>
<td>1.53</td>
<td>0.73</td>
<td></td>
</tr>
<tr>
<td>Gs</td>
<td>Lime - cement</td>
<td>Siloxan</td>
<td>1</td>
<td>1.54</td>
<td>1.53</td>
<td>2.21</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2</td>
<td>1.54</td>
<td>1.53</td>
<td>COLOUR FADING 2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3</td>
<td>1.53</td>
<td>0.73</td>
<td></td>
</tr>
</tbody>
</table>
On the field, as shown in figures 6 and 7, the buildings covered with lime plaster + lime paint and those with “common” mortar + siloxane paint, have been subject to more consequences than the others, because of rising dampness, micro-cracks, temperature variations. For what concerns performances of adhesion of the plasters on masonry, there have not been significant differences in ten years of monitoring. A previous project verified that the lack of adhesion begins from the 15th year.

![Image](image.png)

**Figure 6-7: micro-cracks and rising dampness in a building with “Gs” solution**

5. CONCLUSIONS

The tests have shown some important evidences:
- the “G” specimens are characterized by good results both in water vapor permeability and in water absorption at a low pressure tests: this is not a well-known information, and it is particularly useful for the designers of recovery interventions;
- also QUV tests show that “G” specimens have, over the time, only a little decay of initial characteristics;
- the behavior of “Gs”, the most used solution in the recovery interventions in the last 10 years in the city of Naples, is the worst among the selected specimens;
- all the results that have just been exposed, actually, find a direct correspondence in what it was possible to observe in all the tested buildings of the centre of Naples;
- during the first ten years of monitoring, the tests on the field effectively showed that the finishing solutions of macroporous plaster + silicate paint and cement plaster + quartz paint have a higher attitude to last during the time for the respective characteristics, with the condition that the designers choose the most fitting solution, not considering the real context of the stress agents;
- the conducted tests seem to confirm the importance of knowing the real performances of the whole system rather than of a single component, especially in relation to a predetermined stressing context;
- the goal is the creation of a handbook for designers, in order to lead to a choice of more appropriate and durable building components.
A METHODOLOGY FOR EVALUATING MAINTAINABILITY AS A TOOL FOR DURABLE AND SUSTAINABLE BUILDINGS

Maurizio Nicolella

Department of Civil, Construction and Environmental Engineering, University of Naples Federico II, Naples – maurizio.nicolella@unina.it

Abstract

Maintainability is a very important requirement in construction projects for making maintenance and management phases easier and less expensive and achieving durable and sustainable buildings. It can be defined as the capacity of a component to be subjected to maintenance interventions.

Nevertheless, maintainability is currently an abstract – although recognizable – concept, that could be very useful for evaluations that could allow, for example, a designer to evaluate the best technological solution or a Public Administration to choose the less expensive among different projects in terms of maintenance.

A method for estimating the “Maintainability degree” of a building was implemented and tested in a range of research programs on planned maintenance that have been carried out since 1988. The method is based on an index which considers, through evaluation, several factors regarding the components in which a building can be broken down.

This Maintainability Index is able to measure the capacity of a building to be subjected to maintenance interventions.

Keywords: maintainability, durability, costs, index.

1. INTRODUCTION

Several definitions have been suggested for the term “maintainability”, mainly in two different acceptations. One relates to the ease of doing interventions on a component, with the aim of bring the performances of a component back to the values that were defined in the project (qualitative definition: “aptitude of receiving interventions of maintenance”), while the other one provides the value of this so-called aptitude in probability terms (quantitative definition: “the capacity to conform to certain conditions in a certain period of time, during which maintenance takes place”).

Four variables interact with the concept of maintainability, and they represent the objects of study for the operative applications. They are:
The ease with which maintenance interventions take place, that is to say the “aptitude” or the predisposition of the element or of the system, to be object of maintenance. It is not only characteristic of technical elements, but also of the of the organizational structure where technical elements are located;

- the operative organization for the recovery of the performances of the component or of the system through maintenance operations;
- the time necessary to the development of maintenance operations;
- the “value” assumed by maintenance, in which quality improvement consists in the increase of the probabilities of being able to complete the maintenance operations of recovery in a certain time.

The maintainability of a component, of a sub-system or of a full building structure is one of the project requirements that most express the sensitivity and the carefulness a designer should have towards the management and the life of the building, mainly through its maintenance. It would be dangerously wrong to think that the requirement of maintainability is simply related to decisions made only in the phase of the management of the building product.

The present work suggests a methodological hypothesis that evaluates maintainability through an “index” that is function of the morphological, typological and technological characteristics of buildings.

2. MAINTAINABILITY FACTORS

The maintainability factors are elements that have influence on maintainability for different aspects: they are designing factors, organizational factors and operative factors, that are syntethized in the following chart. It explains how it is possible to make influences on the ease of carrying over maintenance interventions in the designing-decisional phase, in the operative one, and also – eventually – in the organizational one, more related to the industrial environment.

Table 1 – Typologies of maintenance factors

<table>
<thead>
<tr>
<th>MAINTENANCE FACTORS</th>
<th>Designing factors</th>
<th>Organizational factors</th>
<th>Operative factors</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level of complexity of the entity</td>
<td>Typological, distributional and geometrical characteristics</td>
<td>Availability of an informative system for the management of the maintenance</td>
<td>Labour ability and its level of specialization</td>
</tr>
<tr>
<td>Level of divisibility</td>
<td>Accessibility of the building structure, sub-systems and components</td>
<td>Logistic organization of the maintenance service</td>
<td>Availability of technical documentation for the execution of interventions</td>
</tr>
<tr>
<td>Dislocation, dimension, organization and ergonomics of the operative spaces</td>
<td>Visibility of components</td>
<td>Efficiency of the organizational structure</td>
<td>Number and quality of the technical means employed for the interventions</td>
</tr>
<tr>
<td>Mobility of components</td>
<td>Modularity of components</td>
<td>Level of involvement of customers for management and maintenance</td>
<td></td>
</tr>
<tr>
<td>Standardization of components</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
3. THE INDEX OF APPTITUDE TO MAINTAINABILITY

It can be initially argued that the aptitude to maintainability of a building can be considered as the summation of the aptitudes to maintainability of the single components, sub-systems and systems. So, defining the index of aptitude to maintainability $N$ as the parameter that measures the ease of executing maintenance interventions for a certain component, it results that:

$$N_{tot} = \sum_{i=1}^{n} N_{i}$$  \hspace{1cm} (1)

The index of aptitude to maintainability is a function of the cost of the intervention of maintenance, $C_{int}$, and of the impracticability that the intervention causes, $T_{i}$.

$$N_{i} = f(C_{int}, T_{i})$$  \hspace{1cm} (2)

In particular, this coefficient is inversely proportional to these two variables since maintainability obviously decreases as they grow.

The numerous other variables to consider, for example everything related to maintenance (construction typologies, connections between the various elements, actuation modalities, etc.), and also the impracticability that is consequent to the execution of maintenance interventions, have been translated into economical values, in the aim of a necessary dimensional homogenization of the index of maintainability.

4. THE TEMPORARY IMPRACTICABILITY DURING MAINTENANCE INTERVENTIONS

It is easy to understand that MTTR (Mean Time To Repair) plays a major role in the evaluation of maintainability.

Actually, MDT (Maintenance Downtime) should be considered instead of MTTR, and that is the time of total impracticability, the sum of the following rates (UNI 9910):

- time for the identification of the malfunction.
- ADT, Administrative Delay Time, null in the case of a preventive maintenance;
- LDT, Logistic Delay Time, due for example to a temporary unavailability of resources, and that should be null as well, in the case of a preventive maintenance;
- time to verify the functioning, to consider in the end of the maintenance intervention;
- MTTR, Mean Time To Repair.

The economical evaluation of the temporary impracticability was executed considering the economical implication of the impracticability of a building, that is to say the lack/decrease of the annual net income, that was calculated as:

$$NI = GI - (Q + Tr + Adm + Svc + VU)$$  \hspace{1cm} (3)
meaning:
GI = gross income;
Q = rates, the expenses that the owner has to sustain for the conservation of the building (reintegration, insurance);
Tr = tributes, include taxes and contributions;
Adm = administration, the expenses that include the management of the contract of rent (consulting, chancery, fiscal assistance);
Svc = services, include the expenses of management of a building in charge of the owner (custody, conciergery, cleaning, illumination, waste disposal, etc.);
VU = vacancy and unmanageability, include the losses, intended as missed incomes, that the owner cannot receive when the property is not being rented, or when the renter does not pay, or pays the rates with a delay.

Once the annual net income has been defined, to obtain the cost of impracticability, it will be sufficient to calculate the income loss per hour, and multiply it for the time of total impracticability:

\[ C_{impr.} = N I_{hour} \cdot T_i \]  \hspace{1cm} (4)

5. THE COST OF MAINTENANCE INTERVENTIONS

The variable of cost is a function itself, both in relation with the single components, and with the whole building, that is to say in relation in the way the single component has influence on the building.

\[ C_i = f'(X_q) \]  \hspace{1cm} (5)

This function depends on various variables, the Xs, which are characterized by two subscripts. The subscript ‘j’ indicates the variable, while the subscript ‘i’ indicates the i-th component the variable is referred to several variables have influence on the determination of the cost of maintenance for every i-th component:

- Length of the life of the component and forms of maintenance
- Frequency of maintenance interventions, and necessity to do them at certain times
- Competence, nature of the title of property
- Modalities of operation during the phase of management, and subjects in charge
- Technical and operational connections
- Dimensions, that is to say the physical and technical units of measure
- Constructional technology, what constitutes the component materials

6. CONSTRUCTION OF THE MODEL

After preliminarily disassembling the structure of the building, for example using the WBS technique, it is possible to calculate the index of aptitude to maintenance N. This indicator N is inversely proportional to the cost of the intervention on the i-th component, and to the cost of the temporary impracticability that it causes, with the consequent discomfort for the residents:
\[ N = \frac{100}{\sum_{i=1}^{n} C_{\text{int},i} + \sum_{i=1}^{n} C_{\text{impr},i}} \]  \hspace{1cm} (6)

meaning:
\( C_{\text{int},i} \) = the parametrical cost of the maintenance intervention for the i-th element;
\( C_{\text{impr},i} \) = the parametrical cost caused by the temporary impracticability that the maintenance of the i-th element causes.

The value 100, present in the numerator, has the aim of representing maintainability as a coefficient of efficiency.

As it has been noted in the previous paragraph, the cost of maintenance depends on eight j-ths variables, that are different for every i-th component.

\[ C_{\text{int}} = f' (X_{j,i}) = f' (X_{1,i}, X_{2,i}, X_{3,i}, X_{4,i}, X_{5,i}, X_{6,i}, X_{7,i}, X_{8,i}) \]  \hspace{1cm} (7)

meaning:
\( X_{1,i} \) = form of maintenance used for the i-th component;
\( X_{2,i} \) = length of the life of the i-th component;
\( X_{3,i} \) = frequency of the interventions of maintenance for the i-th component;
\( X_{4,i} \) = competence, nature of the title of property of the i-th component;
\( X_{5,i} \) = subjects in charge of the maintenance of the i-th component;
\( X_{6,i} \) = technical and operational connections of the i-th component;
\( X_{7,i} \) = dimensions, that is to say physical and technical units of quantitative measure of the i-th component;
\( X_{8,i} \) = constructional technologies, the constitutional nature of the materials the i-th component is made up by.

Some of these variables will be carried out in the model using the factors of maintainability, that have already been quoted, while others will be subjects of a further analysis.

Regarding the operative and technological connections, it should be noted that the cost of intervention, \( C_{\text{int}} \), can be higher or lower, depending on whether the technological connections, that make the maintenance cost grow, are prevalent, or the operative connections, which optimize the cost, are more present. A technological connection is intended as the necessity to operate on a certain element when an intervention is done on others.

Therefore, the technological connection is the interconnection between elements, that makes the intervention on two or more elements at the same time, unavoidable, and that interconnection is present, for example, between finishing elements, such as plaster and paint. It is necessary to clarify that the connection is not bi-directional: it is obvious that an intervention on a plaster necessary influence the paint as well; however, an intervention on a paint does not necessarily influence the plaster.

This final consideration allows to define opportunely the operative connection that, on the other hand, concerns elements that should be regarded by interventions of maintenance in the same periods of time, not because of an interconnection between them, but because they have similar executive and organizational characteristics, which create the opportunity to do interventions on an element when interventions are done on the other elements, in order to make use of the same means of maintenance with the same cost, and reducing the discomfort.
of residents during time. A typical example is provided by the inspections of the installments: if it is hypothesized, for example, to have various installments going through the same false ceiling, the interventions and the verifications should happen in the same period of time, in order to reduce the total costs of organization and site management, and also the residents’ discomfort. Another example concerns all the interventions of maintenance that require the use of scaffolding (for evident reasons of cost and temporary discomfort).

In order to easily examine all the connections existing between the components of a building, a “Matrix of Connection” (see figure 1) can be realized: it highlights the typology of connections that exist between any two components, and their intensity.

In the matrix, every square individuated by the intersection of lines and columns is divided in four triangles that define different connections according to the different colors, as shown in figure 2, which can be:

- weak technological connections;
- strong technological connections;
- weak operative connections;
- strong operative connections.

![Figure 1: Typical matrix of connections](image)

![Figure 2: Chart of connections](image)

In order to consider these connections, the expression of the coefficient $N$ can be rewritten as:

$$N = \frac{100}{\sum_{i=1}^{m} \left[ C_{\text{int},i} \cdot (1 + \lambda_i - \lambda_i^*) \right] + \sum_{i=1}^{n} [C_{\text{impr},i}]}$$

In this formula:
\( \lambda_i' \) is the ratio between the weighted number of technological connections and the number of total connections that the i-th element has with all the other elements;

\( \lambda_i'' \) is on the other hand the ratio between the weighted number of operative connections and the number of total connections that the i-th has with all the other elements:

\[
\lambda_i' = \frac{\sum_n L_i'}{\sum_n L_i' + \sum_n L_i''} \quad \lambda_i'' = \frac{\sum_n L_i''}{\sum_n L_i' + \sum_n L_i''}
\]  

(9-10)

The coefficient that multiplies the parametrical cost is greater than 1 if there is a prevalence of technological connections increasing the cost, is equal to 1 if the technical connections are the same number as the operative connections, or lesser if there are more operative connections (that optimize the cost of maintenance). Every connection, both technological and operative, has a value of 1 if it is weak, and of 2 if it is strong.

The factors of maintainability, defined before, should be considered as well: they can be indirectly calculated using the norm UNI 8290, that is to say non-tarnishability, buffability, viewability, repairability, relievability, complexity, decomposability, moveability, standardizability, and availability of maintenance plan.

It is important to note that, while some factors can only be referred specifically to the single components, or to the single sub-systems, other factors can be referred to the whole building structure. Some factors can also be referred to multiple entities, for example the identificability of the state of functioning can be related to a whole sub-system, or to each of its single components. So, the first step is to refer each interested factor to the various entities that could be conditioned by the value that can be attributed to the factor.

The second step is that of expressing them as percentages whose sum is always equal to 1. Each of the chosen factors of maintainability \( K \) has also to be corrected with a factor \( h \), which is used to quantify the level of influence of each component to the value of the factor itself. The value of \( h \) is also expressed in a percentage, the nit varies between 0 and 1:

\[
\sum_{i=1}^{n} K_j^i \cdot h_j^i
\]

(11)

\( C_{int} \) is then divided by this summation, because, the smaller is the unitary behavior of the i-th element, the smaller is its maintainability, and since the value of the summation \( K_j^i \cdot h_j^i \) is between 0 and 1, this is placed in the denominator of \( C_{int} \). But the same reasoning can be made for the cost of impracticability \( C_{impr} \): in fact, some factors of maintainability mainly influence this cost, rather than the cost of the intervention itself. Of course, this coefficients can also vary in relation to the end use of the building or of its parts, because the end use can attribute different functional importance to the same typology of sub-system.

Taking account of this, the previous expression becomes:

\[
I_m = \frac{100}{\sum_{i=1}^{n} \frac{C_{int, i} \cdot (1 + \lambda_i' - \lambda_i'')}{\sum_{j=1}^{n} K_j^i \cdot h_j^i} + \sum_{i=1}^{n} C_{impr, i}}
\]

(12)
Concerning the cost of impracticability \( C_{\text{imp}} \), it represents the loss of income that is verified in the period of time during which the substitution or the restoration of the \( i \)-th component takes place. It is influenced by two main aspects:

- **the localization of the element** that has been submitted to a specific operation of maintenance, the criticality of the component or of the sub-system examined, in the context where it is located;
- **the duration of the intervention**, that is the total impracticability, as it has already been defined.

So, the cost of the impracticability of the \( i \)-th element has been defined as the percentage rate estimated in relation to the total cost that has been predicted for the interventions of maintenance in a period of 50 years. The mentioned costs, in order to be summed, have been first projected in the valuable period of life according to an annual medium increase (slightly more than 3\% for the cost of interventions and a bit more than 2\% for the cost of impracticability, in relation to the lease), and then summed according to the VAN function with a rate of annual discount equal to the rate of legal interest (2.5\%).

7. CONCLUSIONS

Thanks to the application of the model to a series of case studies, it has resulted that the value of the index of maintainability can be approximated to a value between 0.9 and 1 in the case of medium-normal costs of maintainability, assuming the connections and the corrections made according to the costs of maintenance do not influence this costs positively, nor negatively. It is lesser than 0.9 if the technological connections are more present than operative connections, and/or if the values of the factors of the \( i \)-th element are close to 1; greater than 1 if there is a prevalence of operative connections and/or when the summation \( K_{ij}h_{ij} \) tends to 1, and therefore does not influence the final value of the index.

If the intervention takes place in a period where there are no diseconomies, approximable to a duration that is lesser than 15 days (it can be considered that the monthly income has been completely lost after the 15th day), then the second term of cost in the denominator is null.

REFERENCES

INVESTIGATION OF THE EFFECTS OF LEACHING ON THE SCALING RESISTANCE OF CONCRETE

Martin Rosenqvist (1,2), Manouchehr Hassanzadeh (2,3), Long-Wei Pham (2) and Adnan Terzie (2)

(1) Vattenfall AB, Älvkarleby, Sweden – martin.rosenqvist@vattenfall.com
(2) Division of Building Materials, Lund University, Lund, Sweden – vv08lp3@student.lth.se; vv08at4@student.lth.se
(3) Vattenfall AB, Stockholm, Sweden – manouchehr.hassanzadeh@vattenfall.com

Abstract
Damage to the concrete surface can be observed at the waterline of hydraulic structures in fresh water bodies in cold climates. Gradual deterioration of the concrete surface leads to exposure of coarse aggregate. Superficial damage at the waterline is commonly assumed to be caused by drifting ice floes. However, deterioration of concrete at the waterline may also involve leaching and frost action. The objective of this study is to investigate if leaching of calcium compounds changes the surface properties of concrete in such an extent that the scaling resistance is reduced.

Specimens with water to cement ratio 0.62 and 0.54 were submerged in deionised water at +20 °C and pH 4 ± 0.1 in order to accelerate leaching of calcium compounds. The scaling resistance of concrete was assessed according to the Swedish test method SS 13 72 44. The results show that the scaling resistance of concrete is reduced if calcium compounds have been leached out from the surface. The results also show that the longer the time of leaching, the greater the reduction in scaling resistance. Superficial damage at the waterline of hydraulic structures in fresh water bodies in cold climates is most likely caused by interaction between leaching, frost action and abrasion.

Keywords: Concrete, Leaching, Scaling resistance, Freeze-thaw cycles, Hydraulic structures
1 INTRODUCTION

Damage to the concrete surface can be observed at the waterline of hydraulic structures in fresh water bodies in cold climates. Hydro power structures in Sweden are an example of such structures. Gradual deterioration of the concrete surface results in exposure of coarse aggregate, see Figure 1. The greatest amount of damage is found at the waterline, which normally corresponds to the maximum water level. The amount of damage decreases with increasing water depth. In a long-term perspective, also the reinforcing steel can be exposed. Hence, the structural integrity and durability of these structures can be reduced.

Superficial damage at the waterline is commonly assumed to be caused by drifting ice floes. Abrasive wear of the concrete surface may occur if ice floes push against the structures. This scenario is true regarding concrete structures at intakes and spillways on the upstream side of hydro power plants where the river current leads ice floes towards the structures. On the downstream side, however, the water flows away from the structures and carries away the ice floes. In spite of this fact, superficial damage at the waterline can be observed on the downstream side of hydro power plants. Such observations indicate that damage to the concrete surface is not exclusively caused by ice abrasion.

The deterioration process of the concrete surface at the waterline may involve carbonation, dissolution and leaching of calcium compounds, frost action and ice abrasion. Even though the resistance of concrete to a certain deterioration mechanism is good, the effects of other mechanisms may significantly reduce the resistance to the first mechanism. Since hydraulic structures often are subjected to a number of deterioration mechanisms, synergy may occur. Synergy is defined as the interaction of two or more elements, which together produce an effect greater than the sum of their individual effects.

The objective of this paper is to investigate if leaching of primarily calcium compounds from the concrete surface reduces the scaling resistance of concrete. If this is the case, it may explain why superficial damage can be seen at the waterline of hydraulic structures in fresh water bodies in cold climates. This paper includes results on the scaling resistance of concrete from both existing structures and concrete specimens subjected to leaching in the laboratory.

Figure 1: Coarse aggregate exposed at the waterline on the upstream face of a concrete dam. The water level was about 0.3 m lower than normal when the picture was taken.
2 DETERIORATION OF CONCRETE

2.1 Exposure conditions

The climate in Sweden is variable, especially in the northern parts of the country where summers are warm and winters are cold. Air temperatures down to -30 °C are common during the winter months. The difference between daily minimum temperatures in winter and daily maximum temperatures in summer is usually in the range of 50 to 60 °C. During the spring and autumn months, the air temperature frequently crosses the freezing point of water.

In Figure 2, the same structure is shown in the morning and in the afternoon on the same day. Ice has formed at the waterline during the night, while it has melted away during the day. The next morning, the concrete surface was covered with ice again. Krus (1996) theoretically showed that concrete structures can be subjected to up to 50 freeze-thaw cycles over a year in northern Sweden. In mid winter, however, a band of ice frozen solid to the concrete surface can be seen for months at the waterline of hydraulic structures.

![Figure 2: The concrete surface at the waterline is covered with ice in the morning [A], while the ice has melted away in the afternoon [B].](image)

Concrete structures partly submerged in water are affected in different ways due to varying water chemistry of the water bodies. The river water in Sweden may be considered aggressive with regard to the risk of leaching of calcium compounds from the concrete surface. Drugge (2001) showed that the calcium ion concentration of the water in Swedish rivers, due to the geology of Sweden, is low in comparison with most other rivers around the world. For example, the calcium ion concentration is about 3 mg/l in the river Lule älv in northern Sweden. Low content of calcium ions in the river water increases the capacity of the water to dissolve calcium compounds from the concrete surface.

2.2 Frost resistance of concrete

The design life of hydraulic structures has traditionally ranged from 50 to 100 years. In cold climates, the frost resistance of concrete is of great importance to the durability of the structures. Powers (1945) stated that concrete subjected to frost action can be damaged either by scaling or internal damage. Scaling deteriorates the surface, whereas internal damage lowers the compressive and tensile strength of concrete. Powers proposed the hydraulic
pressure theory to explain frost damage. The theory was based on the fact that the volume of water expands by about 9% upon freezing. When excess water is forced out of the capillary pores, a hydraulic pressure gradient is created by the flow resistance in the cement paste. Frost damage occurs if the hydraulic pressure exceeds the tensile strength of cement paste.

Later findings by Powers and Brownyard (1948) showed that the freezing point of water in cement paste is dependent on the pore size. Nucleation of ice crystals becomes more difficult as the pore size decreases. When water freezes in large capillary pores, water in small capillary pores and gel pores remains unfrozen. The amount of freezable water increases with decreasing temperatures. Based on these findings, Powers and Helmuth (1953) proposed the microscopic ice lens growth theory. Ice crystals in large capillary pores attract water from the gel and smaller capillary pores. Consequently, water moves into the large capillary pores and causes the ice crystals to grow to microscopic ice lenses.

The frost resistance of concrete is normally improved when an air entraining agent is added to the concrete mix. Artificially created air voids are not filled with water by capillary forces and therefore remain air filled. Hence, Powers (1949) explained that air voids provide empty spaces within the concrete where ice can form without exerting pressure on the pore walls.

### 2.3 Leaching of calcium compounds

All hydration products of Portland cement are soluble in water to varying degrees. The rate at which calcium compounds are dissolved in water depends on the aggressiveness of water and the permeability of concrete. The process of dissolving calcium compounds in water is called leaching. Dissolution of the cement paste increases the pore volume and thus porosity. The most soluble hydration product is calcium hydroxide (CH), also known as portlandite. Calcium compounds are also dissolved from other hydration products, such as the CSH gel.

According to Beddoe and Dorner (2005), dissolution and leaching of calcium ions from the CH start when the pH value of the pore solution is below 12.6. Regarding the CSH gel, the corresponding value is 10.5. At pH values between 4.0 and 6.5, only a small amount of calcium remains in hydration products containing iron and aluminium. Calcium ions from the other hydration products are dissolved to a much lesser extent as long as there is CH left. At pH values below 2, the cement paste has been reduced to a silica gel residue.

Ekström (2003) investigated the effects of percolating water on the leaching process of concrete in hydro power dams. Besides the effects of percolating water, he also measured the remaining content of calcium ions in concrete specimens submerged in deionised water for two years. The results showed that leaching of calcium compounds was greater close to the surface than deeper into the specimens. In order to verify results from specimens produced and tested in the laboratory, it is also important to test specimens from existing structures.

### 3 STENKULLAFORS POWER PLANT

Stenkullafors power plant is situated along the river Ångermanälven in northern Sweden. Construction work began in the early 1980s and the power plant was commissioned in 1983. Slag cement called Massivcement was used in the concrete. The slag content of the cement was 65%. At the time of construction, concrete with cement content 300-350 kg/m³ and water to cement ratio (w/c-ratio) between 0.50 and 0.55 was normally used in hydro power structures. In order to improve the frost resistance of concrete, an air entrainment agent was added to the concrete mix. Between 2004 and 2006, after more than 20 years, the spillway section of the power plant was rebuilt and the spillway crest was lowered by 4 m.
Hasanzadeh (2010) studied the scaling resistance of left over concrete from the spillway section. The scaling resistance was assessed according to the Swedish test method SS 13 72 44, which was originally developed to evaluate salt scaling resistance of concrete, see Svensk Standard (2005). The results showed that the scaling resistance of the outer surface was remarkably reduced compared to the cut surface. Some of the results from the study are presented in Table 3. The reduction in scaling resistance of the outer surface could, however, not be fully explained. Bleeding, carbonation and/or leaching of calcium compounds from the concrete surface were suggested as possible causes. Especially the uncertainty about the effects of leaching on the scaling resistance of concrete showed the need for further studies.

4 MATERIALS

In order to investigate the effects of leaching of primarily calcium compounds from the concrete surface, two concrete mixes were cast; w/c-ratio 0.62 and 0.54. The mix proportions, shown in Table 1, were selected to represent concrete mixes used during the most intense period of the Swedish hydro power development. The mix with w/c-ratio 0.62 is typical for concrete used in hydro power structures built between 1930 and 1950, whereas the mix with w/c-ratio 0.54 is typical for concrete used between 1950 and 1970. The w/c-ratio of concrete mixes used after 1970 is somewhat lower due to the use of more efficient admixtures.

A CEM I Portland cement (Anläggningscement) was used in the concrete mixes. It is a moderate heat and low alkaline binder developed in the 1980s. The clinker phase composition of the Anläggningscement is relatively similar to the Limhamn cements, which were often used in the construction of Swedish hydro power structures between 1935 and 1980.

Table 1: Proportions of the two concrete mixes with w/c-ratio 0.62 and 0.54.

<table>
<thead>
<tr>
<th>w/c-ratio</th>
<th>Cement (kg/m³)</th>
<th>0-1 mm (kg/m³)</th>
<th>0-8 mm (kg/m³)</th>
<th>1-4 mm (kg/m³)</th>
<th>4-8 mm (kg/m³)</th>
<th>8-16 mm (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.62</td>
<td>325</td>
<td>335</td>
<td>440</td>
<td>65</td>
<td>110</td>
<td>835</td>
</tr>
<tr>
<td>0.54</td>
<td>300</td>
<td>320</td>
<td>300</td>
<td>75</td>
<td>200</td>
<td>950</td>
</tr>
</tbody>
</table>

After pre-mixing of cement and aggregate, water was added and mixed for three minutes. Next, concrete was cast into moulds of size 150x150x150 mm. The top surface of the moulds was covered with plastic foil until demoulding 24 hours later. The concrete cubes were then stored in lime saturated water at 20 °C until the test of the 28-day compressive strength. Properties of the fresh and hardened concrete mixes are presented in Table 2. At the age of 28 days, the cubes intended for use were split into specimens of size 150x150x50 mm. A water sealant was applied to all surfaces except the test surface, see Figure 4.

Table 2: Properties of the fresh and hardened concrete mixes.

<table>
<thead>
<tr>
<th>w/c-ratio</th>
<th>Air Content (%)</th>
<th>Slump (mm)</th>
<th>Density (kg/m³)</th>
<th>Strength 28 Days (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.62</td>
<td>1.3</td>
<td>80</td>
<td>2375</td>
<td>40.2</td>
</tr>
<tr>
<td>0.54</td>
<td>4.1</td>
<td>25</td>
<td>2350</td>
<td>43.0</td>
</tr>
</tbody>
</table>
5 METHODS

Sets of three specimens were subjected to leaching and/or freeze-thaw cycles in order to study the effects of leaching on the scaling resistance of concrete. Dissolution and leaching of calcium compounds were accelerated when the specimens with w/c-ratio 0.62 and 0.54 were submerged in deionised water at +20 °C and pH 4 ± 0.1. The pH-value was maintained by automatic addition of 1 mol/l nitric acid (HNO$_3$). The solution was renewed once a week. The test setup is shown in Figure 3 and it was based on work by Rozière et al (2009).

![Figure 3](image)

Figure 3: Test setup used to accelerate leaching of calcium compounds from the surface of the concrete specimens. Nitric acid was automatically added in order to keep the pH value at 4.

The scaling resistance of concrete was assessed according to the test method SS 13 72 44, see Svensk Standard (2005). The specimens were insulated on all sides except the test surface, which was covered with deionised water throughout the frost test. The freeze-thaw cycle was 24 hours long and during this period of time, the concrete surface temperature alternated between -20 ºC and +20 ºC. A full frost test procedure consists of 56 freeze-thaw cycles. The freeze-thaw cycle and the cross section of an insulated specimen are shown in Figure 4.

Reference specimens were subjected to the full frost test, whereas the specimens subjected to leaching prior to the frost test were subjected to only seven freeze-thaw cycles. The first set of specimens was submerged for one week prior to the frost test, whereas the second set of specimens was submerged for eight weeks. The effects of the frost test were quantified as the weight of loose materials collected from the concrete surface of each specimen. The weight of the loose materials was recalculated to weight loss per square meter (kg/m$^2$).

![Figure 4](image)

Figure 4: Concrete specimen [A], water sealant [B], insulation [C], deionised water [D] and plastic foil [E]. The freeze-thaw cycle is shown in the picture to the right.
6 RESULTS

When the scaling resistance of concrete from the spillway section of Stenkullafors power plant was assessed by Hassanzadeh (2010), it was shown that the scaling resistance of the outer surface was remarkably reduced compared to the cut surface. The scaling resistance of the cut surface was classified as Very good (< 0.1 kg/m$^2$), whereas the outer surface was classified as Unacceptable (> 1.0 kg/m$^2$). The results are presented in Table 3.

After 56 freeze-thaw cycles, no loose materials had been collected from the surface of the reference specimens with w/c-ratio 0.54. For the reference specimens with w/c-ratio 0.62, only a small amount of loose materials was collected throughout the frost test. When the specimens were submerged in deionised water at pH 4 for one respectively eight weeks prior to the frost test, the amount of loose materials collected from the surface increased somewhat.

The smallest amount of loose materials was collected from the specimens with w/c-ratio 0.54, which were subjected to leaching for one week prior to the seven freeze-thaw cycles. The greatest amount of loose materials was collected from the specimens with w/c-ratio 0.62, which were subjected to leaching for eight weeks prior to the seven freeze-thaw cycles.

Table 3: Accumulated weight of loose materials collected from the surface of specimens from Stenkullafors power plant (SPP) and from specimens with w/c-ratio 0.62 and 0.54.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Leaching</th>
<th>Surface scaling (kg/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPP outer surface</td>
<td>20+ years</td>
<td>0.07 0.59 1.37 1.55 1.64</td>
</tr>
<tr>
<td>SPP cut surface</td>
<td>-</td>
<td>0.01 0.01 0.02 0.02 0.03</td>
</tr>
<tr>
<td>0.54 cut surface</td>
<td>-</td>
<td>0.00 0.00 0.00 0.00 0.00</td>
</tr>
<tr>
<td>0.54 cut surface</td>
<td>1 week</td>
<td>0.01 0.00 0.00 0.00 0.00</td>
</tr>
<tr>
<td>0.54 cut surface</td>
<td>8 weeks</td>
<td>0.04 0.04 0.04 0.04 0.04</td>
</tr>
<tr>
<td>0.62 cut surface</td>
<td>-</td>
<td>0.00 0.00 0.01 0.02 0.02</td>
</tr>
<tr>
<td>0.62 cut surface</td>
<td>1 week</td>
<td>0.02 0.02 0.02 0.02 0.02</td>
</tr>
<tr>
<td>0.62 cut surface</td>
<td>8 weeks</td>
<td>0.06 0.06 0.06 0.06 0.06</td>
</tr>
</tbody>
</table>

7 DISCUSSION

The results show that the scaling resistance of concrete is reduced if calcium compounds are leached out from the surface. Therefore, it is reasonable to assume that leaching increases the porosity and thus the average pore size in the surface layer. Hence, the risk of frost damage increases. The scaling resistance of the concrete mixes used in this study was reduced when the specimens were subjected to leaching for one week prior to the frost test. It was also shown that the longer the time of leaching, the greater the reduction in scaling resistance.

Regarding the specimens taken from the left over concrete from Stenkullafors power plant, the scaling resistance was remarkably reduced for the outer surface compared to the cut surface. In comparison with the specimens with w/c-ratio 0.62 and 0.54, the outer surface has been exposed to river water for more than 20 years. Even if the river water is less aggressive than deionised water at pH 4, the duration of exposure has increased the frost susceptibility of
the surface layer. However, it has to be kept in mind that the type of cement is different and bleeding as well as carbonation may have affected the scaling resistance. Nevertheless, leaching of calcium compounds from the concrete surface has taken place over the years.

The concrete from Stenkullaflors power plant and the concrete cast in the laboratory have in common that the scaling resistance is reduced when calcium compounds are leached out from the surface. Damage to the concrete surface at the waterline of hydraulic structures in cold climates, see Figure 1, is most likely caused by interaction between leaching and frost action. These deterioration processes together produce an amount of damage greater than the sum of their individual effects. In reality, also abrasive wear occurs if ice floes push against the structures. The damaged surface layer is thus removed and a new surface is exposed.

A possible deterioration process of concrete at the waterline of hydraulic structures in cold climates is that leaching of calcium compounds from the surface starts during the snowmelt runoff period. Hence, the concrete surface becomes frost susceptible and during the following winter, the surface layer is damaged by frost action and eventually removed by ice abrasion.

8 CONCLUSIONS

Based on the results presented in this paper, the scaling resistance of concrete is reduced if calcium compounds are leached out from the concrete surface. Hence, damage to the concrete surface at the waterline of hydraulic structures in fresh water bodies in cold climates is most likely caused by interaction between leaching, frost action and abrasion.

ACKNOWLEDGEMENTS

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REFERENCES

SERVICE LIFE OF INNOVATIVE SILOXANE COATING FORMULATIONS

Daniele Enea (1), Giuseppe Alaimo (2)

(1) Department of Architecture, University of Palermo, Palermo – daniele.enea@unipa.it
(2) Department of Architecture, University of Palermo, Palermo – giuseppe.alaimo@unipa.it

Abstract
The use of durable products on the external surfaces of buildings ensures high performance and low maintenance. The world market in recent decades has developed a wide range of solutions of limited thickness coatings; the Italian territory is particularly interested due to the strong link, by tradition, with plaster as surface finishing. The knowledge of the performance over time of such innovative products is one of the main research activities of the Italian network on durability and becomes essential due to the new EU regulation No. 305/2011 which introduced, among the requirements for the construction works, sustainability and durability with it. The research concerns the assessment of the durability of some innovative coatings based on acrylic-siloxane resins, in three different colors. The parameters investigated are: the surface appearance, by scanning electron microscope and optical microscope, the color, by spectrophotometer, the absorption of water and the wettability by contact angle measurement. The paper reports the results of the experimental study on siloxane coatings, produced by Hydratite Srl, performed according to the ISO 15686, by monitoring the behavior over time of samples artificially aged in climatic chamber with variable temperature and relative humidity, rain and UV irradiation and samples exposed outdoors, as well as the monitoring of a case study, a residential building, for a period of over a year. The results demonstrate the effectiveness of the coating and its ability to maintain high performance over time of hydrophobicity and maintenance of the color.

Keywords
Sustainability, Durability, Silossanic coatings, Colour
1 INTRODUCTION

External surfaces of the building envelope, especially in Italy, are mostly finished with plaster, and cover about 80% of the Italian building stock. The market for such products is much diversified and is divided mainly into base plasters, finishing plasters and monolayers, with additions relative to the performance specifications of the outer layer exposed to the atmosphere. The introduction of the EU Regulation No. 305/2011, repealing the Directive 89/106/EEC, has included among the minimum requirements of the construction works, the durability, as part of the seventh requirement “Sustainable use of natural resources”. Thus making mandatory the knowledge and the declaration of the durability of building materials and components, that is their capability to maintain performance characteristics over time above the minimum levels guaranteed. This paper reports the results of a study that examines the issue of the durability of an innovative formulation of decorative coating based on acrylic-siloxane resin. The experimental work, performed on samples subjected to accelerated aging tests and on a selected case-study, was focused on monitoring the most significant performance of the coating: the color, the surface appearance, the water absorption and wettability of the surface. The color, in particular, by means of the class of the appearance requirements [1], not only contributes to the definition of the technological quality of the building, but also improves the environmental context (relevant architectural bodies, planning the color of the facades in the historical centers, etc.). The surface appearance helps to assess the variations in the morphology of the surface due to atmospheric agents; the water absorption and the wettability connote the breathability of the decorative coating [2].

2 THE RESEARCH METHODOLOGY, MATERIALS AND INVESTIGATED PARAMETERS

The research methodology to assess the durability of the acrylic-siloxane coating (ISO 15686 and UNI 11156) provides for the assessment of the reference service life of the component, by means of correlations between accelerated aging tests and natural aging monitoring [3-4]. The tested product can be included among the coatings modifying the shape of the support, referred to as “rasanti” (coatings) in the UNI 8752:1985, applied on supports in reduced thickness between 2 mm and 4 mm, based on acrylic-siloxane resin. The use of such resins confers remarkable water repellency to the coating, according to the classification of the UNI 998-1. The preparatory phase involves the identification of the possible stressing agents that may affect the service life of the component and the decay effects are assumed to cause variations in the characteristic properties. The experimental investigation was carried out by means of accelerated aging tests on samples representing the technical component inside climatic chamber and natural outdoor aging tests, on selected case-study, one building where the product was applied. Finally, the time rescaling was done by comparing the results obtained from the monitoring of the samples differently aged [5]. The measurement of the surface color was carried out, in accordance with the Normal Recommendations 43/93, by using a Konica Minolta Sensing Europe colorimeter CR-410. The observations were made with the Standard Illuminant D65, as defined by the CIE, with an inclination angle of 0°.
The color space chosen to represent the color is to the L*a*b*, better known as CIELAB, defined by the CIE in 1976, based on the brightness, L*, and on the two chromaticity coordinates a* and b* [6]. From the values of the coordinates measured at time zero, it was possible to observe the color difference over time, $\Delta E_{ab}^*$, expressed by the formula:

$$\Delta E_{ab}^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

The measurements\(^1\) carried out both on the prepared samples and on the external surfaces of the case-study, a residential use building located in Palermo, provide for each point the mean of five spots, on a representative area of about 1 square meter. The evaluation of the static contact angle was conducted according to the UNI EN 15802, by means of the measurement of the angle, in degrees, formed between the sample surface and the tangent to the drop of water in the point of contact. The contact angle measurement was carried out by means of a pendant drop tensiometer. The coefficient of water absorption by capillarity was measured according to the methodology described by the UNI EN 1015-18, by measuring the mass of the samples immersed in water over time. The surface morphology was investigated by the use of scanning electron microscope which has allowed evaluating the effects of accelerated aging on the surface of the samples; the simultaneous EDAX elemental microanalysis has provided data on the chemical composition.

### 2.1 Natural aging and the case-study

Monitoring the effects of natural aging on the coating of the building case-study was conducted through the observation of a representative set of points identified on the facades, in the four different exposures. The building (Fig. 1) was realized between October and November 2010, designed by the architect F. Renda.

![Fig. 1. Aerial photography and N-W facade of the case-study](image)

The finishing product of the facades was produced by Hydratite Ltd of Palermo. The building, five floors high, has a square floor plan, with a courtyard, and the facades are oriented

\(^1\) The experimental activities were carried out in the Laboratory of Constructions of the Department of Architecture.
according to the four directions NE, NW, SE and SW. On each side, the representative elements have been identified to be monitored, in particular, the base parts, easily accessible and the terraces, all covered with the same pink coating. The external and the internal surfaces were monitored, so as to assess any color difference found between surfaces parallelly oriented. The results of the colorimetric measurements are referred to a period of three years of natural aging, from October 2010 to October 2013.

2.2 Artificial aging and samples

The samples for laboratory testing, reproducing the solution applied on the surfaces of the building case-study, are constituted by a support in brick and have the final dimensions of 230x300x25 mm, on which the applied coating is about 2 mm thick. The edges of the samples were protected with a layer of waterproofing cementitious mortar to prevent the absorption of water. For the test of artificial aging inside climatic chamber, it was developed and calibrated a cycle of aging [7], from the meteo-climatic data of Palermo [Table 1].

<table>
<thead>
<tr>
<th>PHASE A</th>
<th>Minutes</th>
<th>T (°C)</th>
<th>RH (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rain (autumn season)</td>
<td>75</td>
<td>20</td>
<td>95</td>
</tr>
<tr>
<td>Cold (winter season)</td>
<td>40</td>
<td>2</td>
<td>50</td>
</tr>
<tr>
<td>Hot-humid (spring season)</td>
<td>115</td>
<td>35</td>
<td>87</td>
</tr>
<tr>
<td>Hot-dry (summer season)</td>
<td>70</td>
<td>70</td>
<td>56</td>
</tr>
<tr>
<td><strong>TOTAL</strong></td>
<td><strong>300</strong></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

| PHASE B | | | |
|---------|---------------------------------|--------|
| Hot-humid + UV radiation | 120 | 35 | 87 |

The basic cycle consists of two phases:
- Phase A, 300 minutes (5 hours) long, with alternating rain, cold, hot-humid and hot-dry;
- Phase B, 120 minutes (2 hours) long, in conditions of hot-humid and UV radiation.

The basic cycle was repeated 24 times in the two phases, A and B, defining a total duration of 120 hours (5 days) for Phase A and 48 hours (2 days) for Phase B. Every step of accelerated aging provides twice complete repetition of the two sub-cycles, therefore with a duration of 5x2=10 days for sub-cycle A, and 2x2=4 days for sub-cycle B, respectively, for a total duration of 14 days for each step. The results reported are related to six steps of artificial aging, corresponding to 2016 hours (84 days) inside climatic chamber. The samples were placed in the climatic chamber at approximately 60° slope (Fig. 2) relative to the loading bed. Color measurements, SEM observations, contact angle and water absorption measurements were taken at zero time ($S_0$), and after each of the six steps (from $S_1$ to $S_6$), every time on a couple of samples.
The experimental work was carried out, not only on the pink coating of the case-study, but also on three additional different colors samples (white, yellow and pink salmon), produced by the addition to the base product of mineral pigments, in granular form.

3 RESULTS ANALYSIS

3.1 The colorimetric study
The data collected on samples artificially aged in laboratory were compared with the colorimetric measurements made on the surfaces of the case-study and referred to the values of the colorimetric coordinates of the samples non aged. The following plots refer to the color difference $\Delta E^*_{ab}$ of the surfaces of the case-study monitored after 3 years of natural aging (Fig. 3) and the samples artificially aged (Fig. 4).

Fig. 3. Color difference of the 4 facades of the case-study after 3 years
Based on the comparisons of the results, the following statements can be made:

- Artificial aging inside climatic chamber produced color difference calculated in the range of 1÷4, for the tested four colors of the study, after six steps of artificial aging;
- The pink salmon undergoes the highest color variation, the white and pink the lowest;
- For the case-study, after about 3 years of natural aging, the color of the walls, both the internal and the perimetral external ones, undergoes a variation between 1.19 (NE int) and 2.13 (SW ext);
- The surfaces of the courtyard, less exposed to natural agents, undergo a variation slightly lower than the external ones, 0.25 points on average, demonstrating there is no particular difference over time between the color of the exterior and interior surfaces.

Regarding the time rescaling, performed only for the pink color, whose data are available both by laboratory tests and monitoring of the case-study, it was observed that the decay of the color produced artificially in laboratory, \( \Delta E_{ab} \approx 2.0 \), after the application of the six steps of accelerated aging (S6), is comparable with the natural degradation measured on the external and internal surfaces oriented to SW and NW, after 3 years of natural aging. The internal and external surfaces oriented to NE and SE, whose color difference relative to the non-degraded surface is on average equal to 1.50 points, show that the six steps of accelerated aging are equivalent to about 5 years of natural aging, thus concluding that time rescaling is the following:

- NE and SE surfaces \( \rightarrow 336 \) hours of accelerated aging (1 step) = 10 months of natural aging (6 steps correspond to 5 years);
- SW and NW surfaces \( \rightarrow 336 \) hours of accelerated aging (1 step) = 6 months of natural aging (6 steps correspond to 3 years).

### 3.2 The morphological appearance by means of SEM

The samples analyzed by means of SEM were eight, four of which were non-aged, one for each color - white, yellow, pink and pink salmon - and four in the same colors, taken at the 6th step of accelerated aging. The non-aged samples showed uniform morphological appearance of the surface with compact matrix and the presence of limited size dispersed particles, not exceeding 15-20 \( \mu m \), with irregular shape, visible at 2000x (Fig. 5).
The chemical composition detected is very similar with the main chemical elements present as follows: Carbon (42-45%), Calcium (6-10%), Silicon (9-10%) and Titanium (5-6%). The aged samples show uniform morphological configuration of the surface with less compact matrix, the nearly absence of dispersed particles on the surface and the presence of micro-canyons due to the erosive action induced by the artificial aging cycles (Fig. 6).
The chemical composition detected shows that Carbon decreases drastically due to artificial aging (from 42% to 22%), being reduced by about half, to the advantage of Calcium that considerably increases (from 8% to 20%), due to erosion of the surface film polymer-based, containing Carbon.

3.3 Water absorption
The test, according to the methodology of the UNI EN 1015-18, consists in subjecting cylindrical samples at the early stage of drying in the oven at 60±5°C until reaching constant mass after 24h, then the immersion in 5-10 mm distilled water for a total period of 24 hours, recording, at predetermined intervals, mass variations. The samples were sealed along the side surface and along the non-treated face with a paintable quartz sealant produced by TEKNICA®. To obtain the cylindrical samples, 2.5 cm high and 5 cm diameter, a wet coring was used. The samples subjected to the test were 3 of white color (BN) and 3 of salmon (B1), respectively, at “zero time” and after the 6th step of artificial aging (2016 hours = 84 days). The results obtained from the test of water absorption showed the maintenance over time of nearly constant values, demonstrating the excellent performance of the material. The values of the absorption coefficient of the samples studied ranged between 0.13 kg/(m²·min⁻⁰.⁵) and 0.20 kg/(m²·min⁻⁰.⁵) and according to the UNI 998-1:2004, these samples were classified belonging to the class W2, whose water absorption coefficient has to be less than or equal to 0.20 kg/(m²·min⁻⁰.⁵).

3.4 Contact angle
The contact angle measurements were performed on 4 samples of white and pink color, 2 at “zero time”, 2 at the 6th step of artificial aging (Fig. 7). The results are reported in Table 2.

![Fig. 7. The beginning of the test (L), the image of the drop deposited (center) and the sample after the test (R)](image)

<table>
<thead>
<tr>
<th></th>
<th>Salmon</th>
<th>White</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero time</td>
<td>99°19’</td>
<td>103°01’</td>
</tr>
<tr>
<td>Accelerated aging</td>
<td>95°20’</td>
<td>101°80’</td>
</tr>
</tbody>
</table>

The measurements carried out showed that the surface of the samples is hydrophobic, with a contact angle greater than 90°, for which the water is able to flow on the surface keeping it clean, even in the presence of low slopes. The contact angle did not change significantly due to accelerated aging. It was found a higher value of the contact angle for the white samples compared to salmon ones, 2° on average.
4. CONCLUSIONS

The research showed how siloxane coatings for exteriors develop particularly good performance over time as the capability to maintain very high levels of water repellency, measured means contact angle and the water permeability, although SEM observation show that the environmental aggression erodes the external micrometer film siloxane-based. The color of the finishing layer varies depending on the hue. Pink remains one of the more stable color. The monitoring of the case-study where the product was applied in the pink hue allowed to defining the time rescaling factor, being different depending on the orientation of tested surfaces of the case study, particularly:

- SW and NW surfaces → 144 cycles of accelerated aging ≡ 3 years;
- NE and SE surfaces → 144 cycles of accelerated aging ≡ 5 years.

This different behavior concerns with facades’ maintenance of color showing distinguished variations barely perceptible to the human eye (higher to the value 2, according to the UNI EN ISO 3668:2002), due to the shape of the building and different orientation of facades. Beyond these periods of natural aging, siloxane coating keeps almost unchanged its performance of water repellency and the water permeability.

ACKNOWLEDGEMENTS

The authors would like to thank the Geolab Ltd. in the person of Dr. Angelo Mulone and Dr. Renato Giarrusso for the availability and use of the SEM, the Department of Chemical Engineering, University of Palermo, in the person of Professor Giuseppe Rizzo and Eng. Bartolo Megna for tensiometer tests, Hydratite Ltd in the person of Mr. Carlo Razzanelli and Eng. Eugenio Nuccio for the availability and provision of samples for testing.

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EVALUATION OF RISING DAMP USING INFRARED THERMOGRAPHY

E. Barreira (1), J.M.P.Q. Delgado (1) and A.S. Guimarães (1)

(1) University of Porto, Faculty of Engineering, Civil Engineering Department, Laboratory of Building Physics, Porto – barreira@fe.up.pt; vpfreita@fe.up.pt; jdelgado@fe.up.pt; anasofia@fe.up.pt

Abstract

Moisture is one of the most deteriorating factors of buildings components. To avoid severe degradation, which affects the building durability, it is important to detect moisture in an earlier stage. Using non-destructive techniques (NDT) to detect moisture is very important, especially in buildings with some historical relevance, where more intrusive testing may not be acceptable.

In this work it was analysed the applicability of infrared thermography, a NDT, to assess moisture in building walls caused by rising damp. Laboratory tests were carried out on a full-scale model, consisting on a limestone wall with about 2.00 x 1.80 x 0.20 m³, which was partially immersed in water. Thermograms were taken before the immersion started and during the absorption period of about 3 weeks. Simultaneously, a moisture detector was also used to evaluate qualitatively the moisture content of the wall.

The comparison between the two testing methods showed a good agreement in the results and proved that infrared thermography can be very useful to detect moisture caused by rising damp in an earlier stage.

1 INTRODUCTION

The problem of moisture in buildings has always aroused great interest, since moisture is one of the main causes of buildings pathology. Moisture may cause degradation of building materials and components, compromising their performance concerning durability, mechanical resistance, waterproofness and appearance. It can also cause unhealthy conditions for users, resulting from biological growth and degradation of materials and building components.

Rising damp coming from the ground that, by capillarity, rises through porous materials, is one of the main degradation causes of historical and ancient buildings. The main pathologies caused by rising damp are stained areas at the wall surface due to moisture and mould growth and, in more severe situations, deterioration of plaster and paint due to salts crystallization at the surface or behind the rendering (Figure 1a). Assessing rising damp is therefore essential to improve technical solutions that ensure the building’s durability.
Infrared thermography is a non-contact and non-destructive testing technology that can be applied to determine the surface temperature of an object. The detectors collect infrared radiation emitted by the surface and convert it into a thermal image with the distribution of the body superficial temperature, the thermograms. In this process, each colour expresses a certain range of temperatures (Figure 1b).

![Image](image_url)

Figure 1: a) Degradation of a wall due to rising damp; b) Thermogram of a radiant floor

Two approaches can be used to obtain the surface temperature distributions using infrared cameras: the passive approach and the active approach (Maldague 1993). The thermal images can be analysed qualitatively or quantitatively (Hart 2001). This technology has been applied to buildings for a couple of decades, to evaluate the building performance (Hart 2001). It has been used to detect insulation defects, air leakages and heat losses. Inspection procedures are well defined in standards such as ASTM C 1060-90 (2003), ISO 6781 (1983) and CEN-EN 13187 (1998).

However, procedures to detect moisture in building components using infrared thermography are still under development. It is known that changes in moisture content can be related with changes in surface temperature and can be detected by infrared thermography, due to three physical phenomena:

- Evaporative cooling at the moist area: the evaporation at the surface is an endothermic reaction, which implies a decrease on the surface temperature (Rosina & Ludwig 1999, Moropolou et al. 2002, Barreira & Freitas 2007, Grinzato et al. 2010).
- Reduced thermal resistance: the heat flow through wet materials is higher than through dry materials, which creates a thermal pattern as the surface temperature over the wet material is higher, if the inspection is made from the outside during the colder season. This effect is pushed to extremes when the wetting occurs in thermal insulation materials (Rajewski & Devine 1996, ASTM C 1060-90 2003).
- Increased heat storage capacity of the moist material: the surface temperature over a wet area responds more slowly to a change in the air temperature than the surface temperature over a dry area. Thus, when the whole surface is cooling, wet areas will cool more slowly. During the sunlight day, wet areas will store more solar energy than dry areas, thus, they will cool more slowly during the night (Balaras & Argiriou 2002, ASTM C 1060-90 2003, Lerma et al. 2011).

Moisture content is traditionally assessed using destructive procedures. They require previous drilling the wall to collect samples, which are weighed in the laboratory, immediately after being removed from the wall and after drying. However, water content can
also be assessed using non-destructive techniques, as moisture detectors. These techniques may not be as accurate as the destructive procedures, however, they are very easy to use and they deliver results in real time. Also, they are the only that can be used in buildings with historical relevance.

In this work, the qualitative and passive approaches were used in the thermographic measurements. A moist area in a full-scale wall caused by rising damp was assessed considering the effect of evaporative cooling. Simultaneously, a moisture detector was also used in the same area to evaluate, qualitatively, the moisture content of the specimen. The comparison between the two methods is presented.

2 LABORATORY MEASUREMENTS

2.1 Setting up the test

Laboratory tests were carried out on a full-scale model, consisting on a limestone wall with about $2.00 \times 1.85 \times 0.20$ m$^3$, which was partially immersed in 0.35 m of water. The specimen is composed of 6 blocks with about 0.30 m high with horizontal mortar joints of about 1 cm thick. The absorption period was about 3 weeks. The measurements were performed only in the central area of the specimen as shown in Figure 2. Average temperature and relative humidity in the laboratory during the test period was around 19º C and 70%, respectively.

![Figure 2: Full-scale model and area of the specimen where measurements were performed](image)

The thermography equipment used was Thermo Tracer TH7800 made by NEC Avio Infrared Technologies Co., Ltd (NEC nd). Thermograms were taken before the immersion started and during the absorption period. During the test period the IR camera was always kept in the same position. Emissivity was set to 0.9. This value may not correspond to the real emissivity value of the surfaces under study, however, as the qualitative approach was used, an estimated value of emissivity was considered acceptable. Standard calibration procedures were adopted before taking thermal images, namely, reflection calibration and ambient and background compensation, according to the operation manual of the equipment (NEC nd).

Simultaneously, a moisture detector was also used to evaluate the moisture content of the wall. The moisture detector used was TRAMEX LS made by Tramex, Ltd. This non-destructive equipment operates on the principle that the electrical impedance of a material varies in proportion to its moisture content. The reading displayed by the equipment is a relative scale in percentage, which indicates the greater or lesser signal (lower values indicates lesser signal that corresponds to lower moisture content). To obtain precise moisture
content the equipment must be calibrated (TRAMEX nd). During this work, only the qualitative approach was used as the moisture detector was not calibrated.

To compare the results obtained by thermography with the ones given by the moisture detector, a correlation between the relative scale of the detector results and the thermograms colour scale was established. As lower temperatures correspond to moister areas, because evaporation is more intense, the colder colour was related with higher values of the detector relative scale and the warmer colour with the lower values (Figure 3).

![Figure 3: a) Thermograms colour scale; b) Correlation between the relative scale of the detector results and the thermograms colour scale](image)

Before using the moisture detector it was defined a grid dividing the area to be analysed (Figure 4a). Using the measured values in each point of the grid a graph was created (Figure 4b). It must be clarified that the reading displayed by the equipment is a relative scale in percentage, which indicates the greater or lesser signal (lower values indicates lesser signal that corresponds to lower moisture content).

![Figure 4: a) Grid of the area under study; b) Example of the moisture detector results](image)

During the absorption period, whenever thermograms or measurements with the moisture detector were made, the highest visible moisture level above the water plan was also assessed. This was not always an easy task because the degradation of the specimen surface, due to previous tests, was very severe, as shown by Figure 2 taken before immersion began.

Numerical simulation was used to assess the expected results of rising damp in the full-scale model. It was performed with WUFI®2D-3.3, a transient two-dimensional model for combined heat, air and moisture transport in building components, developed by Fraunhofer IBP – Holzhirchen (WUFI 2013).
2.2 Measurements results

Figure 5 shows the variation in time of the highest visible moisture level. During the first 52 hours after partial immersion, the rise of the visible moisture level was very slow, increasing significantly in the next 220 hours. After that, the stabilization of the maximum value of the visible moisture level began. The delay of 24 hours in the measured visible moisture level points to some kind of hygric resistance to the water uptaken, related with salt crystallization due to previous tests.

Figure 5: Variation in time of the highest visible moisture level above the water plan

Figures 6 to 10 show the thermal images and the results of the moisture detector along 532 hours of absorption. Figure 6 shows the thermal image (Figure 6a) and the moisture detector results (Figure 6b) before immersion began. Both results show that the specimen was dry.

Measurements 52 hours after the imbibition began (Figure 7) show that moisture was rising due to capillarity. The highest visible moisture level above the water plan is about 0.04 m (Figure 5), which correspond to the green isothermal above the water plan (blue line in Figure 7a). The yellow isothermal shows a transition area between the wet and dry surfaces of the specimen. The moisture detector results don’t show any rising damp, what was expectable as the moisture level didn’t achieved the first row of the grid (0.07 m).
Figure 7: Measurements 52 hours after the imbibition began:
a) thermal image b) moisture detector results

At the end of 340 hours after the imbibition began the highest visible moisture level above the water plan is about 26 cm (Figure 5), which corresponds to the highest point of the green isothermal (Figure 8a). Once more, the yellow isothermal shows a transition area between the wet and dry surfaces. The thermal image shows that the moisture level (green isothermal) is higher in the centre of the image and lower near the verticals edges, which was not perceptible visually. That may be related with the effect on evaporation of rising damp of the edges of the specimen or with salt crystallization due to previous tests.

Figure 8b shows the results of the moister detector. The level of the moister area (dark blue) is above 0.28 m and it is almost constant. Comparing the thermal image with the results of the moisture detector, they are not completely in accordance, which is expectable as thermography only detects surface evaporation and the moisture detector assesses inner moist (around 2 cm penetration depth). Also the results of numerical simulation show that at the surface, the moisture level is lower than inside the specimen (Figure 9).

Figure 8: Measurements 340 hours after the imbibition began:
a) thermal image b) moisture detector results
Figure 9: Distribution of relative humidity across the specimen obtained with numerical simulation

Figure 10 shows the results at the end of the test (532 hours of imbibition). Thermal images taken at 340 and 532 hours after the imbibition began are very similar, which indicates a stabilization of the rising damp at the surface. These results are in accordance with Figure 5 in which the measured value of the highest visible moisture level above the water plan is very similar.

One the other hand, the moisture detector results show that, inside the specimen the moisture level is still increasing (Figures 8b and 10b). This may be explained because evaporation is more preponderant than capillarity at the surface, which allows the stabilization of the superficial moisture level. Inside the specimen capillarity is the main driving force and the moisture level keeps increasing.

Figure 10: Measurements 532 hours after the imbibition began (end of the test): a) thermal image b) moisture detector results

3 CONCLUSIONS

Experimental tests showed that thermography can detect moist areas due to capillarity, with the highest visible moisture level corresponding to a green isothermal. Thermography also allows to “see” a moist area corresponding to the transition between the wet and dry surfaces of the specimen, which is not visually detected.

There is an agreement between the thermal images and the results obtained with the moisture detector. The moisture detector points to higher level of rising damp, which was expected as thermography only detects surface evaporation and the moisture detector assesses
inner moist. Numerical simulation results point that at the surface, moisture level is lower than at mid-thickness of the specimen.

In conclusion, this work indicates that thermography ought to be considered as a nondestructive assessment tool for the detection of moisture in porous materials, even when there are no visible signs on the surface. However, further tests must be carried out, namely, it is necessary to establish test procedures and evaluation criteria to avoid misinterpretation.

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EFFECT OF CONFINEMENT AND CONCRETE STRENGTH UPON CRACK BEHAVIOR INDUCED BY CORROSION-PRODUCT EXPANSION

Yasuji Shinohara (1) and Aris Aryanto (2)

(1) Structural Engineering Research Center, Tokyo Institute of Technology, Dr. Eng., Yokohama – shinohara.y.ab@m.titech.ac.jp
(2) Structural Engineering Laboratory, Institute Technology of Bandung (ITB), Dr. Eng. Bandung – aris_aryanto@yahoo.com

Abstract

Corrosion has known as a major problem on deterioration of RC structures because it may induce cracking of concrete cover as well as bond deterioration. Most of studies on the cracking behaviors have used single bar in a concrete prism or a cylinder and absence of stirrup bars, which may not represent the real boundary condition of reinforced concrete structures. The main objective of this paper is to investigate the influence of stirrup confinement, longitudinal bar diameter, concrete strength and corrosion rate on the cover crack propagation. Six beams were prepared, and designed to experience a specified corrosion by performing an accelerated corrosion through the electrochemical process. The current flowed on each bar and the strains on the stirrups were recorded using data logger. The corrosion-crack behavior was frequently observed using digital microscope with the resolution of 0.01mm. Furthermore, finite element analysis was performed to study the internal cracking behaviors and the bond-slip relationship between concrete and stirrups.

1 INTRODUCTION

Cracking of concrete cover due to corrosion product expansion of a steel bar is one of the main factors in determining durability performance and service life of RC structures. Although number of experimental studies evaluating the cracking behavior of concrete under corrosion of reinforcement for past years, most of studies used a single bar in concrete prism or cylinder and absence of transverse bars (1). The corrosion of steel induces not only cracking of concrete cover, but also stressing on the transverse bar. This may reduce the residual shear capacity of RC structures and it has not been widely explored in the previous studies.

The present study is intended to explore the influence of actual confinement provided by surrounding concrete and transverse bar on the cover crack behaviors induced by corrosion.
product expansion of steel bar, and to obtain a general tendency on crack propagation, crack width and stress behavior of transverse bar.

2 MECHANICAL PROPERTIES OF CORROSION PRODUCTS

Figure 1 illustrates a deformation of corrosion product around corroded steel and ring tension induced by corrosion expansion pressure. When the corrosion product can expand to free-increase radius $a$, so there is no expansion pressure will be generated. However, considering the compaction effect of corrosion product due to confinement of surrounding concrete as mentioned by Lundgren \(^2\), the free-increase radius will reduce to the actual radius $u_{cor}$. By assuming corrosion penetration, $x$, and volume of the rust relative to corroded steel, $v$, the radius of free increase, $a$, can be calculated by equating $v$ times corroded steel with the corrosion product in the Figure 1.

$$a = -r_b + \sqrt{r_b^2 + (v-1)(2r_b x - x^2)}$$

(1)

The value of relative volume of the rust to corroded steel, $v$, greatly depends on corrosion product formation. Common values used in analysis were taken between 2.0 and 4.0. The strain of corrosion product layer, $\varepsilon_{cor}$, due to compaction effect is defined as follows \(^2\)

$$\varepsilon_{cor} = \frac{u_{cor} - a}{x + a}$$

(2)

Evaluating the deformability of corrosion products around corroded bar, a nonlinear relation \(^2\) has been proposed between internal pressures, $p$ developing around bar and corresponding strain of corrosion product layer, $\varepsilon_{cor}$ in radial direction may be expressed as

$$p = 7000\varepsilon_{cor}^\gamma \text{ (N/mm}^2)$$

(3)

3 CORROSION TEST OF RC BEAMS

The proportion of concrete mixture used in a series of accelerated corrosion test is shown in Table 1. The beam specimens were cured for 28 days before accelerated corrosion test. The specified compressive concrete strength of 28 days was 22 N/mm\(^2\) and 49 N/mm\(^2\) for Fc24 and Fc48 respectively. The test variables include the transverse bars ratio and configuration, diameter of longitudinal bar and concrete strength (Table 2 and Figure 2). The non-corroded
transverse bars were used by covering with vinyl tape to protect gages during accelerated corrosion of longitudinal bars. The tank containing 3% of NaCl solution was put below the specimens. The solution penetrated to the concrete through water sponge. Moreover, a constant 10 Volt was given and the current was monitored and recorded using data logger (see Figure 3(a)). The longitudinal bars were corroded up to approximately 6% of weight loss where cover crack width estimated larger than serviceability limit (e.g. 0.3-0.5mm). To accurately determine the corrosion rate of reinforcing bar, the steel bars were removed from the concrete, chemically and mechanically cleaned, and measured the weight loss.

Table 1 Mixture proportions

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>w/c (%)</th>
<th>Cement (kg/m³)</th>
<th>Water (kg/m³)</th>
<th>Sand (kg/m³)</th>
<th>Gravel (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fc24</td>
<td>74</td>
<td>257</td>
<td>190</td>
<td>830</td>
<td>979</td>
</tr>
<tr>
<td>Fc48</td>
<td>46</td>
<td>392</td>
<td>180</td>
<td>807</td>
<td>979</td>
</tr>
</tbody>
</table>

Table 2 List of test variables for specimens

<table>
<thead>
<tr>
<th>Designation</th>
<th>$\sigma_b$ (N/mm²)</th>
<th>L. bar</th>
<th>$p_{w}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fc24-LB4-Pw0</td>
<td>22</td>
<td>4D19</td>
<td>0%</td>
</tr>
<tr>
<td>Fc24-LB4-Pw015</td>
<td></td>
<td>4D19</td>
<td>0.15%</td>
</tr>
<tr>
<td>Fc24-LB4-Pw03</td>
<td></td>
<td>4D19</td>
<td>0.3%</td>
</tr>
<tr>
<td>Fc24-LB4-Pw03M</td>
<td></td>
<td>4D19</td>
<td>0.3%</td>
</tr>
<tr>
<td>Fc24-LB3-Pw03</td>
<td></td>
<td>3D22</td>
<td>0.3%</td>
</tr>
<tr>
<td>Fc48-LB4-Pw03</td>
<td>49</td>
<td>4D19</td>
<td>0.3%</td>
</tr>
</tbody>
</table>

$\sigma_b$: Concrete strength  
L. bar: Longitudinal bar  
$p_{w}$: transverse bars ratio

Figure 2 Typical specimen configuration and gage attachment

Figure 3  (a) Overview of accelerated corrosion test  (b) Digital microscope measurement
The cracks on the surface were visually observed and the crack width at certain locations was frequently measured using digital microscope having a resolution of 0.01mm to monitor the crack width increment (Figure 3(b)). To measure the strain development on transverse bar due to corrosion cracking, three gages were installed at each transverse bar as shown in Figure 2. The strain on transverse bar was recorded by data logger in 60 minutes increments.

4 CORROSION TEST RESULTS

4.1 Corrosion rate

Table 3 shows measured corrosion loss of each bar. A different corrosion loss of each bar (i.e. T, CT, CB and B) on each specimen was obtained although the measured output current of each bar was relatively similar. The higher corrosion loss was obtained from the corner bar (T and B) having two directional surfaces and the largest one was particularly located at top in casting (T). This can be because the bar located at top of concrete casting tends to have higher porosity than at bottom casting due to settlement of fresh concrete (Kurumisawa et.al 3)).

The appearance of localized corrosion (pitting) can be seen after removing the bar from the concrete. The sign of localized corrosion and more severe corrosion mostly occurred at the side of steel bar which facing chloride solution penetration (Figure 3(a)). This could be attributed to current flow and diffusion chloride solution only came from one direction.

### Table 3 Corrosion in weight loss (%)

<table>
<thead>
<tr>
<th>Designation</th>
<th>Bar Location</th>
<th>i\text{corr} (predicted) mA.hr/cm²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>T</td>
<td>CT</td>
</tr>
<tr>
<td>Fc24-LB4-Pw0</td>
<td>10.6</td>
<td>4.7</td>
</tr>
<tr>
<td>Fc24-LB4-Pw015</td>
<td>8.9</td>
<td>4.9</td>
</tr>
<tr>
<td>Fc24-LB4-Pw03</td>
<td>8.2</td>
<td>4.4</td>
</tr>
<tr>
<td>Fc24-LB4-Pw03M</td>
<td>7.4</td>
<td>4.5</td>
</tr>
<tr>
<td>Fc24-LB3-Pw03</td>
<td>7.4</td>
<td>3.9</td>
</tr>
<tr>
<td>Fc48-LB4-Pw03</td>
<td>6.6</td>
<td>4.0</td>
</tr>
</tbody>
</table>

Note: number in the parenthesis shows the equivalent of accumulative current density estimated by Faraday’s Law in mA.hr/cm²

4.2 Corrosion crack pattern

The first crack (crack initiation) on the concrete surface was visually observed within a few days after accelerated corrosion being started for all specimens. The crack was initiated at bottom side of beams and mostly located near beam edge or corner bar. The crack then propagated and formed a continuous crack approximately parallel to the longitudinal bar. The crack patterns at final corrosion test and the crack width up to corrosion loss of 6% are shown in Figure 4. A relatively close spacing of longitudinal bar seems to promote the corrosion crack among bars to propagate horizontally connecting each longitudinal bar. This can be confirmed from the stain on the concrete after removing the bottom cover.

The relations between observed maximum crack width and average corrosion penetration are also presented in Figure 4. The average corrosion penetration was determined from means value of corrosion depth penetration for all longitudinal bars in one specimen. The corrosion penetration at first concrete cracking is around 30-60 micrometers for all specimens. For the
Fc48-LB4-Pw03 specimen, it clearly shows that the crack width is larger than specimens having normal concrete strength at similar corrosion levels. This trend can be attributed to lots of corrosion products localized in a crack because of lower porosity of high strength concrete. Moreover, the amount of pores greater than 1μm² is also increase for higher w/c ratio, as reported by Kurumisawa et.al 3). Because of that, the corrosion product may be difficult to penetrate or to diffuse in the cement paste for high strength concrete. Therefore, it can generate higher expansion pressure than normal strength concrete.

As shown in Figure 4, the effect of transverse bar seems insignificant on the cover crack width growth. This can be attributed to the use of vinyl taping may reduce the bond between transverse bar and concrete so that the crack are more concentrated at corner bars.

### 4.3 Corrosion-induce transverse bar strain

The strain increment of transverse bar greatly depends on cracking behavior due to corrosion expansion and the bond interaction between transverse bar and concrete. Figure 5 shows the relation between corrosion of longitudinal bar and measured strain of transverse bar at the middle of bottom leg (SC). For specimen with normal concrete strength and transverse bar ratio of 0.15-0.3%, the observed strain on the transverse bar was relatively small lower
than 200µm. However, a higher strain at transverse bar was observed for beam specimen with high strength concrete, and maximum strain reached to 700µm. If using normal strength of transverse bars e.g. 295 N/mm², it then will reduce almost 50% of its capacity. The observed stirrup strain from the test is not so large compared to the estimated strain by assuming a complete bond. It can be due to the bond between steel bars and concrete is not perfect due to vinyl taping. Moreover, corrosion of transverse bar also will reduce the gross area of transverse bars. Therefore, it may significantly reduce the residual shear capacity of RC structures under service loading or seismic loading.

5 NONLINEAR ANALYSIS FOR CORROSION CRACK BEHAVIOR

5.1 Modeling approach

For the sake of simplicity, two-dimensional (2D) analysis with plane stress elements having a thickness of exposed length (400 mm) were used to model the concrete and only a half of beam sections was an object of analysis. The longitudinal bars were modeled as holes and the transverse bars were modeled using truss elements (Figure 6). To simulate corrosion-product expansion, the expansion pressure around corroded bar was applied by internal pressure. The internal pressure of corner bars which were greatly corroded were assumed to be two times of middle bars in order to obtain a crack pattern from tests.

The mechanical properties of concrete, transverse steel and bond-slip relationship are shown in Figure 7. The concrete behavior under compression was assumed to be linear since the concrete behavior in this analysis is dominantly governed by tensile cracking at low levels.
of compressive stresses. Concrete crack initiates when its maximum tensile stress reaches the tensile strength. After cracking, bi-linear tension softening model was adopted. The transverse steel used a bi-linear model without strain hardening. Because no available test data on bond-slip relationship for transverse bar covered by taping, the maximum bond stress and slip at maximum bond stress were determined according to CEB-FIP Model code as plain bars.

5.2 Cover crack propagation

Figure 8 shows crack strain behaviors obtained from FE analysis at the first cracking of the surface. The expansive pressure $p$, maximum crack strain $\varepsilon_{cr,max}$, and maximum transverse bar stress $\sigma_{s,max}$, are also provided in the figure. The depth penetration $x$, and corrosion weight loss $w$, were calculated using equation (1) to (3) with the relative volume of the rust to corroded steel, $v = 2$. As shown in Figure 8, the crack initially tends to form a horizontal crack pattern connecting each longitudinal bar, and then mostly propagates to concrete surface near the corner beam indicated by higher crack strain. This crack pattern shows a good agreement with the experimental results where cracks mostly located near corner bar (Figure 4). From the analytical results, when the cracks reach the concrete surface the corrosion penetration is around 30 to 50 micrometers. This shows that the corrosion penetration to induce cracking on concrete surface is relatively low, and is also reported by other researchers (1), (4).

5.3 Cover crack width

A comparison of the total crack width on the concrete surface in the experimental and the analytical results are shown in Figure 9. The total crack width from the experiment was determined as the summation of crack width at one section of the exposed length. Meanwhile the total crack width from FE analysis was determined by the relative nodal displacement of two corner beam section because concrete tensile strain is generally negligible. For specimens
with normal concrete strength, the analytical result shows a good agreement with the test by assuming $v = 2$. However, for specimens with high strength concrete, it generates an underestimate prediction compared to experimental results. This indicates that a large expansion ratio is required for high strength concrete because only a small amount of corrosion products can penetrate to the lower porosity. The ratio of volume increase of $v = 2.5$ to $3$ gives a good agreement with test results.

6 CONCLUSIONS

- Corrosion rate is not uniformly distributed among longitudinal bars in a beam and over perimeter of bar caused by the different of bar location in casting direction, the direction of chloride diffusion, and the supply availability of water and oxygen.
- Both experimental and analytical results indicate that crack initiated in concrete surface occurs in relatively small amount of corrosion penetration around 30 to 60 micrometers.
- Transverse bars have an insignificant effect on the cover crack width growth because the use of vinyl taping may reduce the bond between transverse bar and concrete.
- Concrete strength has a significant influence on the crack width growth rate and the stress increase of the transverse bars because of the difference of porosity in hardened cement paste. This stress may reduce the residual shear capacity of RC structures.

ACKNOWLEDGEMENTS

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REFERENCES

A NEW TEST METHOD TO EVALUATE THE RESISTANCE OF CEMENTITIOUS MATERIALS EXPOSED TO BIOGENIC ATTACK IN SEWER NETWORKS

Matthieu Peyre Lavigne (1,3,4), Alexandra Bertron (2)*, Arnaud Cockx (1,3,4), Jean-Noël Foussard (1,3,4), Gilles Escadeillas (2), Etienne Paul (1,3,4)

(1) Université de Toulouse; INSA, UPS, INP; LISBP, 135 avenue de Rangueil, Toulouse, France mpeyrela@insa-toulouse.fr
(2) Université de Toulouse; INSA, UPS ; LMDC, 135 avenue de Rangueil, Toulouse, France bertron@insa-toulouse.fr
(3) INRA, UMR792 Ingénierie des Systèmes Biologiques et des Procédés F-31400 Toulouse, France
(4) CNRS, UMR5504, F-31400 Toulouse, France

Abstract

An innovative test method for evaluating the biodeterioration of cement linings in sewer-like conditions was designed and implemented. The development of the test, involving a lab-scale pilot design, was based on (i) identification of the limiting phenomena, (ii) analysis of the sulfur cycle in the condensate phase, (iii) experimental identification by mass balance of a substitute sulfur substrate for microorganisms, and (iv) enhancement of the local biological acidification by control of the environmental conditions applied to the wall of various pipes. This step ensured, with a non-specified inoculum (activated-sludge microbial consortium), immediate sulfur-oxidizing activity, its intensification and its stabilization.

Preliminary work enabled thiosulfate to be identified as an intermediate substrate for biological acidification, by comparison between a biotic pipe and an abiotic pipe covered with CAC lining. Then, CAC or BFSC cement mortars were evaluated under the same biotic conditions to test the pilot configuration. The resistance of the cement linings during the test was evaluated by monitoring the pH and the Ca and Al concentrations of the leaching solutions. At the end of the test, the physical, chemical and microstructural changes of the cement mortars were investigated using XRD, SEM coupled to EDS and EPMA analyses in order to identify the mechanisms of alteration of the matrices.

1 INTRODUCTION

In sewer networks, severe deterioration of cementitious materials is primarily related to the production of H$_2$S in the headspace of the sewers. Subsequently, in a moist environment in contact with cementitious materials, chemical oxidations of H$_2$S lead to a variety of reduced sulfur compounds (elemental S$^0$, thiosulfate S$_2$O$_3^{2-}$, etc.) [1-4]. Sulfur-oxidizing bacteria (SOB) are able to grow by oxidizing these reduced sulfur compounds, leading to a biological acidification of the local medium in contact with the cementitious material and to a local
production of sulfate ($SO_4^{2-}$) as an end product [2,4-5], linked to the migration of the acid and the sulfate produced by the microorganisms into the depths of the materials [4,6]. During this migration, secondary expansive precipitation occurs, depending on the cementitious matrix composition. The first step involves the formation of gypsum CaSO$_4$.2H$_2$O [7], and the second step the reaction of the gypsum and the aluminium compounds to form ettringite, CaO.Al$_2$O$_3$.3CaSO$_4$.32H$_2$O [7]. Inside the porous media, the precipitation of expansive compounds such as gypsum and ettringite leads to local swelling that causes cracks and increases the local porosity, thus allowing accelerated penetration of the aggressive agents [6-8].

Today, standard NF EN 598 for ductile cast iron pipes imposes chemical testing to evaluate the resistance of the cementitious linings to aggressive environments. Several studies [3-8] have indicated the limits of the ability of these tests to evaluate the resistance to biogenic acidification (lack of biological interactions with the material – total immersion of the material in an acid solution).

In this context, an innovative test method to evaluate the biodeterioration of cement linings in sewer-like conditions is proposed. Its development is based on: (i) implementation of major phenomena involved in the biodeterioration of sewers; (ii) mass balances to quantify these transformations; (iii) a soluble reduced sulfur substrate to accelerate the rate of biogenic transformations; and (iv) an easily available inoculum with high microbial diversity.

The study aims to evaluate the test design in terms of biodeterioration efficiency and of how representative the transformations are of real industrial products (at the scales of (i) biological acidification and biological sulfur transformation, and (ii) the cementitious linings). Two types of cementitious linings with different behaviours were subjected to intensive biogenic degradation under the same controlled environmental conditions for 107 days. Afterwards, the mineralogical and chemical changes of the linings were investigated.

2 MATERIALS AND METHODS

2.1 Experimental set-up for the tests

The pilot was composed of two vertical pipe-reactors in parallel, each being mainly composed of one segment cut from a real sewer pipe (ductile cast iron pipes with centrifugally cast mortar linings based on cementitious materials) (Figure 1). The pipe segments were 200 mm long and 80 mm in internal diameter (exposed surface: 0.05 m$^2$). The initial protective linings were around 6 mm thick.

To represent the condensation and flow of water at the top of the pipe in real sewers, a constant flow of a mineral solution was fed into the reactors by trickling (50 ml/h). Because of the vertical position of the pipe segments, the water ran down the pipe walls by gravity. To limit preferential paths for the trickling solution, the orientation of the inlet cone was changed by 90 degrees every day. The feeding solution at the inlet of the pipe was composed of NH$_4$Cl, (NaPO$_3$)$_3$, MgCl$_2$.6H$_2$O, FeCl$_2$.4H$_2$O, MnCl$_2$.4H$_2$O, and a solution of trace elements (B- Mo- oxides, Co, Zn, Cu, Ni). Sulfur was provided by dissolving Na$_2$S$_2$O$_3$ in deionized water.

The pipe walls were inoculated with a consortium prepared from an activated sludge (sampled in a treatment plant near Toulouse, France). A previous study of CAC linings with an activated sludge consortium under thiosulfate environment showed that, after 240 days of test, around 50 % of the populations identified were sulfur-oxidizing bacteria.
As a control of the biological activity on the cementitious materials, a CAC specimen was tested in the same conditions, without inoculation, and was run abiotically for 107 days, by nitrogen starvation in the feeding solution.

(1a) pipe segment with BFSC lining
(1b) pipe segment with CAC lining
(2) thiosulfate and phosphorous feeding solution
(3) nitrogen and other nutrients feeding solution
(4) aerated feeding solution
(5) peristaltic pumps
(6a) bottle to collect the leaching solution from the BFSC lining
(6b) bottle to collect the leaching solution from the CAC lining

Figure 1: Experimental set-up based on two segments of real sewer-pipes.

2.2 Chemical analyses of the leaching solutions

During the test, to evaluate the sulfur-oxidizing activity, the sulfur transformations were analysed by measuring the concentration of thiosulfate as the substrate (S\textsubscript{2}O\textsubscript{3}\textsuperscript{2-}), and of sulfate (SO\textsubscript{4}\textsuperscript{2-}) as the end product in the leaching solutions, by anionic chromatography (DIONEX: IC25, IonPacTM AS19). The dissolution of the cementitious materials was analysed by measuring the calcium concentrations in the leaching solution by cationic chromatography (DIONEX: ICS 2000, IonPac CS12). The pH of the leaching solutions was measured with a glass electrode (SCHOTT).

2.3 Cementitious material analyses

At the end of the exposure period (107 days), the segment pipes were first sawn with a hand grinder to collect 5 x 5 cm\textsuperscript{2} specimens (ductile cast iron envelope + mortar lining).

The mortar linings were then easily detached from the ductile cast iron tube, with no associated deterioration. Afterwards, the mortar lining pieces were sawn more accurately with a diamond saw to prepare 1 x 1 cm\textsuperscript{2} specimens intended for microstructural testing.

Chemical modifications of mortars were analysed using an electron microprobe (CAMECA SX 50). The measurements were performed according to [9-11]. These analyses were performed on two specimens. A control specimen (sound lining) was also analysed. Mineralogical analyses using X-ray diffraction were completed on the specimens ( E D 000) according to [9-11]. Water intrusion porosity tests were performed on the control specimens according to [12]. Microstructural observations and chemical analyses of exposed and control specimens of mortar linings were performed using a scanning electron microscope (Jeol JSM-6380LV) fitted with an EDS detector (Rontec XFlash® 3001).

3 RESULTS AND DISCUSSION

3.1 Characterization of control specimens

Figure 2 shows SEM observations (BSE mode) of cross sections of BFSC and CAC linings (control specimens). The linings, 5.6 mm thick, show a heterogeneous, double-layered structure, caused by the production process (centrifugation): (i) the inner layer (in contact
with the biofilm), 1.6 mm thick, is a paste layer and (ii) the outer layer (in contact with the ductile cast iron tube) is a mortar layer (4.0 mm thick).

The CAC mortar layer shows more compact granular packing with smaller aggregates than BFSC mortar. For both linings, the paste layer comprises a less dense layer (300 or 400 µm thick) probably linked with young age desiccation and the superficial carbonation.

![Figure 2: SEM observations (BSE mode) of cross sections of control specimen mortar linings, 1) BFSC and 2) CAC.](image)

Because of the heterogeneity of the specimens, the porosity of control specimens was measured (i) on the paste layer (ii) on the mortar layer and (iii) on the total specimen for each type of lining (Table 1). The samples in each zone were taken by sawing with a diamond saw.

Table 1: Water porosity of BFSC and CAC lining specimens.

<table>
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<tr>
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<th>BFSC</th>
<th>CAC</th>
</tr>
</thead>
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<tr>
<td></td>
<td>Paste layer</td>
<td>Mortar layer</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>33.8 ± 1.4</td>
<td>21.0 ± 1.0</td>
</tr>
</tbody>
</table>

The porosity of CAC lining was much lower than that of BFSC. In particular, the paste layer (in contact with the biofilm) of the BFSC lining was twice as porous as the CAC lining.

The composition of both cement pastes used for lining production is given in table 2 (based on EPMA profiles of control specimens). The oxide percentages were corrected by the porosity measurements presented in table 1. Based on these values (Tables 1 and 2) the calcium content in the first layers of the materials could be calculated as references for evaluating the dissolution of Ca during the test for both cementitious linings. Considering the first 200 µm, the Ca content was 5% higher for the CAC lining than for the BFSC lining (the higher content of BFSC paste was offset by the higher porosity of the initial material).

Table 2: Compositions of oxides of the cement paste for BFSC and CAC linings (based on mean values obtained from EPMA profiles and porosity measurements).

<table>
<thead>
<tr>
<th>BFSC paste composition of oxides</th>
<th>CaO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>MgO</th>
<th>SO₃</th>
<th>K₂O</th>
<th>TiO₂</th>
<th>Na₂O</th>
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<td></td>
<td>52.54%</td>
<td>26.98%</td>
<td>7.97%</td>
<td>1.21%</td>
<td>3.20%</td>
<td>4.52%</td>
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<td>0.10%</td>
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</table>

<table>
<thead>
<tr>
<th>CAC paste composition of oxides</th>
<th>CaO</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>Fe₂O₃</th>
<th>MgO</th>
<th>SO₃</th>
<th>K₂O</th>
<th>TiO₂</th>
<th>Na₂O</th>
<th>Tot</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>37.86%</td>
<td>5.80%</td>
<td>43.76%</td>
<td>2.04%</td>
<td>0.25%</td>
<td>0.08%</td>
<td>0.09%</td>
<td>2%</td>
<td>0.05%</td>
<td>91.94%</td>
</tr>
</tbody>
</table>
3.2 Biogenic acid production and dissolution of cementitious materials during the test

Figure 3 presents the results of the analyses of the leaching solutions for the abiotic CAC lining, and for both inoculated linings (CAC and BFSC): pH of the leaching solution, sulfate and calcium concentrations in the leaching solution. For all the systems, the sulfur mass balances were achieved by measurement in the leaching solution (uncertainty: 10%) and indicated that only a low proportion of sulfate was able to penetrate the cementitious materials if this phenomenon occurred.

Compared to the results obtained in abiotic conditions, Figure 3 confirms the importance of the biological activity for the acidification of the environment, and thus for calcium dissolution.

During the first 60 days, for both inoculated materials, the pH evolutions of the leaching solutions were similar but, at the same time, the sulfate production was higher for the BFSC linings than for the CAC linings, indicating a difference in the sulfur oxidizing activity developed in contact with the material. This difference was correlated with a more intense Ca dissolution for the inoculated BFSC lining than for CAC lining. Thus a similar pH of the leaching solution revealed different behaviour of the two materials.

After 60 days, the pH of the leaching solution for the test with the BFSC lining became unstable because of the significant dissolution of alkaline materials. The pH instabilities had a direct negative impact on the SOB activity (sulfate production decreased). Consequently, after 100 days, the amount of sulfate produced reached the same intensity for both linings.

3.3 Characterization of linings tested

Figure 4 shows cross sections of BFSC linings exposed to the test conditions. The top of the figure (1 and 2) corresponds to the inoculated surface.
Micro-cracks are present throughout the specimens, in both the paste and mortar layers. At 300 µm depth from the surface, a thicker crack, almost parallel to the surface is notably visible (figure 4.2). The location of the crack matches the transition between (i) zone a, a partly decalcified layer and (ii) zone b (the rest of the specimen) enriched in sulfur. Observation of the paste layer after abrasion of the first 400 µm confirmed the generalized micro-cracking (figure 4.4).

The EDS analyses showed the elemental composition of ettringite almost everywhere in the paste layer and notably in the porosity of the matrix and in the cracks. Elemental composition of gypsum was also detected at the beginning of the mortar layer, at the aggregate/paste interface and observation of the micro-crack network after abrasion of the first 400 µm of the decalcified zone.

Micro-cracks are present throughout the specimens, in both the paste and mortar layers. At 300 µm depth from the surface, a thicker crack, almost parallel to the surface is notably visible (figure 4.2). The location of the crack matches the transition between (i) zone a, a partly decalcified layer and (ii) zone b (the rest of the specimen) enriched in sulfur. Observation of the paste layer after abrasion of the first 400 µm confirmed the generalized micro-cracking (figure 4.4).

The EDS analyses showed the elemental composition of ettringite almost everywhere in the paste layer and notably in the porosity of the matrix and in the cracks. Elemental composition of gypsum was also detected at the beginning of the mortar layer, especially at the interface between the aggregates and the paste layer.

Figure 5 shows observations of cross sections of CAC linings exposed to the test conditions. The top of figures 5.1 and 5.2 corresponds to the inoculated surface. No crack is visible anywhere in the specimen. The paste shows high density and compactness except in a layer of 100 to 200 µm (in contact with the biofilm), which is very porous, decalcified and slightly enriched in sulfur (see the EDS spectrum and the associated values for main oxides in figure 5.3 compared to the values presented in Table 2 for a sound CAC lining).
3.4 EPMA chemical composition profiles

The results of EPMA analysis of BFSC and CAC linings exposed to the test, according to the distance to the inoculated surface are shown in Figure 7 and Figure 7 respectively.

For BFSC lining, the paste layer (zones a and b) and the mortar layer (zone c), as defined in Figure 4, are reported. The average $SO_3$ content of the control specimen (4.2% by mass) is represented by the lower dotted line. The upper dotted line represents the average CaO content of the control specimen. Zone a, 400 µm thick, is partly decalcified and the $SO_3$ content tends to zero at the surface. $Al_2O_3$ and $SiO_2$ contents are maintained in this zone. Sulfur enrichment was observed, (i) at the end of the decalcified zone (zone a) and at the beginning of zone b, at the location of the main crack observed in Figure 4, and (ii) deeper in the mortar layer (zone c), corresponding to the deeper cracks observed in Figure 4-3 at the aggregate/cement paste interface.

The results of EPMA analysis of the CAC lining tested, according to the distance to the inoculated surface are shown in Figure 7. The paste and mortar layers and also the gel zone, as defined in Figure 5, are reported. The surface zone, 100-200 µm thick, is decalcified, enriched in sulfur (up to 1.7% of $SO_3$ vs. an average of 0.08 % in the rest of the paste) and contains almost exclusively alumina. In the rest of the specimen, the composition of oxides is almost constant and matches the composition of the control specimen.

3.5 Mineralogical characterization by XRD (figures not shown)

The surface of the BFSC specimen was mainly amorphous. In zone a, calcite precipitation was observed, and peaks of anhydrous residual grains of $C_3S$ and merwinite (component of slag) could be seen. In the paste layer of zone b, ettringite had precipitated. In the mortar
layer, the presence of intense peaks of aggregates did not allow the crystallized phases of the cement paste to be adequately observed and identified.

The core of the paste layer of CAC lining showed typical anhydrous (CA, $C_{12}A_7$) and hydrated ($C_3AH_6$, AH) crystallized phases of CAC matrix. In the layer in contact with the biofilm, the structure was mainly amorphous with precipitation of $AH_3$ and dissolution of $C_4AH_6$ and of the other crystallized phases.

### 3.6 Discussion

The efficiency of a biodeterioration test is defined by, (i) the representativeness of the transformations involved in the test compared to the transformations observed in the real environment, (ii) the reproducibility of the transformations involved in the test (and so the control of the biological system), and (iii) the acceleration of the deterioration phenomena.

The results confirmed the different behaviour of BSFC lining compared to CAC lining under biogenic attack in a sewer environment [3-5-8-13].

For the BFSC lining, after the time of exposure, an amorphous surface was observed (300-500 µm thick) because of a low surface pH due to biogenic acid production. Massive and generalized cracking of the paste and the mortar layer were observed. In the paste layer, in accordance with some other studies [6-7-8], the detection of expansive secondary precipitation as ettringite explains this cracking phenomena. In the deteriorated sample, little gypsum was observed compared to ettringite. However, the low portlandite content of the BFSC lining used in this study could explain the small amount of gypsum formed. On the other hand, as ettringite formation needs preliminary gypsum formation [6], these observations may, interestingly, confirm a description of biogenic attacks on BFSC-based mortars in two steps: firstly, a surface phenomenon with biological production of acid and sulfate and, secondly, the penetration of sulfate into the material with local precipitation of gypsum and/or ettringite [3-6-7-8], depending on the proportions of the composition of the lining under test.

For the CAC lining, in the 60 first days of the test, the biogenic acid production was weaker than for the BFSC lining, (around 20%) quantitatively confirming the effect of CAC paste on the implantation of the biological SOB activity [5-8]. After the time of exposure, an amorphous inner layer (100 µm thick) was detected. This layer was composed of alumina gel ($AH_3$). It was enriched with sulfur and had no detectable mineralogical structure, probably because of absorbed sulfate and/or sulfur substrate. As reported in some studies [8], no penetration of sulfur compounds, so no secondary precipitation, was detected deeper than the inner layer. This may have been due to: (i) the protection provided by the alumina gel formed during the biogenic attack ($AH_3$) [13], (ii) unfavourable thermodynamic conditions despite the release of calcium from the gel layer.

Although the reproducibility of the test has not yet been evaluated, the work without $H_2S$ as a sulfur source guarantees the composition of the liquid medium applied at the inoculated surface, and so the rate of biological reactions. The accelerated factor of the tests must be evaluated also (by comparison with in-situ measurements). Nevertheless, this study shows that the discrimination between two materials is possible in 15 weeks.

### 4 CONCLUSION

An innovative testing method for real sewer-pipes was designed without hydrogen sulfide ($H_2S$) as a sulfur source. $H_2S$ is a toxic gas and a compound with high abiotic reactivity, so its
use is dangerous and makes safety controls necessary. It also limits dynamical measurements and the validation by sulfur mass balances. In this study, thiosulfate was used as a model sulfur substrate. The testing method, safe in the operating conditions described, is representative of the biodeterioration observed in sewer networks. After 15 weeks of exposure, under a controlled environment, the test configuration ensured the development of a sulfur oxidizing activity at the surface of the materials and thus the discrimination between two types of cementitious linings (BFSC and CAC). The better behaviour of the CAC cement against biogenic attacks was confirmed by analyses of the local chemistry and the microstructure of the linings after exposure to similar biological conditions. Nevertheless, the analyses of the materials also revealed the importance of the stratification and the initial porosity of the product in the phenomenon.

Further tests will be carried out to assess the reproducibility of the test method.

ACKNOWLEDGMENTS

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REFERENCES

DURABILITY OF QUATERNARY CEMENTS CONTAINING CALCAREOUS FLY ASH, SLAG AND LIMESTONE

Pawel T. Durdziński (1), Mohsen Ben Haha (2) Karen L. Scrivener (1)

(1) Laboratory of Construction Materials  Ecole Polytechnique Fédérale de Lausanne – pawel.durdzinski@epfl.ch; karen.scrivener@epfl.ch
(2) HeidelbergCement Technology Center GmbH, Leimen – mohsen.ben.haha@htc-gmbh.com

Abstract

The durability of blends containing two calcareous fly ashes of different chemical and mineralogical compositions was studied with respect to sulfate and chloride ingress in binary, ternary and quaternary systems (Portland clinker, fly ash, slag, limestone). The results show that durability can change substantially depending on the composition of fly ash and that benefits can be obtained from combining fly ash with slag or limestone.

The differences in durability were compared to phase compositions measured with XRD Rietveld and TGA. The results show that using a calcareous fly ash with around 4 wt.-% C₃A does not mitigate expansion under sulfate attack, possibly due to high content of monosulfate. In this case, adding limestone promoted monocarbonate formation instead and significantly decreased the expansion. Chloride ingress can be successfully limited by using calcareous fly ashes or slag or their combinations. Limestone addition can have different effect depending on the system where it is added.

1 INTRODUCTION

The effect of supplementary cementing materials (SCMs) on the durability of composite cements is both physical and chemical. SCMs tend to refine the pore size distribution of hardened cement paste [1, 2]. As a result, permeability is decreased and the ingress of aggressive media is reduced. SCMs also modify the phase assemblage and therefore affect the physical and chemical mechanisms of interaction between cement paste and aggressive ions (e.g. chloride binding).

According to recent findings [3-5], sulfate attack is related to the crystallisation pressure exerted by the formation of ettringite from fine monosulfate crystals intermixed in nanometric pores with C-S-H [5, 6]. Therefore the important factors influencing sulfate attack are: (i) from the chemical point of view, the amount of monosulfate in nanometric pores available to
react with the sulfate ions and (ii) from the physical point of view, the permeability of the cement matrix [7].

The impact of limestone addition is mainly physical. Up to 5 wt.-% reduces porosity [4]. However, larger amounts (>10 wt.-%) increase porosity and lower the sulfate resistance [4, 7, 8]. The initial compressive strength is decreased and subsequent decrease of compressive strength under sulfate attack is larger [8].

Using 40-50 wt.-% replacement of PC by slag (Al$_2$O$_3$ < 11 wt.-%) was found effective in increasing sulfate resistance [9, 10]. This was explained by lower binder permeability and portlandite content. The lack of detrimental effect of higher alumina content was explained by the fact that alumina was bound in C-A-S-H and not available for ettringite formation. In this case, it was suggested [11] that damage should proceed by disintegration and softening due to the consumption of portlandite and decalcification of C-S-H rather than expansion owing to ettringite formation. Indeed, adding limestone as a source of calcium was reported to improve sulfate resistance of PC-Slag cements [12].

Calcareous fly ashes (CFA) are of interest to industry as alternative SCMs because slags and siliceous fly ashes become more and more limited. Compared to siliceous fly ashes CFA s are less efficient in reducing sulfate attack [13].

Attempts were made to find out whether there is a relationship between calcium content, chemical and mineralogical composition of the fly ash and the sulfate resistance [14]. In general, fly ashes containing glass of mullite or anorthite type tend to have a beneficial effect on sulfate resistance, those of gehlenite type were more likely susceptible to sulfate attack [15, 16]. The calcium-aluminosilicate glass (gehlenite type) was found to be more reactive and supply more alumina. Also CFAs may contain C$_3$A and C$_4$AF which are believed to decrease sulfate resistance [16]. A possibility to increase the sulfate resistance of PC-CFA mixes by adding slag was reported [17].

Chloride ingress also depends on physical and chemical aspects. Chloride penetration is linked to tortuosity of the matrix and is often assessed using a macroscopic chloride diffusion coefficient. The refinement of pore structure by SCMs has an important impact on reducing chloride penetration. From a chemical point of view, chloride can be bound to hydration products by physical adsorption on C-S-H, AFm and AFt phases and by chemical reaction of monosulfate to Friedel’s salt. Adding SCMs, in particular fly ash, can significantly increase the aluminate content and thus increase chloride binding [18, 19]. Also, higher overall AFm, AFt and C-S-H content thanks to SCMs should result in a higher chloride binding potential.

In this study, binary, ternary and quaternary mixes of PC, Slag, CFA and limestone were investigated for durability against sulfate and chloride ingress. Improvement is expected from blending of the materials.

2 MATERIALS

CEM I 42.5R ordinary Portland cement (PC), two calcareous fly ashes (FA1 and FA2), slag (S), limestone powder (LL) and gypsum were used for the study. The materials were ground to similar Blaine surfaces around 4200 cm$^2$/g (PC, FA, S) and 7200 cm$^2$/g (LL). Bulk chemical composition determined by XRF and mineralogical composition by XRD-Rietveld are provided in tables 1 and 2 below.
Table 1: XRF-analysis [wt.-%]

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<tr>
<th></th>
<th>PC</th>
<th>FA1</th>
<th>FA2</th>
<th>Slag</th>
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<tr>
<td>Al₂O₃</td>
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<tr>
<td>P₂O₅</td>
<td>0.2</td>
<td>0.3</td>
<td>0.9</td>
<td>-</td>
</tr>
<tr>
<td>Sum</td>
<td>97.8</td>
<td>97.2</td>
<td>97.7</td>
<td>100.0</td>
</tr>
</tbody>
</table>

Table 2: XRD-Rietveld mineralogy [wt.-%]

<table>
<thead>
<tr>
<th></th>
<th>PC</th>
<th>FA1</th>
<th>FA2</th>
<th>Slag</th>
</tr>
</thead>
<tbody>
<tr>
<td>C₃S</td>
<td>66.8</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>C₅S</td>
<td>8.8</td>
<td>2.5</td>
<td>0.5</td>
<td>-</td>
</tr>
<tr>
<td>C₄AF</td>
<td>9.0</td>
<td>2.0</td>
<td>1.5</td>
<td>-</td>
</tr>
<tr>
<td>C₃A</td>
<td>8.7</td>
<td>1.0</td>
<td>4.0</td>
<td>-</td>
</tr>
<tr>
<td>Quartz</td>
<td>0.5</td>
<td>1.3</td>
<td>3.5</td>
<td>-</td>
</tr>
<tr>
<td>Periclase</td>
<td>-</td>
<td>-</td>
<td>2.0</td>
<td>-</td>
</tr>
<tr>
<td>Anhydrite</td>
<td>3.0</td>
<td>1.8</td>
<td>2.0</td>
<td>-</td>
</tr>
<tr>
<td>Free lime</td>
<td>-</td>
<td>1.7</td>
<td>0.6</td>
<td>-</td>
</tr>
<tr>
<td>Gypsum</td>
<td>2.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Calcite</td>
<td>1.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Amorphous</td>
<td>-</td>
<td>89.7</td>
<td>85.9</td>
<td>100.0</td>
</tr>
</tbody>
</table>

3 METHODS

3.1 Compressive strength

Compressive strength was tested on 4x4x16 cm mortar samples according to EN 196-1 with compositions [wt.-%] as presented in table 3. In limestone containing systems, 10 wt.-% of PC was substituted by limestone. The total cement substitution by fly ash and/or slag was 45 wt.-%. To adjust the SO₃ content of all the mixes to 3.5 wt.-% adequate amounts of gypsum were added. Water to binder ratio was 0.5 by mass and curing times prior to testing were 1, 7, 28 and 90 days under 100% RH conditions.

Table 3: Mix compositions

<table>
<thead>
<tr>
<th>Name</th>
<th>PC</th>
<th>LL</th>
<th>FA1</th>
<th>FA2</th>
<th>Slag</th>
</tr>
</thead>
<tbody>
<tr>
<td>PC</td>
<td>100</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PC-FA1</td>
<td>55</td>
<td>-</td>
<td>45</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PC-FA1-S</td>
<td>55</td>
<td>-</td>
<td>25</td>
<td>-</td>
<td>20</td>
</tr>
<tr>
<td>PC-S</td>
<td>55</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>45</td>
</tr>
<tr>
<td>PC-FA2-S</td>
<td>55</td>
<td>-</td>
<td>-</td>
<td>25</td>
<td>20</td>
</tr>
<tr>
<td>PC-FA2</td>
<td>55</td>
<td>-</td>
<td>-</td>
<td>45</td>
<td>-</td>
</tr>
<tr>
<td>PC-L</td>
<td>90</td>
<td>10</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PC-L-FA1</td>
<td>49.5</td>
<td>5.5</td>
<td>45</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>PC-L-FA1-S</td>
<td>49.5</td>
<td>5.5</td>
<td>25</td>
<td>-</td>
<td>20</td>
</tr>
<tr>
<td>PC-L-S</td>
<td>49.5</td>
<td>5.5</td>
<td>-</td>
<td>-</td>
<td>45</td>
</tr>
<tr>
<td>PC-L-FA2-S</td>
<td>49.5</td>
<td>5.5</td>
<td>-</td>
<td>25</td>
<td>20</td>
</tr>
<tr>
<td>PC-L-FA2</td>
<td>49.5</td>
<td>5.5</td>
<td>-</td>
<td>45</td>
<td>-</td>
</tr>
</tbody>
</table>
3.2 Sulfate attack and chloride ingress

For sulfate attack and chloride ingress investigations, mortars were prepared together with those for compressive strength testing. The curing time prior to exposure was extended to 90 days to ensure sufficient time for the SCMs to react. The curing was done under saturated lime solution.

Sulfate attack was investigated similarly to [20] by measuring expansion of a series of three 2x2x16 cm mortar bars immersed in 3g/L Na$_2$SO$_4$ solution at room temperature. The mortar bars were obtained by removing 1 cm from each of the four longer walls of the 4x4x16 cm bars. Each sample was stored separately in a sealed plastic box keeping a constant ratio of 25 between the volume of the sulfate solution and the total volume of the mortar bars. The measurement of expansion and renewal of sulfate solution was done every 2 weeks for a period of almost 80 weeks.

For chloride ingress testing, the mortar bars were cut into cubes with 2 mm of top surface removed, in order to avoid the wall effect. After sealing of the five remaining faces, the cubes were immersed in 0.5M NaCl solution that was renewed every 5 weeks during 6 months. To obtain the chloride penetration profile, the cubes were gradually ground at 2.5 mm intervals perpendicular to the direction of chloride ingress, using automated grinding equipment. The obtained powdered material was dried to constant mass in an oven at 110°C, weighed and leached with 6M HNO$_3$ for at least 12 hours. Extracted sample was analysed by chromatography for Cl- concentration.

3.3 Phase assemblage

Phase assemblages were determined using paste samples cured 90 days similarly to the mortars. Quantitative XRD Rietveld and TGA analysis were carried out on powders obtained by crushing of paste samples dried in isopropanol and kept in a desiccator during 7+7 days to stop hydration.

4 RESULTS AND DISCUSSION

4.1 Compressive strength

The results of compressive strength test are shown in figure 1. At early ages, the strengths of fly ash and slag blended cements are significantly lower compared to neat PC or to PC-L. This is caused by dilution of cement with SCMs that are not yet reacting. Around 90 days, the strengths of blended cements are comparable to PC and PC-L.

It can be noticed that there exists a positive correlation between fly ash 2 (around 4 wt.-% \( \text{C}_3\text{A} \)) and limestone that produces higher strengths than observed in system without limestone. At all ages, using a combination of fly ash and slag increased the compressive strength compared to replacements using only fly ash (for both fly ashes) or only slag (in case of fly ash 2).
4.2 Sulfate attack

Substitution of 10% by mass of PC with limestone powder resulted in earlier and faster expansion. The damage occurred at lower expansion as showed in figure 2 (left), which confirms the results of previous study [4].

Most of the tested PC-SCM mix compositions did not show significant expansion (values below 0.025%) and are not presented here. The only expanding formulations were PC–FA2 and PC–FA2–S (fig.2 right). Fly ash 2 contains around 4 wt.-% of C₃A and significant amounts of calcium-aluminosilicate glass that can react to monosulfate. Hydrated PC-FA2 system had large monosulfate content (fig.3 right). With lower monosulfate content due to limestone addition, the expansion of PC-L-FA2 was reduced.

Other systems with high (PC-FA2-S) and medium (PC-S) monosulfate content did not show expansion. A possible explanation could be linked to a reduced permeability of these systems due to slag presence, but such hypothesis needs to be further investigated via permeability assessment. Expansion was not observed in systems without monosulfate.
4.3 Chloride ingress

The chloride penetration profiles after 6-month exposition are presented in figure 4, where total chloride is plotted against sample depth. The slope of the penetration profile depends on tortuosity of the matrix and the physical and chemical binding of chloride. The magnitude of total chloride binding can be related to the chloride concentration close to the sample surface.

Blending of PC with SCMs improved chloride resistance. This effect can be attributed to more compact and refined microstructure and thus lower overall permeability in addition to binding of chloride. However, the chloride penetration in samples with FA2 was larger than expected. The permeability of these mortars should be investigated to explain the effect behind. Also, in case of the mixes containing FA2 the binding can be an important factor in reducing the chloride penetration, especially due to the large amount of AFm phases (fig. 5).

Adding limestone has different effects depending on the combination (fig. 6): (i) it increases chloride penetration in PC and slag samples, (ii) it has no influence on FA1 and FA1-S samples and (iii) it decreases the penetration in FA2 and FA2-S samples.

Figure 3: Comparison of the linear expansion of mortars and the monosulfate content.

Figure 4: Chloride profiles in PC systems

Figure 5: AFm phases in studied mixes (XRD)
Total chloride consists of free chloride in pore solution, chloride bound physically and chloride incorporated chemically into Friedel’s salt. The overlapping effects behind chloride binding could be clarified and separated by further investigations including XRD analysis of the phase composition of the layers from sample surface to bulk, sorption isotherm experiment and SEM measurements. Tortuosity derived from migration experiments like ASTM C1202 could be used for the assessment of permeability.

5 CONCLUSIONS

- The durability against sulfate attack and chloride ingress of binary, ternary and quaternary mixes of Portland cement, calcareous fly ash, slag and limestone was assessed. The results demonstrate that blending with the investigated slag and CFAs increased the durability compared to PC.
- The positive chemical interactions between calcareous fly ashes and limestone increased the durability of quaternary systems. However this was not the case for binary and ternary respective PC or PC-slag mixes.
- Expansion caused by sulfate cannot be mitigated by adding only FA2 (around 4 wt.-% C3A). A combined addition of FA2 and limestone can significantly decrease the negative effect.

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RATING BUILDING ASSET DEGRADATION WITH EFFICIENCY INDEXES

Fulvio Re Cecconi (1), Mario Claudio Dejaco (2) and Sebastiano Maltese (3)

(1) Department of Architecture, Built Environment and Construction Engineering (ABC Dept.), Politecnico di Milano, Milano, Italy – fulvio.recececoni@polimi.it
(2) Department of Architecture, Built Environment and Construction Engineering (ABC Dept.), Politecnico di Milano, Milano, Italy – mario.dejaco@polimi.it;
(3) Department of Architecture, Built Environment and Construction Engineering (ABC Dept.), Politecnico di Milano, Milano, Italy – sebastiano.maltese@polimi.it

Keywords: Building Condition Assessment, degradation, service life, documents, Key Performance Indicator

Abstract
Aim of this research is to give building process’s stakeholders some Key Performance Indicators to help them in making correct decisions during operation, maintenance and repair phases. Decisions regarding maintenance, upgrading and also transactions are frequently lacking information and this can cause serious problems, like poor performances, expensive and ineffective maintenance and eventually low yield investments. In order to cover this lack, several Building Conditions Assessment procedures have been developed during the last fifty or more years, but these are often inaccurate, unreliable, subjective or too expensive. In order to help decision-makers in their work, a quick but accurate building survey procedure and some KPIs have been developed to gather and re-elaborate data to rate buildings (and their parts). The procedure analyzes both documentation, as an indicator of building ability to fulfill required performances, and degradation. The procedure output is a rating for the whole building, made of two different indexes: documents and technical. First one has been developed starting from a checklist where each technical document is weighted according to its importance. Technical index is made by two different indexes: the first takes into account anomalies and the second considers each component age in relation to its RSL.

1 INTRODUCTION

Nowadays asset managers’ decisions are becoming more and more complicated so a deep knowledge of the asset condition is needed [7] and this, unfortunately, is not always available. Typically, asset managers must take decisions about maintenance and renewal alternatives based on sparse data about current state of their own assets [17] and this often causes the
waste of much money. Same problems can also be found at building handover when facility managers typically receive many “bankers’ boxes” full of information about their facilities and must, at best, pay to have data keyed into relevant data systems. At worst, facility maintenance contractors are paid to survey existing building to capture as-built conditions [8]. The lack of information, therefore, causes more or less directly a series of other problems, which lead to the use of unsafe buildings, i.e. buildings that do not comply with basic law requirements, to unsatisfactory buildings, i.e. buildings with poor performances, and to low yield investments. This lack of information can be solved by Building Condition Assessment (BCA), which can be applied to an asset with different levels of depth and accuracy.

BCA techniques have been studied since the birth of the necessity of knowing what current asset performances are, to consequently act in the most effective way. Baird et al. [1] defined nine different types of evaluation techniques, from empirical to theoretical and from internal to external, most of the assessment techniques found in literature can be categorized inside this scheme. Shohet et al. [16] described some methods that differ for objectives and measuring parameters, as instance: a) qualitative evaluation criteria; b) cost-driven and/or physical state Key Performance Indicators (KPIs) in combination with standards and regulation compliance evaluation, as described also by Johnston et al. [9]. Assessment methods can vary also according to the building scale that they want to evaluate: from general to particular, where each component has a specific and detailed evaluation method, like in Rodrigues et al. [11] for façades. In these last years energy/sustainability-oriented refurbishments became more and more important and BCA also became more and more oriented to support decision related to this kind of refurbishment; this can be done, as written by Menassa [10] et al. and Caccavelli et al. [3], by linking to the assessment, which can be seen as a building picture, an analysis of main criticalities and potentials. These topics are obviously related to facilities management, on which Shohet et al. [15] showed possible improvements. TOBUS and EPIQR project demonstrate that evaluation techniques, to be more effective, should be targeted to building function, as instance offices or residential [2]. Last but not least, evaluation techniques should give as output an index, rating, mark [12] [13] [14] to ease decision makers’ work and to create a buildings ranking inside the asset to prioritize maintenance works and evaluate refurbishment alternatives.

The requirements analysis of building evaluation techniques showed the extreme difficulty in achieving a detailed survey with a small effort. Two possible solution, which are a good compromises between a detailed survey and a quick one, are listed below: a) a quick survey on the whole building to rate it and compare with other buildings of the same asset; and b) a slower survey, more detailed and applied on a small buildings group, focused mainly on more deteriorated components, with a rating associated.

2 KPIs FOR BCA

The idea of creating a rating system for BCA was born together with the necessity to create and manage a well-organized building logbook, which allows stakeholders (owners, users, professionals, etc.) to get a better and faster analysis of the building itself. This work highlighted the necessity of mainly two types of rates: a technical index to assess building conditions in terms of aging and anomalies of its components; a documents index to describe the quality and quantity of available building documents taking into account legal
requirements. This paper focuses mainly on the first index, while for the second some publications have been already published [4].

The technical index is used to assess building condition, by measuring building components degradation and service life. This index is made by three sub-indexes: the first two (called service life indexes) comparing the Actual Service Life (ASL) of each component with its Reference Service Life (RSL) and the third index evaluating anomalies found on each building component (called degradation index). The two service life indexes are alternative, \( D^+ \) for components with \( ASL \leq RSL \) (1) and \( D^- \) for components with \( ASL > RSL \) (2). On the other hand, the degradation index of a component consists of three sub-indexes (5): for low, medium and serious anomalies. These three sub-indexes are aggregated into one describing the component state (called A), through a weighted average (6).

Since the technical index of a building is a function of the indexes of its components, a standard Work Breakdown Structure (WBS) has been created. This WBS, following UNI 8290 standard [6], has been organized through five levels, from general to detailed, and the three indexes are calculated for building components and then grouped for technological units (i.e. family of building components, level 2 of the WBS) (3) (7), and, eventually, grouped for the whole building (4) (8). When moving from level 2 of the WBS to the whole building indexes, the different importance of each technological unit may be taken into account using two different series of weights: the first related to the economic value and the second related to the criticality (i.e. the importance of component performances in order to obtain the required building performances) of each component. The economic weight is proportional to the percentage contribution of each technological unit to the total construction cost. A pair comparison method (Analytical Hierarchy Process – AHP) has been used to assess the relative importance of each technological unit then converted into criticality weight.

Besides the WBS, some other tools are needed to compute building components and building efficiency indexes, namely a RSL database and a list of possible anomalies for each building component. The former has been built starting from a literary review of major existing databases and from experts’ interviews while to make the latter building components anomalies have been classified, according to the magnitude of their damages on the component itself (minor, medium and serious) and to their typology: step change (on/off) and gradual change. Each anomaly has been given an unequivocal code, a name, a description and a measuring parameter. The complete list is made by totally 431 anomalies and each component has meanly 12 anomalies, up to totally 5100 possible cases. These three tools, the WBS, the RSL database and the list of anomalies, are the core of a collection of diagnostic forms (up to 438 forms have been created) to be used in building inspection (it is trivial that in a building there is not the contemporaneous presence of all the database components). Each building component can be evaluated using a diagnostic form, which consists of four parts: a) form data (code, name, number); b) component data (code, name, notes, Actual Service Life ASL); c) anomalies check list (with the possibility to select the anomalies for the component and, if need be, to put the extension) and d) indexes output (automatically calculated as soon as the data are entered). These forms are collected in 18 folders, which correspond to the 18 technological units in the WBS.

The necessity of having a single index for building technical rating is clear: with a single index a ranking can be done among more buildings and also decision making becomes easier, avoiding possible misunderstanding. The technical index of the whole building is the comparison between the selected building state and its perfect ideal state, without anomalies.
and maintained properly but with the same age of the real building and it is computed as the ratio (10) between the area of two triangles with the three indexes as vertexes [5].

\[
D_C^+ = \frac{RSL - ASL}{RSL} \tag{1}
\]

\[
D_C^- = 1 - \frac{ASL - RSL}{ASL} \tag{2}
\]

\[
D_{TU}^+ = \begin{cases} \sum_{i=1}^{n} D_{C,i}^+ & \text{if } n \geq 1 \\ 0 & \text{if } n = 0 \end{cases} \quad D_{TU}^- = \begin{cases} \sum_{j=1}^{m} D_{C,j}^- & \text{if } m \geq 1 \\ 1 & \text{if } m = 0 \end{cases} \tag{3}
\]

\[
D_{Bld}^+ = \frac{\sum_{k=1}^{o} D_{TU,k}^+ * W_k^{E/C}}{\sum_{k=1}^{o} W_k^{E/C}} \quad D_{Bld}^- = \frac{\sum_{k=1}^{o} D_{TU,k}^- * W_k^{E/C}}{\sum_{k=1}^{o} W_k^{E/C}} \tag{4}
\]

\[
A_L = \frac{\sum_{i=1}^{l} P_{L,i} * E_i}{L} \quad A_M = \frac{\sum_{j=1}^{m} P_{M,j} * E_j}{M} \quad A_S = \frac{\sum_{k=1}^{s} P_{S,k} * E_k}{S} \tag{5}
\]

\[
A_C = \frac{A_L * W_L + A_M * W_M + A_S * W_S}{(W_L + W_M + W_S)} \tag{6}
\]

\[
A_{TU} = \frac{\sum_{i=1}^{q} A_{C,i}}{q} \tag{7}
\]

\[
A_{Bld} = \frac{\sum_{k=1}^{o} A_{TU,k} * W_k^{E/C}}{\sum_{k=1}^{o} W_k^{E/C}} \tag{8}
\]

\[
I_{Tech} = \frac{\text{Area}_{Building}}{\text{Area}_{Optimal}} \times 100 \% \tag{9}
\]

So the technical index is the ratio between two building conditions:

a) ideal: the best situation in which the building can be. No anomalies (A = 100%), all the components have been replaced according to their RSL (D* = 100%). D* represents the age of the building, which physiologically gets older. This is the best results that the actual building can get;

b) actual: this is the current building situation. Index A decreases when anomalies are found and D* decreases when one or more components pass RSL limit. D* is always equal to the ideal one because, as said before, a component can become older without any problem.

The more the actual condition is smaller then the ideal, the more the building has a low technical index, which means the presence of anomalies and components that do not fully satisfy their requested performances. This can be represented also in a Kiviat graph, which does not substitute the analytic calculation but it is a good way to show results in a more comprehensive (for a non-technician) way than just a number.

3 CASE STUDIES

Some case studies have been already presented in other proceedings [11], in this paper a comparison among five case studies is presented to highlight the possible use of efficiency
indexes in asset management, to point out relationships between indexes and buildings behavior and to define, at the end of this research project, potentials and future developments.

Case studies have been selected with the aim of having building of different ages, functions, quality and also degradation level. Here are the selected buildings: CS1) residential building (low quality, built in 1933); CS2) residential building (high quality, built in 1953); CS3) university building (built in 1966); CS4) office building (previously residential, built in 1890) and CS5) a primary school (built in 1895). Here main results are presented at different aggregation level, to better understand system complexity.

3.1 Building level

Many interesting data come out from building level analysis. The first output, outlined in Figure 1, is the final building index, split also in documents and technical (in case that one of the two is missing, the final index is just the remaining).

![Figure 1 – Building Efficiency Index](image)

Building index is the average (10), when possible, of the previous two and in these five case studies goes from 60% to 90%, while the technical index goes from 60% to almost 100%, which means the best condition.

It is now interesting to analyze the relationship between types and quantities of encountered anomalies and the technical index, as shown in Figure 2. Minor anomalies have a really low influence on the degradation index calculation because they are related mainly to visual aspects and not to functionalities; therefore medium and serious anomalies are more “interesting” and they need accurate analysis. The more medium and serious anomalies are found, the more the technical index decreases: it is necessary to act firstly to solve these problems and then, with a lower priority, it is possible to restore visual aspects and minor damages. It can also be noticed, from Figure 2, that minor anomalies are more or less the 60% of the whole failures encountered, while there are 26% of medium and 12% of serious ones.

Moreover, at this macroscopic level, other correlations can be analyzed, as can be seen in Figure 3. Technical index is not connected to the construction year and to the year of last maintenance operation, whilst the correlation with the maintenance policy (intended as quantity and quality of maintenance performed across years) is really interesting: the more operations are done, the higher the index, but many other factors, hard to control with a tool like this presented here, influence the final result too. These factors are related to interventions extent (in terms of number of components replaced and repaired), to managers’ decisions (many times the feasible solution is not the better option, due mainly to economic aspects) and also to maintenance done to solve/lower temporarily the problem, but without getting rid of its causes (this is frequent in anomalies related to water leakages and humidity). So this correlation cannot be easily standardized but it requires a precise reasoning each time.
3.2 Technological unit level

Speaking about “building average age” is too generic (a building is made by too many components, so a single number can be made by thousands of combinations) but the average age of each technological unit (TU) seems to be interesting, if connected with its technical efficiency index, as shown in Figure 4.

A TU is a group of components with homogeneous functions so an average age of each TU can be defined. The technical efficiency index increases when new components are replaced or restored because it depends both from anomalies detected and components age. In addition to this, plants have a different RSL, usually shorter than other building components, as can be seen in TUs from 12 to 18 (dedicated to plants). Comparisons among TUs of these five case studies have been searched but they have different, functions, ages and policies, so correlations have not been found.
### 3.3 Component level

At component level, comparisons and connections seem not really useful, because the case studies have different functions, age and maintenance policies; the same it is not true if an entire asset is under analysis. On the contrary, statistics on different components can be gathered from the diagnostic forms filled during the survey, to recalibrate the RSL and check if anomalies are missing or under/overestimated. In Table 1 a quick example for the internal plaster is presented.

#### Table 1: External plaster statistic

<table>
<thead>
<tr>
<th>Parameter</th>
<th>CS1</th>
<th>CS2</th>
<th>CS3</th>
<th>CS4</th>
<th>CS5</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASL</td>
<td>20</td>
<td>60</td>
<td>47</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Serious anomalies [-]</td>
<td>2</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Medium anomalies [-]</td>
<td>3</td>
<td>5</td>
<td>2</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Minor anomalies [-]</td>
<td>4</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>1</td>
</tr>
<tr>
<td>Interventions performed</td>
<td>repainting</td>
<td>local replacement, repainting</td>
<td>local replacement, repainting</td>
<td>–</td>
<td>all old plasters replacement</td>
</tr>
</tbody>
</table>

Obviously this is just an example; statistics can go more in depth, by analyzing anomalies types and extension. A system like this can really help asset managers in taking their decisions, prioritizing interventions and also allocating budget.

### 4 ONGOING DEVELOPMENTS

This research was focused on BCA not as a stand-alone instrument, but as a part of a bigger instrument, able to help building process stakeholder in managing their asset. Uses, functionalities and potentials have been highlighted in this paper, but further steps are needed. Connection with a maintenance operations database is under development, with the aim of going from the building survey to interventions scheduling and budgeting; for this issue, parametric costs have to be gathered from price lists and existing projects. Also sustainability assessment, in terms of environmental impacts, has to be connected with maintenance operations and refurbishment alternatives. Documents index, not described here, is useful both as a checklist (during design phase) and for handover, refurbishment or maintenance, because it gives to the users the precise asset situation in terms of documents (correct, to be updated, missing). Current KPIs can be also be dynamic, by using them to evaluate ex-ante or ex-post interventions and they also can be attached to an existing quantity survey, to improve the accuracy of future budgeting. The overall system helps users in getting the true economic building value and/or the amount of money needed to restore this value, as it is well known that buildings value depends from many parameters (distance from the center, building quality, etc.).
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DURABILITY OF FIBRE-CEMENT ROOFING CORRUGATED SHEETS

Juliano Fiorelli (1), Holmer Savastano Junior (1), Gustavo H. D. Tonoli (2) and Sergio Francisco dos Santos (3)

(1) Department of Biosystems Engineering, University of São Paulo, Pirassununga, SP, Brazil – julianofiorelli@usp.br; holmersj@usp.br

(2) Department of Forest Sciencea, Federal University of Lavras, Lavras, MG, Brazil – gustavotonoli@yahoo.com.br

(3) Department of Materials and Technology, University of the State of São Paulo, Guaratinguetá, SP, Brazil – sfsantos1@usp.br

Abstract

The durability of fibre-cement roofing sheets is in direct correlation with the type of fibres used, the structure of the matrix, and the curing conditions. This work is comprised of a study of the degradation of polyvinyl alcohol (PVA) and polyethylene terephthalate (PET) fibres in contact with a cement solution (pH~11.5); an evaluation of the methodology for accelerated aging test (200 soak & dry cycles) established by EN 494 (1994); and an evaluation of the influence of certain raw materials as well as natural weathering exposure on the appearance of edge cracks in fibre-cement roofing sheets produced by the Hatschek process and disposed in plastic wrapped piles. The results show that PET fibres presented degradation after approximately one month immersed in a cement solution and with a consequent tensile strength reduction of 53%. With respect to the parameters of the cycles of soaking and drying, the phenomena involved in the inlet and outlet water proved to be distinct from each other, with greater ease of absorption in comparison to drying out water. Corrugated sheets after 24 months of natural weathering exposure in rural area presented higher incidence of cracks on the north face of the stacked roofing sheets, while fibre cement sheets reinforced with PVA fibre and without silica fume in the matrix composition presented lower incidence of edge cracks when compared to other formulations evaluated in this work.
1 INTRODUCTION

The fibre cement industry produces cost-effective and functional building materials, which stand out in developed and developing countries, especially due to their versatility and easy manufacture.

Currently, the use of cellulosic fibres, especially through pulps, has considerable potential for application in fibre-cement production. They present several interesting advantages, in particular their low density, their bio-renewable character and their widespread availability at competitive cost and in a variety of morphologies and aspect ratios [1]. The pulps however are of such a length (2.5 to 3 mm) that they bridge the openings of the sieve and form an initial filter layer in the felt of the Hatschek machine. The Hatschek process is the most widely employed process in the production of fibre cement components [2]. The pulps form a film that blocks the sieve and allows for the capture of other cement and added mineral particles [3]. However, air cured cement composites only reinforced with natural fibres do not reach levels of mechanical performance with medium and long term reliability, mainly due to the degradation of cellulosic fibres in the alkaline environment [4]. One of the production methods to mitigate this drawback is the high pressure steam cured composites reinforced by cellulose fibres (autoclaved products), mainly for external cladding, sidings and internal partitioning as well as sealing [3].

At present times there is an alternative method for the production of roofing elements, usually for corrugated sheet, using traditional Hatschek technology [5]. This process is based on the use of air cured hybrid fibre-cement with refined wood pulp, such as pine and eucalyptus, and alkali-resistant synthetic fibres such as polyvinyl alcohol (PVA) and polypropylene (PP).

The textile type fibre, such as a reinforcement of polyethylene terephthalate (PET) high-performance yarn coated by resin, is recognized as an important synthetic construction material (high-tensile strength and excellent creep properties). However, its use presents challenges, such as a deterioration of mechanical properties and weak chemical stability (due to the hydrolysis of the PET ester group) [6].

Edge crack and delamination is another common phenomenon in air cured hybrid fibre-cement, which affects the finishing qualities of the roofing tiles. There is a lack of information about the effects and contribution of edge, or initial, cracks and delamination on the long-term and mechanical performance of theses composites produced by the air cured Hatschek process.

The main objective of this work is to present and discuss several variables involved with the degradation of fibre-cement corrugated sheets used for civil construction. Raw materials, processing and aging conditions will be evaluated in order to explain the several agents and mechanisms of decay of the asbestos free fibre cement composites. Consequently this paper presents a study of the degradation of polyvinyl alcohol (PVA) and polyethylene terephthalate (PET) fibres when in contact with a cement solution (pH~11,5); an evaluation of the methodology for accelerated aging testing (200 cycles) established by EN 494 (1994) [7]; and an evaluation of the influence of some raw materials and natural weathering exposure on the appearance of edge cracks in hybrid fibre-cement roofing tiles produced by the Hatschek process.


2 AGING TEST OF PVA AND PET FIBRES

The synthetic filaments are responsible for reinforcing, toughening and dimensional stability of the fibre-cement composite under utilization conditions during its service life. The chemical stability of these reinforcing elements in the cement environment is a key issue regarding the integrity and durability of the roofing corrugated sheets.

2.1 Materials and Method

The aging test of PVA and PET fibres immersed in saturated cement solution was carried out in the following conditions: 3 L of deionised water saturated with Portland cement CPV-ARI, 5 g of fibres, temperature 40°C and pH ~11.5. The fibres were previously dispersed for 5 min at 3600 rpm. The fibres were analysed after 7, 21 and 35 days of immersion. The SEM images were performed using a Hitachi TM3000 microscope.

2.2 Results

This section presents SEM images, as well as the results for tensile stress of PET and PVA commercial fibres after performing the aging test in a saturated cement solution.

![Figure 1: SEM Images of PET fibres exposed to saturated cement solutions by different periods of time](image)

Figure 1: SEM Images of PET fibres exposed to saturated cement solutions by different periods of time
Figure 2: SEM Images of PVA fibres exposed to saturated cement solutions for different periods of time

Figure 3: Evaluation of tensile stress: PET fibres and PVA fibres after different periods of exposition to severe alkaline attack.

After 35 days of immersion in saturated cement solution, degraded regions (holes) on the fibre surface and reduced fibre tensile strength of 52% were observed for the PET fibres (Figure 3). This behaviour is compatible with previous experiments carried out by Silva et al. [8] using PET recycled fibres in cement based materials with the consequent reduction of toughness of the studied mortars under aging conditions. For PVA fibres, the degradation of
the surface of the fibre was not observed, however there was a reduction in the tensile strength of around 19% (statistically significant) but with a tendency of stabilization of mechanical strength after 21 days of exposition to the aggressive environment. These results indicate that the PET fibres under evaluation are not alkali resistant and their use in fibre-cement will possibly weaken the mechanical properties of the composites over time.

3 AGING TEST OF FIBRE-CEMENT ROOFING SHEETS

The accelerated aging test exposed the composite to cycles of immersion and drying in order to simulate the environmental conditions to which the material would be subjected in the actual applications.

3.1 Methodology

The procedure recommended by the International Standards [7] requires the completion of 50 soak & dry cycles. Each cycle is composed of immersion in a water tank at room temperature for 18 h and 6 h drying in a ventilated oven at 60°C. In this study the referred aging procedure was adapted, with respect to the period of duration of each cycle: approximately 3 h immersion and 3 h drying. The total number of cycles was also increased in the attempt to better characterize the behaviour of the different tested materials. This work adopted 200 ageing cycles (around 50 days of accelerated aging test) based on previous experience reported elsewhere [9,10].

To assess the composites behaviour submitted to the accelerated aging cycling, several formulations were sected with different reinforcing fibres: coir, PVA and cellulose pulp, mainly after 28 days of curing. Some of these composites were subjected to fast carbonation curing in the initial age as proposed by Almeida et al. [11].

3.2 Results

Dias [12] highlights the degradation mechanisms involved in the cycles of immersion and drying, such as alkali attack and densification of the fibre/matrix interface region. Furthermore, the cited work refers to progressive damage caused to the material, which corresponds to the stresses generated by the dimensional movements during the thermal and hygroscopic cycles.

Table 1 shows the water absorption and water loss for the half cycle (2 h 50 min), and the maximum absorption and water loss observed for up to 9 h (540 min). Water absorption was observed to be faster and almost completed within 2 h and 50 min. Differently the carbonated materials only demonstrated 68% of the maximum water absorption during immersion what can be understood by the lower incidence of open capillary pores. The sealing generated by the carbonation reaction prevents the material from absorbing water during the course of 2 h and 50 min, but the same is not true for the other treatments. This refinement of the pore structure and limited access of water to the fibrous composite may explain the lower shrinkage of the carbonated material before and after 200 aging cycles (Figure 4).

With respect to the loss of water during drying process (Table 1), it was observed that the loss water movement takes longer for all the analysed treatments what would determine greater periods (even longer than 9 h) for loosing higher amount of free water. The carbonated material, following the previous result of absorption, also showed the lowest relative loss compared to non-carbonated materials.
Table 1: Water absorption/loss average numbers of different composites in one cycle of the soak & dry test

<table>
<thead>
<tr>
<th>Treatments</th>
<th>Water absorption (%)</th>
<th>Relation (%)&lt;sup&gt;1&lt;/sup&gt;</th>
<th>Water loss (%)</th>
<th>Relation (%)&lt;sup&gt;1&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Curing - reinforcing fibre</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbonated - cellulose pulp</td>
<td>6.28</td>
<td>9.22</td>
<td>68</td>
<td></td>
</tr>
<tr>
<td>Non-carbonated - cellulose pulp</td>
<td>16.13</td>
<td>16.83</td>
<td>96</td>
<td></td>
</tr>
<tr>
<td>Non-carbonated - PVA fibre</td>
<td>19.27</td>
<td>19.53</td>
<td>99</td>
<td></td>
</tr>
<tr>
<td>Non-carbonated - coir fibre</td>
<td>18.28</td>
<td>18.73</td>
<td>98</td>
<td></td>
</tr>
</tbody>
</table>

<sup>1</sup>Water absorption/loss after 170 min in relation to 540 min.

Figure 4: Variation on drying shrinkage composite over time

4 EDGE CRACKING OF FIBRE-CEMENT CORRUGATED ROOFING SHEETS

Edge cracking is a serious pathology that commonly affects air cured fibre cement materials during drying conditions when the corrugated sheet are still in piles. It is normally generated in the initial ages by the stresses that take place when the water evaporation in the lateral edge of the sheet stack is faster that the internal diffusion of the water in the pile.

4.1 Materials and Method

The roofing sheets evaluated in the study were industrially produced by the Hatschek process and were composed of: polymeric fibres, PVA and/or PP, refined bleached pine pulp, refined unbleached eucalyptus pulp, Portland cement, silica fume and limestone. The fibre-cement roofing sheets after were submitted to ten days of saturated curing and subsequently to 24 months of natural weathering exposure in a rural environment located in Pirassununga, São Paulo state, Brazil (21° 59'S latitude and 627 m of altitude). The roofing sheet piles were oriented with coverage wave positioned to the magnetic north and waiting wave to the south for the maximum incidence of solar radiation in the critical face of the pile (for the south
hemisphere). All piles were packed with transparent plastic for the 24 months of analysis, in order to avoid excessive evaporation of water to the environment and simulating the real conditions of sheet stacks in the storage conditions. Table 2 lists the treatments studied, according to the formulations containing different types of plastic fibres (PVA and/or PP).

Table 2: Formulations of the roofing sheets submitted to edge-cracking study

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Formulation (fibre + pozzolan addition)</th>
<th>Treatment</th>
<th>Formulation (fibre + pozzolan addition)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T1</td>
<td>PVA+ silica fume</td>
<td>T4</td>
<td>PP</td>
</tr>
<tr>
<td>T2</td>
<td>PVA</td>
<td>T5</td>
<td>PVA+PP+ silica fume</td>
</tr>
<tr>
<td>T3</td>
<td>PP+ silica fume</td>
<td>T6</td>
<td>PVA + PP</td>
</tr>
</tbody>
</table>

The study of edge cracks and delamination was performed by means of a visual analysis in order to observe the magnitude, positioning in the sheet edge/stack and number of defects detected. The lateral faces of the piles of fibre-cement sheets were divided into nine sections to facilitate the identification and the quantification of the incidence of edge cracks and delamination. The percentage of cracked roofing sheets was calculated dividing the number of cracked sheets by the total number of sheets in the piles. The analyses were performed at 28 days and 2, 6, 12 and 24 months after production.

4.2 Results

Table 3 shows that the lateral side of the sheet stacks facing north generally yielded the highest percentage of edge cracking, regardless of the formulation (T1-T6). These results suggest a direct relationship between severity of the natural weathering exposure such as higher temperatures (average temperature > 25°C) which maximise the rate of water loss, and the corresponding shrinkage of the material with the consequent stress-induced edge cracking. The increased diffusion and evaporation of water in the region close to the stack edge, when compared with the central zone of the pile results in a significant moisture gradient and with the preponderant stress factor across the edge of the corrugated sheets. No edge crack propagation to the centre of the roofing sheets was noted without compromise to the tightness or mechanical performance. Furthermore, there was no identification of significant incidence of delamination in the roofing sheets under analysis.

Table 3: Proportion (%) of cracked roofing sheets on north (N) and south (S) faces - different ages

<table>
<thead>
<tr>
<th>Age / Face</th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
<th>T4</th>
<th>T5</th>
<th>T6</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>N</td>
<td>S</td>
<td>N</td>
<td>S</td>
<td>N</td>
<td>S</td>
</tr>
<tr>
<td>28 d</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2 m</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>6 m</td>
<td>0</td>
<td>0</td>
<td>2</td>
<td>29</td>
<td>0</td>
<td>7</td>
</tr>
<tr>
<td>12 m</td>
<td>22</td>
<td>4</td>
<td>54</td>
<td>30</td>
<td>92</td>
<td>9</td>
</tr>
<tr>
<td>24 m</td>
<td>62</td>
<td>70</td>
<td>56</td>
<td>30</td>
<td>93</td>
<td>13</td>
</tr>
</tbody>
</table>

Legend - d: days; m: months; N: North; S: South
Table 3 highlights the T1 and T2 formulations’ superior resistance against edge cracking after the natural weathering exposure period, i.e. fibre cement roofing sheets reinforced with PVA fibres, with and without silica fume, presented less edge cracks on the north face of the piles than other formulations. There was no significant contribution of silica fume to the reduction in edge cracking in the roofing sheets for the observed formulations.

Figure 5 depicts the results of edge cracks on the north face of the piles and the rainfall index for different periods. The existence of a correlation between the number of edge cracks in the piles and a low rainfall index (winter season) was observed. Periods of lower relative humidity exert an increase in the rate of water loss of the roofing sheets, resulting in the occurrence of edge cracks. However, it was also noted that for formulations T1 to T6, the incidence of edge cracks was less than 40%, during the 6 month summer period, characterized by the high rainfall indexes.

**Figure 5**: Rainfall index (▲) and the proportion of sheets in piles with edge cracks in different ages (a: T1 – T2; b: T3 - T4; c: T5 - T6)

**FINAL REMARKS**

The durability of asbestos free air cured fibre cement material is resultant of several variables related to raw materials, curing procedures and weather exposition. Accelerated tests also are limited to reproduce the absorption/loss of water movement. The PET filaments show a much greater reduction in the reinforcing capacity in comparison to PVA fibre exposed to the alkaline cementitious environment. Fast carbonation curing contributes for the reduction of water movement and with the consequent dimensional stability of the fibre cement.

The edge cracks of the fibre cement roofing sheets disposed in piles showed a consistent evolution after 24 months of natural weathering exposure. Fibre cement sheets reinforced with PVA fibres, with and without silica fume addition in the cement matrix, presented lower percentage of edge cracks on the north face than other formulations.

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REFERENCES


THERMAL SHOCK EFFECTS ON BONDING OF CEMENT MORTAR RENDERINGS

Juan F. Temoche Esquivel (1), Mercia Barros (2)

(1) Department of Exact Sciences of Nove de Julho University, São Paulo – jftemoche@uninove.br
(2) Department of Civil Construction of Polytechnic School of University of Sao Paulo, São Paulo – mercia.barros@poli.usp.br

Abstract

The use of cement mortar renderings in external walls either as an intermediate layer or finishing layer is an extended practice for almost every residential building in Brazilian cities. Nevertheless durability of these renderings is still a concern for technicians, mainly about cracking and loss of bond.

This work aims to analyze experimentally, thermal shock degradation effects on bonding of cement mortar renderings.

In order to support experimental model a cement mortar rendering bonded to a concrete substrate was simulated under thermal stresses caused by a thermal shock and a Finite Element Analysis was made. This numerical model let define a critic scenario with the existence of bond flaws in the interface of cement mortar and concrete substrate during the event of thermal shock and also geometrical characteristics of experimental model. Other boundary conditions were defined using standards parameters.

Finally, pull-off tests were made on experimental models after the thermal shock cycles and the results were analyzed with statistical support.

Experimental results showed a decrease of bonding resistance of the rendering with the increase of rates of bond flaws and degradation of bonding resistance was increased after the thermal shock cycles.

1 INTRODUCTION

One of the factors that rules performance of cement mortar renderings is its bonding on the substrate independently of materials of production processes used. And bonding is influenced: by substrate characteristics, such as water absorption, porosity, roughness and its chemical nature [1]; by application energy [2] and by rheological characteristics of mortar at the moment of initial contact with the substrate and afterwards when setting and finishing it. Both substrate and mortar characteristics and production process will define effective contact area called bonding extension which may be measured by the volume of interface flaws.
During service life, mortar renderings degrade due to many environment agents which affect renderings randomly and this makes durability analysis complex.

One of these degradation agents is temperature because its variation causes stresses being tensile stresses the more critical for cement mortars. Microscopically stresses are due to differences between thermal expansion of cement paste and aggregates. Macroscopically stresses are due to variation of internal temperature of mortar and movement restriction due to bonding. This thermal stresses may affect cement mortar bonding.

When mortar rendering initially in thermal equilibrium suffers a quick fall in surface temperature meanwhile the interface is still close to initial temperature a thermal shock happens [3] causing tensile stresses mainly in the external surface. If stresses were greater than tensile strength, material fracture may occur.

In tropical countries like Brazil, this kind of thermal shock may occur frequently, however very few research has been made about effects of thermal shock on external renderings.

This paper aims to analyze synergetic effect of interface flaws of cement mortar renderings when exposed to thermal shock. In order to this, a numerical static models were used to analyze thermal stresses caused by thermal shock and helped to choose the more influential mortar properties and afterwards an experimental model was developed.

2 INTERFACE FLAWS IN MORTAR RENDERINGS

During the hardening process transition areas are made between paste and aggregates which will turn into pores, micro cracks, grain edges, air bubbles which are discontinuity areas in the rendering which inherent flaws of mortar rendering. Otherwise, in the interface area between mortar and substrate the effective contact area is responsible for rendering bonding. This contact area may be quantify by the interface flaws rate which was obtained by [4] from Digital Image Processing of transversal sections of mortar rendering samples as shown in figure 1a.

![Figure 1a](image1a.png)
![Figure 1b](image1b.png)

Figure 1: a) Transversal sections of mortar rendering samples and one quadrant image analysis. b) Format and dimensions (8 mm x 1 mm) used to simulate interface flaws

As the flaws were used in the bi-dimensional numerical simulation, its geometry and dimensions were define from images of [4] using graphic scales of the images as shown in figure 1b.

3 THERMAL SHOCK IN MORTAR RENDERINGS

Under environment conditions of exposition of cement mortar renderings, some authors [5][6][7][8] define thermal shock as a phenomena produced by the quick fall of temperature
of the external surface of the cement mortar rendering, such as at the beginning of a storm after a long period of insolation.
In this scenario, temperature variation would be more critical as increase in surface temperature and rate of increase would be greater. Otherwise, surface characteristics of radiation recipient surface such as pitch, surface roughness, color, bearings, also have influence in the surface temperature rise.
Environment temperature decrease lasts some minutes (it’s not instantaneous) and begins with some cold wind (less than 20ºC) and then rain. As water contacts the rendering surface the difference of temperature has decrease some degrees due to convection caused by cold wind, however water presence will saturate rendering surface and may accelerate heat flows. The gradient produced will depend on the highest temperature reached.

4 MATERIALS AND METHODS

To defined experimental model, a three-layered finite element bi-dimensional model was developed, using Ansys®, with focus on 3 variables: deformation module of mortar, thermal properties of mortar and quantity of interfacial (rendering/substrate) flaws. A static analysis was considered with a rendering surface temperature of 70°C and 20°C on the opposite surface (substrate), considering the initial temperature of the model as 20°C as detailed in [9]. Samples of cement mortar rendering were produced to simulate thermal shock. As a substrate was used a prismatic concrete block and on its interface surface flaws were created in a proportion defined with the numerical model.

4.1 Materiais

4.1.1 Substrate

A prismatic concrete substrate was produced with the characteristics shown in the following table.

Table 1: Substrate characteristics

<table>
<thead>
<tr>
<th>Material</th>
<th>Dimensions (m)</th>
<th>E (GPa)</th>
<th>Density (kg/cm³)</th>
<th>Surface characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concrete</td>
<td>0.30 x 0.40 x 0.10</td>
<td>25</td>
<td>2.400</td>
<td>Defined by [10]</td>
</tr>
</tbody>
</table>

4.1.2 Interface flaws

After modeling, the interface flaws were defined as a circular hub with 8 mm of diameter and height of 1 mm, immersed into the mortar and bonded to the substrate (Figure 3) which would result in rendering layer such as shown in Figure 4. The area occupied by the interface flaws was defined in 35% of the contact area.

Figure 3: Simulation of solid ar flaws on the substrate surface
To simulate flaws over the rough and porous surface of concrete samples it was used an expansive acrylic emulsion and used in silk painting. After application over the concrete surface, heat was applied to produce volumetric expansion in the flaw (1 mm high) as shown in figure 5. The flaw rate printed in the concrete surface was 35% as shown in figure 5.

![Figure 4: Resulting flaws on the cement mortar rendering](image)

![Figure 5: Printing interface flaws in concrete substrates at a rate of 35%](image)

### 4.1.3 Cement mortar for rendering

A volumetric proportion of 1:4 (dry materials) was defined in order to power elasticity modulus effect on cement mortar. After some pre-tests water ratio was defined in 16% of dry materials mass. Table 2 shows materials mass proportion resulting.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mass of dry materials per cycle of mixture (20 liters of mortar) in Kg</th>
<th>Proportion in mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland Cement CP II F</td>
<td>5,177</td>
<td>1</td>
</tr>
<tr>
<td>quartz sand</td>
<td>29,818</td>
<td>5,759</td>
</tr>
<tr>
<td>Water</td>
<td>5,599</td>
<td>1,08</td>
</tr>
</tbody>
</table>

Mortar renderings were produced over a rigid concrete prismatic sample (0,30m x 0,40m x 0,10m) with surface characteristics of a standard substrate [10]. Cement mortar was produced by mechanical mixing and was applied on the substrate by mechanical projection with compressed air equipment at a constant pressure of 90 psi, as shown in figure 6:
4.2 Test methods

Separated test were done on mortar materials, cement mortar and on mortar renderings.

4.2.1 Tests on cement mortars

Cement mortars materials were tested as summarized in Table 3;

Table 3: Materials tests

<table>
<thead>
<tr>
<th>Material</th>
<th>Test</th>
<th>Nº of repetitions</th>
<th>Standard reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand</td>
<td>Dry-rodded density</td>
<td>3</td>
<td>[12]</td>
</tr>
<tr>
<td></td>
<td>Dry density</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Wet mortar</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cement mortar</td>
<td>Mass density</td>
<td>3</td>
<td>[13]</td>
</tr>
<tr>
<td></td>
<td>Air content</td>
<td>3</td>
<td>[13]</td>
</tr>
<tr>
<td>Hardened mortar</td>
<td>Tensile strength by flexural stress</td>
<td>6</td>
<td>[14]</td>
</tr>
<tr>
<td>Mortar Rendering</td>
<td>Elasticity modulus -</td>
<td>6</td>
<td>[15]</td>
</tr>
<tr>
<td></td>
<td>Recommendations for the measurement of dynamic modulus of elasticity</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Apparent density</td>
<td>3</td>
<td>[16]</td>
</tr>
<tr>
<td></td>
<td>Thermal expansion</td>
<td>3</td>
<td>[17]</td>
</tr>
<tr>
<td></td>
<td>Thermal shock</td>
<td>30</td>
<td>[18]</td>
</tr>
</tbody>
</table>

4.2.2 Thermal shock tests

Thermal shock parameters, such as heating time, cooling time, number of cycles, etc. were defined from analysis of European and Brazilian Standards [18-23] and experimental works [23]. Selected parameters for thermal shock tests are shown in Table 4:

<table>
<thead>
<tr>
<th>Test parameter</th>
<th>Defined parameters for tests</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximal surface temperature</td>
<td>70°C</td>
<td>[22]</td>
</tr>
<tr>
<td>Minimal surface temperature</td>
<td>23°C (environment)</td>
<td>[22]</td>
</tr>
<tr>
<td>Heating time</td>
<td>3 h</td>
<td>[23]</td>
</tr>
</tbody>
</table>
As shown maximal surface temperature used was 70°C obtained by lamps heating. Cooling was produced by water injection from sprinklers. Temperature control was made with thermocouples connected to an acquisition equipment. Heating and cooling structures are shown in figure 7.

Figure 7: Heating and cooling of rendering samples.

5 RESULTS.

5.1.1 Fresh mortar properties
Air content and fresh mortar density were measured in cement mortar used for tests as shown in Table 6:

Table 6: Properties of fresh cement mortar

<table>
<thead>
<tr>
<th>Properties</th>
<th>Test method</th>
<th>Cement mortar 1:4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh mortar density</td>
<td>[13]</td>
<td>1.99</td>
</tr>
<tr>
<td>Air content (%)</td>
<td>[13]</td>
<td>5.20</td>
</tr>
</tbody>
</table>

5.1.2 Hardened mortar rendering
Pull-off test were made on cement mortar rendering samples which results are shown in table 7:
Comparing mean values of pull-off tests considering statistical parameters (figure 8) it is possible to verify a drop in bond strength with samples exposed to thermal shock in both situations: in rendering samples with and without created interface flaws. When interface flaws don’t interfere, decreasing of bond strength caused by thermal shock is 63.3%.

![Figure 8: Bond strength results for rendering samples exposed to thermal shock](image)

However, existence of created interface flaws has a greater influence in decreasing of bond strength (70.6%) and understates thermal shock effect reducing drop in bond strength in samples with interface flaws to 34.4%.

6 CONCLUSIONS

Experimental tests showed that thermal stresses caused by environment thermal shock in surface of cement mortar renderings have a decreasing effect on its bond strength. Results showed durability of adhesion of cement mortar renderings are affected by cyclical events of thermal shock even with not severe temperature changes.

In the other hand, experimental simulation of interface flaws between mortar and substrate agreed with numerical simulations.

7 REFERENCES

EFFECT OF CARBONATION ON THE CHLORIDE DIFFUSION OF MORTAR SPECIMENS EXPOSED TO CYCLIC WETTING AND DRYING

Raphaele Malheiro(1), Aires Camões(1), Rui Miguel Ferreira(2), Gibson Meira(3), Teresa Amorim(4)

(1) Department of Civil Engineering University of Minho, Guimarães – raphamalheiro@gmail.com; aires@civil.uminho.pt
(2) Technical Research Centre of Finland, Espoo – miguel.ferreira@vtt.fi
(3) Federal Institute of Education, Science and Technology of Paraíba, João Pessoa – gibsonmeira@yahoo.com
(4) Department of Textile Engineering University of Minho, Guimarães – mtamorim@det.uminho.pt

Abstract

Carbonation and chloride ingress are the two main causes of corrosion in reinforced concrete structures. Despite the combined action of these mechanisms being a reality, there is little research on the effect of carbonation on the chloride diffusion in concrete. This work intends to study the influence of carbonation on chloride diffusion of mortar specimens. Cubic mortar specimens were cast with 0.55 of water-cement ratio. After curing, the specimens were subjected to 56 days of wetting and drying cycles. Half the samples were immersed for a day in a 3.5% NaCl solution and then placed for 6 days in a carbonation chamber (4%CO₂); the other half were also kept a day in 3.5% NaCl, but after were kept during 6 days in laboratory environment. Afterwards, the depth of chlorides and CO₂ penetration was evaluated. Complementary tests were also carried out, such as rapid chloride migration coefficient and water capillary absorption. The results show that carbonation has a direct influence on chloride penetration, decreasing it. The noncarbonated samples showed a profile with higher amount of chloride than carbonated ones. This fact can be related to the refinement of the mortar pores caused by carbonation and observed in water absorption tests.

Key words: chloride, carbonation, mortar, deterioration, combined degradation, accelerated testing
1  INTRODUCTION

A significant number of concrete structures around the world have been damaged by steel reinforcement corrosion. Among the factors that contribute to this kind of damage is the aggressiveness of chloride ions, one of the main reasons for damage to ordinary concrete structures due to reinforcement corrosion [1].

According to Tilly and Jacobs [2] simultaneous chloride ingress and carbonation account for over 50% percent of the deterioration of concrete structures. It is well known that the corrosive effect of chlorides is based on their ability to destroy the electrochemical condition of the passive state of reinforcement even under the existing alkalinity in the concrete. Regarding carbonation, the corrosive effect is based on the decreasing trend of alkalinity of pore liquid in concrete and the resulting depassivation of steel, enabling the conditions for corrosion to occur.

The service life of concrete structures is directly affected by its durability. Thus, several authors have been studying the action of chloride ions and carbonation [3, 4]. However, previous studies were confined mostly to the deterioration of concrete structures under a single deteriorating factor, although the real environment is actually a combination of factors [5].

Recently research has begun to look into the effect of combined degradation mechanisms. Chloride ingress and carbonation are among the most studied, however, there is no consensus on the effect of the combined action of chlorides and carbonation on the durability of concrete. In their experimental research, Chengfang et al. [6], for example, conclude that after carbonation, chloride diffusivity coefficient increases and it will increase with the increasing of carbonation time. On the other hand, Backus et al. [7], for example, say that the combination of carbonation with the entrance of chloride can act reducing the penetration of chlorides in concrete. However, the lack of standardization about the various parameters involved in the tests such as type of test, temperature, humidity and concentration of NaCl and CO₂ makes it difficult to compare results.

This experimental work studies the influence of carbonation on chloride transport into mortar specimens using accelerated tests.

2  EXPERIMENTAL WORK

Cubic mortar specimens with 50x50x50mm³ were cast with ordinary Portland cement (CEM I 42.5) based mortar manufactured with 0.55 of water-cement ratio and a common river sand. Chemical composition of cement is presented in Table 1. Mortar mixture and its main characteristics in fresh (flow table test) and hardened state (compressive strength at 28 and 90 days, open porosity and permeability to oxygen and water) are presented in Table 2.
Table 1: Chemical composition of cement

<table>
<thead>
<tr>
<th>Composition</th>
<th>Portland cement</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>19.65</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>4.28</td>
</tr>
<tr>
<td>FeO$_3$</td>
<td>3.35</td>
</tr>
<tr>
<td>CaO</td>
<td>61.35</td>
</tr>
<tr>
<td>MgO</td>
<td>1.7</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>3.36</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>0.89</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>0.19</td>
</tr>
<tr>
<td>Insoluble residue – IR</td>
<td>1.7</td>
</tr>
<tr>
<td>Loss on ignition – Li</td>
<td>2.82</td>
</tr>
</tbody>
</table>

Table 2: Mixture and properties of mortar

<table>
<thead>
<tr>
<th>Material and Properties</th>
<th>Mortar</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement: sand (kg)</td>
<td>1:3</td>
</tr>
<tr>
<td>Flow value (mm)</td>
<td>190</td>
</tr>
<tr>
<td>Compressive strength (MPa) – 28/ 90 days</td>
<td>37.6/ 39.1</td>
</tr>
<tr>
<td>Open porosity (%)</td>
<td>21.4</td>
</tr>
<tr>
<td>Oxygen permeability (K) - 10$^{-16}$ m$^2$</td>
<td>0.797</td>
</tr>
<tr>
<td>Water permeability (K$_w$) - m$^2$</td>
<td>11.16</td>
</tr>
</tbody>
</table>

After casted, these specimens were covered with a plastic sheet and they were stored, for one day, in a controller chamber (21ºC and 98%RH). After, they were removed from the moulds and they were cured for 28 days in wet a chamber (21ºC and 100%RH). Completed curing the specimens one had painted five of their six faces with an epoxy resin (3 days). Finished painting the specimens were drying more 4 days in laboratory environment (20ºC and 60%RH).

After this, in order to simulate the combined action of chloride ions and carbonation, the specimens were subjected to wetting and drying cycles during 56 days. Half of the specimens were immersed for a day in 3.5% NaCl solution and then placed for 6 days in a carbonation chamber (20ºC, 55%RH and 4%CO$_2$); the other half specimens was used as reference specimens and were also kept a day in 3.5% NaCl solution, but after were stored 6 days in laboratory environment, Figure 1.
After the exposure period, the depth of penetration of chlorides and CO\textsubscript{2} was determined. To evaluate the chloride penetration, a chloride profile was determined with 5mm interval until 30mm depth. Total chloride content was measured using the Volhard’s method [8]. To evaluate the carbonation depth, the specimens were splitted along the direction of penetration of CO\textsubscript{2}. Afterward, the fractured surfaces were sprayed with phenolphthalein and then the carbonation depth was measured [9]. Complementary tests were also carried out such as rapid chloride migration coefficient [10] and water capillary absorption [11].

3 RESULTS

3.1 Wetting and drying cycles

Figure 2 shows chloride profiles obtained after wetting and drying cycles. Each point depicted represents the average value determined from three samples for each depth.
The chloride penetration achieved for specimens under combined cycles (A) was lesser than the one achieved for specimens under chloride cycles (C). This condition is more pronounced on the surface but it is maintained along almost entire profile.

The carbonation average depth obtained to specimens under combined cycles was $4.9 \pm 0.04$ mm.

3.2 Complementary tests

The rapid chloride migration coefficient was evaluated for two types of specimens: specimens that were kept 56 days in a carbonation chamber only and specimens that were stored 56 days in a laboratory environment. The rapid chloride migration coefficient obtained for carbonated specimens was $8.35 \times 10^{-12}$ m$^2$/s, while the reference specimens showed higher results, $15.15 \times 10^{-12}$ m$^2$/s.

Figure 3 shows capillary absorption results obtained after the wetting and drying cycles. Each point depicted represents the average value from three specimens. The capillary absorption obtained for specimens under combined cycles (A) is bigger than results obtained for specimens under chloride cycles (C).

![Figure 3: Water capillarity absorption to the situations studied](image)

4 DISUSSION

4.1 Influence of carbonation on chloride penetration

The carbonation can reduce the capacity of chloride binding [12] and, consequently, lead to an increase in the rate of chloride ion ingress. On the other hand, carbonation can reduce the porosity and, consequently, lead to a decrease in the rate of chloride ion ingress. The chloride profiles presented in Figure 2 clearly show the influence of carbonation on chloride penetration for the studied mixtures. In this case, the carbonation acts by reducing the amount.
of total chlorides present throughout the depth of the specimens. This fact can be related to the refinement of the pores of the mortar caused by carbonation.

There is a consensus that carbonation promotes microstructural changes that culminate in the densification of pores. The carbonation reaction gives rise to calcium carbonate (CaCO$_3$) which solubility is limited and, therefore, tends to precipitate during the carbonation process [13]. This precipitation, at an initial phase, results in the aforementioned pore densification and consequent reduction of permeability.

The reduction in the permeability of the carbonated mortar can be confirmed by the water capillarity absorption results showed in Figure 3. It is possible to observe that the situation where the specimens were subjected to carbonation are those with a smaller water capillary absorption.

It is important to note that capillary absorption plays a key role in the wetting and drying cycles. In these tests, the chloride profile is generally different from those in which the relative humidity is approximately constant, Figure 4. For wetting and drying cycles there is a tendency to the formation of chloride’s peaks in the region close to the surface. These peaks are typical formations of the cycles of gain and loss of moisture in the material surface [14].

![Figure 4: Simulation of chloride penetration in an environment with constant relative humidity (RH) and environment subjected to wetting and drying [13]](image)

The chloride profiles presented in Figure 2 show the expected configuration for this type of test. However, the difference between the porosity of carbonated and non-carbonated zones may have increased the chloride peak observed in the combined cycle profile (A). The formation of a peak of chlorides after the carbonation front had already been verified by Lee and Yon [4] in a study on the deterioration of concrete, taking into account the combined action of carbonation and chloride ions. The skin effect cited by Andrade et al. [15] may explain this behaviour.
Concrete skin is considered the closest zone to the surface of concrete cover of reinforcements. It usually has a different composition than the internal concrete due to wall-effect phenomena or segregation of aggregates. In addition, environmental actions induce a gradient of moisture along the cover depth. These circumstances sometimes produce an irregular chloride profile in the cover, which either exhibits a maximum of chloride content some millimetres inside the outer surface or sometimes shows an anomalously high chloride concentration right at the concrete surface [15].

The extension of the test period and thus the carbonation depth may help to indicate the actual cause of the formation of the chloride peak observed in the combined cycle profile (A).

Concerning migration test, the results obtained emphasize the idea that the carbonation acts reducing the penetration of chlorides in mortars subjected to the combined action of the carbonation and chloride.

4.2 Test time and humidity control

Based on the obtained depth of carbonation, the carbonation front needs a longer test time to accentuate its depth and to clearly interact with the mechanism of chloride penetration. The time influence on the wetting and drying cycles was also observed by Lee et al. [5] for specimens subjected to the combination of carbonation and chlorides. Furthermore, the prolongation of the test time can cause leaching of precipitated calcium carbonate which may lead to open pores and cause an opposite effect.

Another important factor for the development of the carbonation front is moisture control. The drying time used (6 days) may have not been sufficient to provide a moisture content that could allow a maximum advance of the carbonation front inside the specimens. The presence of epoxy resin on five of the six faces of the specimens may have contributed to this behaviour.

5 CONCLUSION

Based on the obtained results one may conclude that the chloride profiles presented clearly show the influence of carbonation on chloride penetration for the studied mixtures. In this case, carbonation reduces the amount of total chlorides present throughout the depth of the specimens. The diffusion coefficients of chloride obtained by the migration test corroborate this idea as they present lower values for carbonated samples than for those non-carbonated.

These statements, together, suggest that the passage of chloride ions through the carbonated zone of studied mortars becomes more difficult. Once the carbonated zone is on the surface of the specimen, it seems that this difficulty leads to a decrease of the initial amount of chlorides which can penetrate the sample and consequently reach its interior. However, more prolonged exposures can cause leaching of precipitated calcium carbonate which may lead to an increase in open pores and can cause an opposite effect.
ACKNOWLEDGMENTS

Authors thank the foundation for science and technology (FCT) for supporting this research.

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ACCELERATED CLIMATE AGEING OF WOOD SURFACE TREATMENTS AND THEIR EVALUATION BY FOURIER TRANSFORM INFRARED SPECTROSCOPY

Bjørn Petter Jelle (1,2), Selamawit Mamo Fufa (1) and Maria Bjørn Olsen (2)

(1) SINTEF Building and Infrastructure, Department of Materials and Structures, NO-7465 Trondheim, Norway – bjorn.petter.jelle@sintef.no; selamawit.fufa@sintef.no

(2) Norwegian University of Science and Technology (NTNU), Department of Civil and Transport Engineering, NO-7491 Trondheim, Norway – bjorn.petter.jelle@ntnu.no; maria@elo.no

Abstract

Various surface treatments are applied in order to protect exterior building wooden claddings towards degradation from natural outdoor climate exposure. Development of new surface treatments, e.g. by applying nanotechnology, may increase the durability of the stain and paint systems themselves, hence offering a better protection and thus a longer lifetime of the wooden claddings.

In this study several surface treatments for wooden claddings like stain, opaque stain, paint and paints with nanoparticle modifications have been investigated when subjected to both natural outdoor climate ageing and accelerated climate ageing. The objective of the study was twofold, i.e. to investigate if the nanoparticle modified paints performed better with respect to durability versus their traditional counterparts, and furthermore to evaluate to what extent Fourier transform infrared (FTIR) spectroscopy may be utilized for assessing any degradation during elapsed ageing time.

1 INTRODUCTION

Wooden claddings are used in many buildings worldwide. These wood materials are then normally protected in various ways towards the outdoor climate exposure, often by applying different surface treatments like e.g. impregnations and paints. The durability of these treatments then becomes of major importance as they are directly influencing the durability of the wooden claddings and in some cases the buildings themselves.

Hence, development of new surface treatments is and will be an important task. A group of emerging paints is the so-called nanoparticle modified paints or for short nanopaints, claiming to exceed the property specifications of traditional paints, e.g. with respect to protection level and durability.
Selecting the appropriate test methods with suitable test conditions for these newly developed surface treatments including e.g. nanopaints will then be crucial. A key strategy is to carry out accelerated climate ageing in the laboratory [1,2]. Furthermore, characterizing important properties before, during and after the accelerated ageing is also of major importance [1,2]. Conducting a robustness assessment [3] of these surface treatments may also be found to be beneficial.

One of these versatile and powerful characterization tools is Fourier transform infrared (FTIR) spectroscopy [4-5], revealing if any chemical reactions have occurred in the tested materials, i.e. detecting any change in chemical bonds with regard to both formation and disappearance of these bonds (i.e. qualitative analysis), and also increasing or decreasing amounts of these bonds (i.e. quantitative analysis). In general, the FTIR method may be utilized for various tasks and materials, e.g. polymers or plastics [6], wood [7-8], wood rot decay and fungus growth [9-10] and various wood impregnations and paints also including paints modified with nanoparticles [11-15]. For further information about the FTIR material characterization technique applied on miscellaneous materials it is referred to the available literature [16-21].

The objective of this work is to investigate if nanoparticle modified paints perform better with respect to durability versus their traditional counterparts, and furthermore to evaluate to what extent FTIR spectroscopy may be utilized for assessing any degradation during elapsed ageing time.

2 EXPERIMENTAL

Two types of nanoparticle modified paints were applied on wood samples and studied in the investigations presented here. Furthermore, for comparison purposes, wood samples treated with commercial stain, deep coloured (opaque) stain and paint, and an untreated wood reference sample, were studied. All the investigated wood samples were spruce samples. The sample details are summarized in Table 1.

Table 1: Sample details, all surface treatments being performed on spruce.

<table>
<thead>
<tr>
<th>Sample notation</th>
<th>Sample name</th>
<th>Primer</th>
</tr>
</thead>
<tbody>
<tr>
<td>NF</td>
<td>Samicolor NanoBois (118F)</td>
<td>-</td>
</tr>
<tr>
<td>NN</td>
<td>Samicolor NanoBois Nature</td>
<td>-</td>
</tr>
<tr>
<td>U</td>
<td>Untreated wood reference (spruce)</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>1 coat Butinox 1 oljebeis (oil-based stain)</td>
<td>1 coat Butinox oljegrunning (oil-based primer)</td>
</tr>
<tr>
<td>D</td>
<td>1 coat Butinox 2 oljedekkbeis (oil-based opaque stain)</td>
<td>1 coat Butinox oljegrunning (oil-based primer)</td>
</tr>
<tr>
<td>M</td>
<td>1 coat Butinox oljemaling (oil-based paint)</td>
<td>1 coat Butinox oljegrunning (oil-based primer)</td>
</tr>
</tbody>
</table>

The samples were exposed to various accelerated climate strains in several ageing apparatuses following different procedures and methods, in addition to natural outdoor ageing. However, for the results presented herein, all the samples were subjected to
Accelerated ageing carried out in an Atlas SC600 MHG Solar Simulator climate chamber with a 2500 W MHG lamp. The samples were tilted a bit from the horizontal (8 ± 2º) in order to let the water run off the surface. The samples were placed in the climate chamber with a distance of approximately 55 cm from the climate chamber glass ceiling to the samples, where the solar radiation intensity is reported to be 1200 W/m² at 100 % lamp power intensity. The UVA and UVB radiation intensities have been measured at various times during the ageing period to be within the interval 60-80 W/m² and 3-6 W/m², respectively. The UV measurements were performed with a radiometer/photometer Model IL 1400A (International Light) with an UVA sensor and an UVB sensor. Note that a 6 % UVA fraction (like in outdoor solar radiation) of the total solar intensity (1200 W/m²) yields 72 W/m² UVA radiation. The exposure duration consisted of 115 whole cycles of 24 hours, each cycle divided into 20 hours with a solar radiation intensity of 1200 W/m² and 4 hours with water spray and no solar radiation exposure. The two water spray nozzles gave each 0.5 dm³/min, i.e. 1 dm³/min in total, which gives approximately 1.7 dm³/(m²min) assuming an even horizontal water distribution at the sample location in the whole climate chamber. The temperature and relative air humidity were held constant at 63ºC and 50 % RH during the solar exposure and at 10ºC and close to 100 % RH during the water spray application.

The Fourier transform infrared (FTIR) material characterization (before and after ageing) was carried out with a Thermo Nicolet 8700 FTIR spectrometer with a Smart Orbit accessory, i.e. a horizontal attenuated total reflectance (ATR) accessory (single reflection) with a diamond crystal, in the wavelength range 4000 cm⁻¹ (2.5 μm) to 400 cm⁻¹ (25 μm) in an atmosphere with minimalized CO₂ and H₂O content through purging by a Parker Balston 74-5041 FTIR Purge Gas Generator. Each FTIR spectrum presented is based on a recording of 32 scans at a resolution of 4 cm⁻¹. In order to ensure satisfactory contact between the ATR diamond crystal and the sample, three or more FTIR spectra were recorded at various locations on the sample. The surfaces of the expanding weather stripping foam samples are relatively porous, which complicates accurate quantitative measurements (height of absorbance peaks) due to varying contact with the ATR crystal for the different samples. Air between sample and ATR crystal results in a weaker absorbance signal. Unless other conditions indicate otherwise (e.g. inhomogenities, impurities, etc.), the FTIR curves with the largest absorbance peaks represent the most correct measurements on one and the same sample with equal ageing time, and hence these curves are chosen as they are assumed to be the most correct ones. Qualitative measurements (location of absorbance peaks at wave numbers) do not represent a problem as long as the contact area is large enough to ensure a sufficient strong measurement signal. The FTIR spectra given in this work have not been ATR corrected, neither with respect to penetration depths nor absorbance band shifts, which both are dependent on the refractive indices of the sample and the ATR crystal (diamond in this case) and the angle of incident radiation. The penetration depth is in addition also dependent on the radiation wavelength, and increases with increasing wavelength (decreasing wave number). That is, non-corrected ATR spectra have much stronger absorbance bands at longer wavelengths (smaller wave numbers) than at shorter wavelengths (larger wave numbers). Note that it should always be stated if an ATR-FTIR spectrum has been ATR corrected or not, e.g. important during computerized database spectra comparison searches. As we in this work is solely comparing the ATR-FTIR spectra measured within this work, there is no need for performing any ATR corrections. Besides, the raw ATR-FTIR data in either transmittance or logarithmic absorbance mode is usually preferred. It should also be
noted that one often do not know the refractive indices of the samples which are measured, thus errors might be introduced in the ATR corrected spectra as the refractive index of the sample is an input parameter in the ATR correction.

3 RESULTS AND DISCUSSION

The six different sample types as given in Table 1, including the untreated wood sample, before ageing, after 1 week accelerated ageing, after 2 weeks accelerated ageing, and after 12 weeks accelerated ageing, are depicted as digital photos in Figure 1. A clear change in the visual surface appearance is observed already after 1 week of accelerated ageing.

Figure 1: Digital photos of the six sample types before ageing and after selected accelerated ageing times up to 12 weeks.
FTIR absorbance spectra in two selected spectral regions, for the NF, NN, and U samples (Table 1), during accelerated ageing in an Atlas SC600 MHG Solar Simulator climate chamber, are depicted in Figure 2. Likewise are the FTIR absorbance spectra for the B, D and M samples (Table 1) depicted in Figure 3. Various changes in absorbance peaks are observed during the accelerated ageing, which then correspond to changes in chemical bonds (i.e. deterioration) as these bonds are absorbing more or less of the infrared radiation. Note that the small epoxy-filled grooves at the lower end of the samples as seen in Figure 1 are outtakes for the FTIR measurements.

Figure 2: FTIR absorbance versus wave number in two selected spectral regions, for the (a, b) NF, (c, d) NN, and (e, f) U samples, during ageing in an Atlas SC600 MHG Solar Simulator climate chamber.
As the absorption of electromagnetic radiation, e.g. infrared radiation, follows the Beer-Lambert law, i.e. the radiation is decreasing exponentially with the penetration depth in the actual material, it is often helpful to plot the spectra on a logarithmic absorbance scale vs. wavelength. Hence, a representative spectrum is chosen from each of the samples and plotted on a logarithmic absorbance scale for quantitative studies. Mathematically and physically it follows that a doubling of the logarithmic absorbance, also called optical density, is interpreted as a doubling of material thickness or a doubling of concentration of absorption active agents.
As mentioned earlier there is observed a clear change in the visual surface appearance already after 1 week of accelerated ageing, as depicted in Figure 1. This rapid change in visual surface appearance correlates to the rapid disappearance of certain absorbance peaks in the FTIR spectra as given in Figure 2 and Figure 3. Already during the first week of ageing, for non-aged NF, NN and U samples the absorbance peak located somewhat above 1500 cm\(^{-1}\) almost disappears (and more gradual disappearance of a peak somewhat above 1700 cm\(^{-1}\)), whereas for non-aged B, D and M samples the absorbance peaks between 3000 to 2800 cm\(^{-1}\) mainly disappears. From Figure 1 it is seen that all samples have been substantially degraded with disappearance of the surface treatments after 12 weeks of accelerated ageing. During the 12 weeks ageing period, pronounced absorbance peaks start to grow up somewhat above 1600 and 1300 cm\(^{-1}\) for the NF, NN and U samples, i.e. exposed wood and its degradation. For the B, D and M samples several absorbance peaks (e.g. somewhat above 1700 cm\(^{-1}\), between 1500 to 1400 cm\(^{-1}\) and between 1300 to 1200 cm\(^{-1}\)) decrease gradually during the 12 weeks of accelerated ageing.

The FTIR method has been shown to be able to readily detect chemical changes in surface treatments of wood. However, as the surface treatments deteriorate during the ageing and thus causing increased exposure of the wood, it may be complicated to differentiate between the surface treatments and the wood itself. Furthermore, the nanoparticle modified paints did not demonstrate a significant improved durability compared to their traditional counterparts for the applied accelerated ageing exposure.

4 CONCLUSIONS

The Fourier transform infrared method has been shown to be a powerful and versatile tool monitoring chemical changes in surface treatments of wood, although to differentiate between the surface treatments and the wood itself is more complicated due to increased exposure of the wood as the surface treatments deteriorate during the ageing. However, for the applied accelerated ageing exposure, no significant improved durability was demonstrated by the nanoparticle modified paints compared to their more traditional counterparts.

REFERENCES


NANORENDERS ON BUILDING FACADES: TECHNICAL, ECONOMIC AND ENVIRONMENTAL PERFORMANCE

António Soares (1), Inês Flores-Colen (1) and Jorge de Brito (1)

(1) Instituto Superior Técnico, Civil Engineering, Architecture and Georresources Department, Lisbon - ortiz.soares@msn.com; ines@florescolen.com; jb@civil.ist.utl.pt

Abstract

The increasing market competition and consumers demand, associated with environmental concerns, has led to the creation of mortar formulations that increasingly differ from traditional ones in order to improve their performance and reduce their environmental impact. To fulfil this goal, the incorporation of new materials in mortar formulations has been studied by several authors.

With the development of nanotechnology, some of the properties of cementitious materials can be significantly improved through the study and modification of their structure or by adding nanoparticles or nanostructures materials.

However, due to the high cost currently associated with the use of nanotechnology and nanoparticles, their application in renders has been limited but should be investigated in order to optimize materials and procedures, minimizing production costs and maintaining the most important technical and environmental features of these materials.

This paper presents a literature review of studies on the application of nanomaterilas in renders, discussing their potential in improving different properties of the mortars and the advantages and drawbacks of the incorporation of each material in mortar taking into account a balance of the technical, economic and environmental aspects from a holistic performance’s point of a view.

1 INTRODUCTION

With nanotechnology the manipulation of materials with dimensions and precision between 0.1 and 100 nm became possible. At nanoscale, surface effects are more important than bulk properties, which can lead to an improved performance of materials properties [1] cited by [2]; [3].

The use of nanomaterials can improve some characteristics of the construction products like strength and durability and introduce new functions such as photocatalytic (self-cleaning, pollution reduction and anti-microbial ability), anti-fogging and self-sensing capabilities [4].
Therefore, the introduction of nanomaterials in traditional construction products can lead to construction materials with excellent properties, and make buildings with a more efficient performance [5] cited by [6]; [7].

Even though nanotechnology is not a new science or technology, only in the two last decades has there been an increase in research in this field [8]. Recently there have been some studies regarding the incorporation of nanomaterials in cement mortars, creating mortars with advanced properties to apply as building renders (nanorenders). Among the studies concerning cement-based materials with nanomaterials, this work focus the main ones regarding nanomaterials which can be applied in building renders and include carbon nanotubes, nanosilica, silica aerogel and titanium dioxide nanoparticles.

2 INCORPORATION OF NANOMATERIALS IN MORTARS

2.1 General considerations

In general, due to their nanosize, nanomaterials affect the strength, durability and shrinkage properties of Portland-cement paste and help to improve the quality and longevity of structures [9, 10]. Other improvements due to nanomaterials consist on preventing the initiation of microcracks through control of cracks at nanoscale with nanosized fibres [11], enhance the thermal performance [12] and give photocatalytic and self-cleaning properties to renders [4].

However, to achieve these improvements it is necessary a good dispersion of the nanoparticles in cement mortars matrix that is difficult to reach with nanomaterials, due to their high surface energy and strong interparticle forces, which could result in a poor dispersion leading to weak zones or potential areas with concentrated stresses [13 - 15] cited by [10].

Another concern with nanomaterials application is the possible risks they could have on health, among them lung inflammation or cancer and, because of that, they should be handled like a material with unknown toxicity [16, 17] cited by [2].

In this chapter some improvements with the incorporation of nanomaterials in cement-based materials will be shown and possible application in building renders will be described.

2.2 Carbon nanotubes

In order to prevent cracks at nanoscale the incorporation of carbon nanotubes (Figure 1) has been studied showing good results with small amounts of highly dispersed multiwalled carbon nanotubes that have a tensile strength and Young’s modulus that are hundreds and tens of times those of steel, respectively, associated with a higher aspect ratio that leads to an effectively arrest of the nanocracks [10, 11, 18].

![Figure 1: Carbon nanotubes: right schematics; left microstructure [2]](image-url)
Results show that with 0.5 wt% of cement concentration of carbon nanotubes it is possible to increase 10% to 25% the flexural strength and 19% the compressive strength, indicating that with a small amount of effectively dispersed carbon nanotubes it is possible to improve the strength of mortars [19 - 21].

According to Konsta-Gdoutos et al. [18], besides the reinforcement’s improvement, multiwalled carbon nanotubes can also improve the durability of the cement matrix because they reduce the fine pores, which results in a reduction of the capillary stresses. Thus, it is possible to achieve a good mortar for protection against chemical attack.

However, to achieve improvements in cement-based materials a good dispersion of the carbon nanotubes is required that, as shown in Figure 2, is difficult to accomplish, because of their poor interaction with cement resulting from their intrinsically hydrophobic nature and because of the tightly bound formation of agglomerates or bundles resulting from their production [22] cited [11]; [23] cited by [10]. Indeed it is very important to get a uniform dispersion of carbon nanotubes in the cement matrix [24] and this issue has been studied by several authors [10].

Despite the possible improvement resulting of the incorporation of carbon nanotubes in cement-based materials, the current cost is too high to allow their use in a large scale, so they are more likely to be used in very specific applications with ultra-high requirements (e.g. nuclear reactor containment vessels) [25]. However, the low-quality carbon nanotubes resulting from large-scale manufacture technology are enough to improve cement-based materials properties and, when used at low concentrations to reinforce concrete, can be economically feasible if compared with fibre reinforced concrete [26]; [27] cited by [10].

2.3 Nanosilica (nano-SiO$_2$)

With the increase of interest in understanding the effect of nanoparticles on cement-based materials several studies have emerged regarding the incorporation of nanosilica with good results in improving the compressive and splitting tensile strength [28] and reducing the permeability of cement mortar [29].

The nanoparticles of silica when mixed in cement-based materials induce an acceleration of cement hydration, increase pozzolanic activity, reduce pore size and improve interfacial bonding between the hardened cement paste and aggregate leading to stronger cement-based materials than those containing SiO$_2$ fume. Despite similar behaviours at advanced ages, these differences between cement-based materials with nanoSiO$_2$ and silica fume can occur due to the higher pozzolanic activity of nanoSiO$_2$ at early ages (Figure 3) in relation to silica fume [28, 29].
However, there are some studies that indicate a higher degree of cement hydration with silica fume than with nanoSiO$_2$ [33, 34] cited by [32].

The drawbacks of nanoSiO$_2$ are the adverse effect on cement-based materials workability due to the high specific surface area and the dispersion of the nanoparticles that can cause lower strength of mortars in later ages [35]. In order to counteract the difficulties in achieving a good distribution of nano-SiO$_2$, some authors suggest low percentage of particles (1–5 wt%) while others propose contents of 10 wt% by weight of cement [36] cited by [37].

### 2.4 Silica aerogel

Another important kind of nanomaterials is bulk insulating materials like aerogel. Aerogel have a very low density (between 3 kg/m$^3$ and 500 kg/m$^3$) and excellent thermal properties (0.01-0.02 W/(m.K)) [38]. The exceptional thermal insulation is also achieved thanks to the unique shape and small size (10-100 nm) of the largest number of its pores that hinder the circulation of gas though the pore structure of the aerogel [39, 40], making it possible to obtain a render with a thermal conductivity of 0.025 W/(m.K) with an incorporation of aerogel between 60 and 90 vol.% (Figure 4) [12].

However, this render costs about 40 €/m$^2$ to 80 €/m$^2$ more than a traditional render [41], which is too high for current applications. Meanwhile, there are studies of low cost aerogel production with environmentally benign techniques that can lead to a good environmental and economic product [38].
2.5 Titanium dioxide nanoparticles (TiO$_2$)

In search for environmental sustainability new materials have been developed to apply in cement-based materials that take advantage of recycled materials or add new functionalities to mortars [42, 43]. With the introduction of TiO$_2$ powder (Figure 5), with photocatalytic and self-cleaning properties, it is possible to achieve mortars that help to reduce the pollutants found in the air, have biocide properties and self-cleaning effects, leading to mortars that can maintain their aesthetic characteristics, such as colour, and decrease the environmental impact of construction materials [4, 10, 44]. Because these particles need light stimulation for catalytic action, they are normally applied in external renders or paints [6]. However, some studies [10] found that the self-cleaning and photocatalytic effect of TiO$_2$ may form harmful side products, which shows that more research on their effects is required.

![Figure 5: SEM photo of nano-TiO$_2$ [45]](image)

Taking into account the good properties of nano-TiO$_2$, the interest on their incorporation in cement has been increasing since nano-TiO$_2$ powders can be conveniently mixed with cement-based construction materials without additional treatments [46] and an acceleration of the hydration reaction was observed with the increase of the content of TiO$_2$ nanoparticles [4]. Usually, it is more efficient to use finer TiO$_2$ particles than higher contents. However, the use of fine particles may negatively affect the rheological properties, resulting in higher water demand and shorter setting time [47, 48] cited by [6]; [46]. According to [45], the fluidity in accordance to GB/T 2419-2005 decreased by 20% and 40% respectively when 5% and 10% cement was replaced by nano-TiO$_2$ and despite an increase of 45% of compressive strength of the mortars in the first day, it suffered a decrease of 10% and 19% respectively at 28 days.

The results of a life cycle analysis showed that TiO$_2$ mixed with cement have significantly higher costs than ordinary cement paste. On the other hand, due the acceleration of cement hydration, they can reduce the time of construction and lead to economic benefits. This features associated to a reduction of pollutant gas concentration improving the urban air quality show that, in the long-term, TiO$_2$ modified cement could be beneficial to decrease the initial higher environmental impact [4].

3 CONCLUSIONS

It has been shown that it is possible to improve various characteristics of mortars by introducing nanomaterials, as shown in Table 1. In general an increase of compacity of the mortars was observed due to the nanosize of the particles which fill the voids of mortar. However, in some cases, a decrease of workability of mortars was noticed due to the high specific surface area of the nanomaterials and there were some difficulties to get a good dispersion of nanoparticles in the mortar matrix.
Table 1: Effect of various nanomaterials on mortars

<table>
<thead>
<tr>
<th>Material</th>
<th>% Workability and mixing</th>
<th>Flexural strength</th>
<th>Compressive strength</th>
<th>Physical properties</th>
<th>Durability</th>
<th>Cost</th>
<th>Environment impact</th>
</tr>
</thead>
<tbody>
<tr>
<td>CNT</td>
<td>Low</td>
<td>▼</td>
<td>▲</td>
<td>▲ capillary stresses</td>
<td>▲</td>
<td>€€€</td>
<td>n.s.</td>
</tr>
<tr>
<td>Nanosilica</td>
<td>Low</td>
<td>▼</td>
<td>▲</td>
<td>▲ water permeability</td>
<td>n.s.</td>
<td>n.s.</td>
<td>n.s.</td>
</tr>
<tr>
<td>Silica aerogel</td>
<td>High</td>
<td>n.s.</td>
<td>n.s.</td>
<td>▲ thermal conductivity</td>
<td>n.s.</td>
<td>€€</td>
<td>▲ initial ▼ final</td>
</tr>
<tr>
<td>NanoTiO₂</td>
<td>Low</td>
<td>▼</td>
<td>n.s.</td>
<td>▲ initial ▼ 28 days</td>
<td>Photocatalytic and self-cleaning properties (new properties)</td>
<td>n.s.</td>
<td>€</td>
</tr>
</tbody>
</table>

Legend: CNT - carbon nanotubes; NanoTiO₂ - titanium dioxide nanoparticles; % - percentage of incorporation; ▼ - decrease; ▲ - increase; €€€ - very expensive; €€ - expensive; € - competitive cost; n.s. - not specified

A concern with the environment has been noticed concerning the use of nanomaterials in mortars. Nevertheless, despite their high initial impact, they can contribute positively in the long-term to making buildings more sustainable. However, more research will be necessary concerning the health impact of the application of nanomaterials in building renders.

Nowadays, nanomaterials have a high production cost, making them almost prohibitive to apply in a large scale in construction or current application. However, there have been efforts to reduce the production cost of some nanomaterials, but there is a considerable lack of information regarding the real costs of renders with nanomaterials. Therefore, it is important to conduct research on the effects of nanomaterials in mortars to identify the aspects to be improved and create the possibility of achieving, in the future, products to apply in renders with advanced properties at a low or competitive cost.

ACKNOWLEDGEMENTS

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REFERENCES


AN INVESTIGATION OF THE EFFECTIVENESS AND DURABILITY CHARACTERISTIC OF BIOMASS ASHES AS POZZOLANIC MATERIALS

Sotiris Demis (1), Conrado S. Rodrigues (2), Luciana A. Santos (2) and Vagelis G. Papadakis (3)

(1) Department of Civil Engineering, University of Patras, Patras, Greece - sdemis@upatras.gr
(2) Department of Civil Engineering, CEFET-MG, Belo Horizonte, Brazil, crodrigues@civil.cefetmg.br, lucianasantos@varginha.cefetmg.br
(3) Department of Environmental and Natural Resources Management, University of Patras, Agrinio, Greece, vgpapadakis@upatras.gr

Abstract

Utilisation of biomass ash from agro-industrial by-products in cement and concrete manufacturing can be an alternative solution to the incorporation of traditionally used supplementary cementing materials (SCMs). These agro-waste ashes, containing large amount of silica in amorphous form, have potential for use as pozzolanic materials replacing cement. An evaluation of a variety of biomass ashes with varying SiO₂ contents in terms of concrete strength (as approached by estimating the efficiency factors) and in carbonation and chloride penetration tests is the focus of this study. Results indicate that these materials are effective on concrete strength development and extremely efficient in reducing the concrete permeability and resistivity especially against chloride ingress. The dependency of this behavior on the level of SiO₂ of the ash content was examined in depth and was concluded that it is affected as well by the composition of other components of the biomass ash.

1 INTRODUCTION

Given the known environmental issues [1] of cement manufacturing, direct reduction of its clinker content through utilization of industrial by-products as supplementary cementing materials (SCMs), is a very promising step in reducing considerably the associated environmental burden. Various types of biomass, from agro-industrial processes, produce ash which under certain conditions (chemical configuration, level of fineness) can have a similar pozzolanic activity to coal fly ash [2-4]. These biomass ashes, containing a large amount of silica in amorphous form, have potential to be used as pozzolanic materials replacing cement.
Such an investigation is the aim of this study. By selecting data of representative biomass ashes from the literature, the feasibility of their utilization in cement manufacturing, as pozzolanic materials is investigated. An evaluation of the ashes in terms of the derived efficiency factors for 28 days compressive strength, further enhanced by their performance in chloride penetration (as defined in ASTM C 1202 [6]) is presented in this study. The main aim is to try to shed some light on the specific influence of the main characteristics of biomass ashes on concrete strength development and performance in chloride exposure.

2 TYPES OF BIOMASS ASHES INVESTIGATED

Based on data from the literature, a range of characteristic types of biomass ashes was selected. Their origin and chemical composition is briefly discussed below and presented in Table 1.

Table 1: Chemical Composition and characterization of ashes

<table>
<thead>
<tr>
<th>Type</th>
<th>SiO$_2$ (%)</th>
<th>Al$_2$O$_3$ (%)</th>
<th>Fe$_2$O$_3$ (%)</th>
<th>CaO (%)</th>
<th>SO$_3$ (%)</th>
<th>Na$_2$O (%)</th>
<th>K$_2$O (%)</th>
<th>LOI (%)</th>
<th>d$_{50}$ (mm)</th>
<th>Density (kg/m$^3$)</th>
<th>*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rice Husk Ash</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GRHBA [7]</td>
<td>87.0</td>
<td>1.08</td>
<td>2.58</td>
<td>1.25</td>
<td>0.09</td>
<td>0.08</td>
<td>3.0</td>
<td>5.71</td>
<td>10.8</td>
<td>2150</td>
<td>k</td>
</tr>
<tr>
<td>GRHBA [8]</td>
<td>74.8</td>
<td>0.20</td>
<td>3.80</td>
<td>9.00</td>
<td>0.50</td>
<td>3.20</td>
<td>2.0</td>
<td>11.2</td>
<td>10.8</td>
<td>2150</td>
<td>k</td>
</tr>
<tr>
<td>BRWA [9]</td>
<td>78.4</td>
<td>2.60</td>
<td>1.70</td>
<td>7.40</td>
<td>1.10</td>
<td>0.20</td>
<td>8.7</td>
<td>3.60</td>
<td>15.5</td>
<td>2100</td>
<td>k</td>
</tr>
<tr>
<td>RHA [10]</td>
<td>91.0</td>
<td>0.35</td>
<td>3.41</td>
<td>1.95</td>
<td>1.21</td>
<td>0.08</td>
<td>8.21</td>
<td>8.50</td>
<td>12.0</td>
<td>2060</td>
<td>k</td>
</tr>
<tr>
<td>RHA [11]</td>
<td>87.3</td>
<td>0.22</td>
<td>2.88</td>
<td>0.48</td>
<td>1.02</td>
<td>3.14</td>
<td>2.10</td>
<td>3.80</td>
<td>2060</td>
<td>k, RCPT</td>
<td></td>
</tr>
<tr>
<td>RHA [12]</td>
<td>92.9</td>
<td>0.31</td>
<td>2.66</td>
<td>0.53</td>
<td>0.08</td>
<td>2.06</td>
<td>1.97</td>
<td></td>
<td></td>
<td>RCPT</td>
<td></td>
</tr>
<tr>
<td>RHA [13]</td>
<td>91.6</td>
<td>0.35</td>
<td>0.40</td>
<td>0.60</td>
<td>0.30</td>
<td>3.60</td>
<td>3.70</td>
<td>7.41</td>
<td>1950</td>
<td>2050</td>
<td>RCPT</td>
</tr>
<tr>
<td>Palm Oil Fuel Ash</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>GPOFA [3]</td>
<td>65.3</td>
<td>2.50</td>
<td>1.90</td>
<td>6.40</td>
<td>0.40</td>
<td>0.30</td>
<td>5.70</td>
<td>10.0</td>
<td>19.9</td>
<td>2170</td>
<td>K</td>
</tr>
<tr>
<td>GPOFA [3]</td>
<td>65.3</td>
<td>2.50</td>
<td>1.90</td>
<td>6.40</td>
<td>0.40</td>
<td>0.30</td>
<td>5.70</td>
<td>10.0</td>
<td>10.1</td>
<td>2330</td>
<td>K</td>
</tr>
<tr>
<td>GPOFA [8]</td>
<td>65.3</td>
<td>2.60</td>
<td>2.00</td>
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<td>0.50</td>
<td>0.30</td>
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<td>10.1</td>
<td>10.1</td>
<td>2330</td>
<td>K</td>
</tr>
<tr>
<td>POFA [14]</td>
<td>55.5</td>
<td>9.20</td>
<td>5.60</td>
<td>12.4</td>
<td>2.30</td>
<td></td>
<td></td>
<td>7.90</td>
<td>10.7</td>
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<td>K</td>
</tr>
<tr>
<td>POFA [13]</td>
<td>63.6</td>
<td>1.60</td>
<td>7.60</td>
<td>0.20</td>
<td>0.10</td>
<td>6.90</td>
<td>6.60</td>
<td>7.20</td>
<td>2250</td>
<td>RCPT</td>
<td></td>
</tr>
<tr>
<td>Sugar Cane Ash</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>SCBA [15]</td>
<td>78.3</td>
<td>8.90</td>
<td>3.60</td>
<td>2.20</td>
<td></td>
<td>0.10</td>
<td>8.50</td>
<td>2.70</td>
<td>2530</td>
<td>K</td>
<td></td>
</tr>
<tr>
<td>SCBA [2]</td>
<td>64.2</td>
<td>5.05</td>
<td>5.52</td>
<td>8.14</td>
<td></td>
<td>0.92</td>
<td>8.35</td>
<td>4.90</td>
<td>1850</td>
<td>k, RCPT</td>
<td></td>
</tr>
<tr>
<td>SCBA [16]</td>
<td>78.4</td>
<td>8.55</td>
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<td></td>
<td>0.12</td>
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<td>k, RCPT</td>
<td></td>
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<tr>
<td>Wood Ash</td>
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<td></td>
</tr>
<tr>
<td>HCWA [18]</td>
<td>28.0</td>
<td>4.10</td>
<td>2.50</td>
<td>39.0</td>
<td>1.00</td>
<td>1.00</td>
<td>7.40</td>
<td>7.22</td>
<td>8.16</td>
<td>2520</td>
<td>k</td>
</tr>
</tbody>
</table>

$k, \text{RCPT}$ denote that the ash was used for strength or for Rapid Chloride Permeability Test assessment

Typical highly reactive types of rice husk ash (RHA), with SiO$_2$ content of more than 90% were selected [10-12]. Rice husk ash (RHA), an agricultural waste material, produced by controlled burning of rice husk has shown to contain highly reactive silica which could contribute chemically to Portland cement ingredients. Rice Husk Bark (RHBA) and bagasse-rice husk-wood ashes (BRWA), comprised of 35% eucalyptus bark (with 65% rice husk) and of 82.5% bagasse (and 2.5% chop wood with 15% rice husk) respectively, were also selected. Based on their chemical compositions and properties, according to ASTM C 618 [19], these particular types of biomass ash can be said to be Class N pozzolans (since the sum of SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$ are higher than or close to 70%, SO$_3$ content is not higher than 4%, and loss of ignition (LOI) is close to 10%). Palm oil fuel ash (POFA) is a by-product of the palm oil industry, containing high amounts of silicon and aluminum oxides (in the amorphous state), recently been accepted as a pozzolanic material [3]. According to the chemical composition of the types of POFA selected in this study [3, 8, 13-14], (SiO$_2$ composition: 55% - 65%, LOI ≈
10%), it can be said that, although POFA is not a natural pozzolan, it may be classified as a Class N (natural) pozzolan (according to ASTM C618 [19]).

Sugar Cane Bagasse Ash (SCBA) is a by-product of the sugar cane and alcohol production industry. Preliminary investigations on SCBA have demonstrated that it presents the appropriate chemical composition for application as a pozzolan, mainly due to its high silica content and presence of amorphous silica [16]. However, it is important to note that the SiO₂ content (78.3%) covers amorphous and crystalline silica and some sand contamination has been observed, identified by the presence of quartz and cristobalite phases in the diffraction patterns of SCBA [16]. Wood ash (WA) is the inorganic and organic residue generated due to combustion of wood and wood related products [17]. In a sense it can be related to fly ash since the latter is obtained from coal which is fossilized wood. The chemical composition of the selected types of WAs indicates very high LOI, high alkali content and very low SiO₂ values (hence do not satisfy the requirements to be classified as Class N pozzolans).

2.1 Methodology Followed for the Evaluation of Biomass Ashes

Based on, the chemical composition of these types of ashes, the mix design used in each particular study and the compressive strength data available from the literature, estimation of the compressive strength for a range of $k$-values [20], took place. Such calculations were possible using an analytical tool for the estimation of concrete strength and service life, well published [21-22], developed and validated by some of the authors of this study. For each type of biomass ash, different sets of compressive strength data were estimated for a range of $k$-values (from the very low value of 0.2 to higher values of 1.7). Each set of strength data, was evaluated in terms of statistical proximity to the experimental strength values, based on the principles of the least squares method. The $k$-value of the set of data that provided the best fit was assumed to be the efficiency factor (for 28 days compressive strength) of the biomass ash. In addition, where data were available, the performance of biomass ashes in terms of chloride ion penetration, expressed as the total charge passed through biomass ash blended concrete specimens (measured according to ASTM C 1202 [6]), were evaluated. Based on the values of charge (Coulombs) passed through each individual biomass ash concrete investigated, with increasing SCM content, the difference (%) to control (no SCM) was calculated, as it is illustrated in Section 3.2 of this study.

3 RESULTS

An overview of the calculated k-factors, per category of the biomass ashes investigated, is illustrated in Figure 1. A first observation is that the various origins and chemical compositions of the individual ashes resulted in different $k$-values. Overall, on average, a $k$-value of 1.38 was calculated for RHAs (and mixtures), while for POFA and SCBA the corresponding efficiency factors were found to be 1.2 and 0.5 (respectively). As it was expected WAs gave $k$-values below 0.2. In particular the following main observations can be made:

- RHAs and mixtures produced $k$-values above 1.3, up to 1.6 (except BRWA [9] with an RHA participation of 15%, ($k$-value of 1.0).
- The low level of fineness (expressed as the $d_{50}$) from 3.8 μm to 10.8 μm, does not seem to have a profound effect on the derived k-factors, since RHA of similar
compositions produced values of 1.5 to 1.6. However, for a median size of 12 μm, the derived efficiency factor was slightly lower (1.3).

Figure 1: k-values of biomass ashes

- Inconsistencies in behavior with the level of fineness were also observed on SCBAs, but for similar (low) d₅₀ values. A particular type of SCBA with a d₅₀ of 2.70 μm, produced a k-value of 0.5 [15], while another with a d₅₀ of 5.4 μm, produced a 0.8 k-value [2]. Such a difference can be attributed to the fact that the SCBA with the low k-value was rich in crystalline silica.

- The level of fineness appears to have some effect on POFA mixtures [3, 8, 13-14]. At POFAs of the same chemical composition, reduction of the d₅₀ from 19.1 μm to 10.1 μm [3] produced an increase on the k-values (from 1.2 to 1.5). However, another type of POFA with a similar level of fineness to 10,1 μm (10,7 μm) [14] produced a k-value of 1.0 (much lower than 1.5).

- POFAs with less SiO₂ than RHAs produced similar k-factors (1.3 [8], 1.5 [3]).

3.1 Chloride Penetration

The performance of biomass ashes under Rapid Chloride Permeability Test (RCPT, according to ASTM C 1202 [6]) was evaluated. The difference (%) to control (no SCM) in terms of charge passed per increasing SCM content is given in Table 2. Cement replacement by RHA drastically reduced the rapid chloride penetrability of concrete from a low (charge: 1000-2000 C) to very low (charge: 100-1000 C) ratings. In general RHA proved to be more drastic, than SCBA and POFA. Incorporation of 20% produced a reduction in the charge passed of more than 60% (and up to 90%), while at lower biomass ash quantities (10%) a steady reduction (for all RHA investigated) of 43% was noticed. Considerable reductions were also noticed for other types of biomass ashes (73.8 % for 20% POFA incorporation and 56.8 % for SCBA).
Table 2: Performance in RCPT in terms of reduction in electrical charge passed

<table>
<thead>
<tr>
<th></th>
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</thead>
<tbody>
<tr>
<td></td>
<td>(C)</td>
<td>(D)</td>
<td>(C)</td>
<td>(D)</td>
<td>(C)</td>
<td>(D)</td>
</tr>
<tr>
<td>0</td>
<td>2780</td>
<td>0</td>
<td>1179</td>
<td>33.6</td>
<td>1108</td>
<td>4.57</td>
</tr>
<tr>
<td>5</td>
<td>2045</td>
<td>26.4</td>
<td>783</td>
<td>34.4</td>
<td>653</td>
<td>33.6</td>
</tr>
<tr>
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</tr>
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<td>40</td>
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<td>-</td>
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<td>250</td>
<td>96.6</td>
</tr>
</tbody>
</table>

(C) Charge passed (in Coulombs), (D) Difference to the control value (%)

charge ranging from 1000 to 2000 C: low permeability, while from 100 to 1000 C: very low permeability

4 DISCUSSION

Trying to identify the specific influence of the main characteristics of the biomass ashes on the efficiency factors, it can be noted that, RHAs, rich in SiO$_2$ content (> 85%), produced high $k$-values, while wood containing ashes, with very low SiO$_2$ content (< 30%) produced the lowest $k$-values. Hence, it appears that the influence of SiO$_2$ should be quite significant on the derived $k$-value of the ash. However, considering that SCBA with higher SiO$_2$ than that of POFA produced smaller $k$-values, the influence of SiO$_2$ content is not straight forward. In the literature [20, 23] there is a consensus that it is the active silica (non-crystalline silica glass, present in the amorphous and mostly vitreous part of the SCM), part of the total silica content that is involved in the hydration reactions producing CSH, to which the strengthening of cement is attributed. In this study, information on the active silica content was unavailable. On this note, benchmarking of the ashes in terms of the influence of SiO$_2$ content and of the added influence of SiO$_2$ with the other primary oxides (Al$_2$O$_3$, Fe$_2$O$_3$), on the derived efficiency factors, was taken place and is partially illustrated in Figures 2a-c.

Although a trend, that with reducing SiO$_2$ content smaller derived k-values are derived is slightly evident (Figure 2a), certain “abnormalities” (points 4, 5, 6 and 11) distort this image. A closer look reveals that ashes with high SiO$_2$ content and low (up to 2.6 %) Al$_2$O$_3$ and Fe$_2$O$_3$ contents produced higher than 1.0 $k$-values. On the other hand ashes with high Al$_2$O$_3$ (more than 8%) and Fe$_2$O$_3$ (above 2.6%) contents, even if they are rich in SiO$_2$ (78%), they produced $k$-values bellow 0.8. The previously mentioned “abnormalities” refer to such types of ashes, namely SCBA (points 5 [16], 6 [15], 11 [2] in Figure 2a). In addition to the above, two further points strike out. A type of POFA [14] (point 12) with high Al$_2$O$_3$ and Fe$_2$O$_3$ contents but also rich in CaO (12.4%) and a mixture type of RHA [9] (point 4) containing a very small portion (15%) of rice husk wood ash, being low in Al$_2$O$_3$ and Fe$_2$O$_3$ contents but high in CaO (7.4%). Hence the influence of the level of SiO$_2$ in the ash does carry a certain validity on the derived value of the efficiency factor, but is also affected by the composition of the ash in other oxides. On that note, the influence SiO$_2$ in connection to the other major oxides (Al$_2$O$_3$ and Fe$_2$O$_3$) was examined (Figures 2b,c). The same trend/mishaps were observed as in the case of the effect of SiO$_2$ content.
Figure 2: Effect of primary oxide contents on $k$-values of biomass ashes; [a] SiO$_2$ (%), [b] SiO$_2$+Al$_2$O$_3$ (%), [c] SiO$_2$+Al$_2$O$_3$+Fe$_2$O$_3$ (%), [d] Al$_2$O$_3$ (%), [e] Fe$_2$O$_3$ (%) and [f] CaO (%)

Up to a certain percentage in Al$_2$O$_3$, Fe$_2$O$_3$ and CaO, high SiO$_2$ content translates into a high $k$-value. At high compositions of CaO (even for low Al$_2$O$_3$, Fe$_2$O$_3$ contents) and/or at high contents of Al$_2$O$_3$, Fe$_2$O$_3$, the derived $k$-values are not “linear” with that high SiO$_2$ content. The way Al$_2$O$_3$, Fe$_2$O$_3$ and CaO contents influence the level of the derived $k$-value, (even for a high contained percentage of SiO$_2$ in the ash) is evident into Figures 2d-f. On the range of biomass ashes investigated in this study, of similar particle median size ($d_{50}$) and LOI, concentration in Al$_2$O$_3$ of more than 4.1% produced efficiency factors below 1.0, even for high SiO$_2$ content (more than 78%), as it illustrated at points 1 [16] and 2 [15] in Figure 2d. In terms of Fe$_2$O$_3$ (Figure 2e) and CaO (Figure 2f), concentration of more than 3.0 % and 8.0 % respectively, produced efficiency factors well below 1.0. It appears that as their percentages in the ash increase, the derived $k$-values drop below 1.0, well even bellow 0.5.

On the influence of CaO it should be noted that results are contradictory. According to the literature, high CaO contents in the ash are expected to contribute to hydraulic activity [24]. The later enhances the formation of C-S-H gel, through the reaction of amorphous silica content of the ash with the free lime from cement hydration [2]. However, high carbon contents can lead to increased water demand of the concrete mix [8, 10].

Although, relatively high alumina contents (and iron oxide) in cement in the form of C$_3$A or C$_4$AF phases, are known for their small contribution to early strength development, their influence in the chloride binding capacity of the cement mix is crucial. High alumina contents
increase cement’s potential for Friedel’s salt production. This process is beneficial, as far as the permeability of chlorides is concerned, since formation of Friedel’s salt results in a less porous structure (reduced “transport” of chlorides into the concrete mix). Such a less porous structure, was observed on a variety of the biomass ashes (RHA, POFA and in some SCBA) investigated in this study, based on the change of the reduction of chloride permeability from “low” to “very low” rating (according to ASTM C1202), as it was discussed in the previous section. In general, the transport of chloride ions (and the consequent electrochemical processes) through biomass ash blended concretes depends on the pore structure of the concrete, while the electrical conduction depends on both pore structure characteristics and electrical conductivity of the pore solution [11]. The finer particles of ashes develop discontinuous and tortuous pore in concrete structure and the pores present in the concrete are completely filled up by finer particles. Since the total charge passed through the concrete depends on the electrical conductance, low unburnt carbon content present in some types of biomass ashes is known to contribute to the significant reduction in the electrical charge passed. When RHA, which has a lower LOI value compared to OPC, was used to partially replace OPC, resistance to chloride permeation was substantially improved. This may be attributed to a decrease in electrical conductivity of concrete due to lowering of unburnt carbon content in RHA, in addition to pore structure refinement and conductivity of pore solution. Such an observation is further reinforced considering the substantial reductions in charge passed achieved when RHA was utilized with low LOI values from 1.97% to 3.7%.

Overall, the chloride-ion penetration results suggest pore refinement due to the pozzolanic reaction of ultrafine biomass ash and demonstrate the significant potential of these types of ashes as mineral admixtures in concrete, as long as an appropriate grinding strategy is used and product fineness is achieved.

5 CONCLUSIONS

In the current study by investigating the efficiency of a range of biomass ashes, identified in the literature, as cementitious materials, in terms of efficiency factors for concrete strength and chloride penetration, it can be concluded that:

- High SiO₂ content in the biomass ash, does not automatically imply an effective pozzolanic material (high efficiency factor).
- It appears that after certain percentages in the concentration of the ash in Al₂O₃, Fe₂O₃ and CaO contents (even for high SiO₂ content, high level of fineness, and low values of LOI) the derived efficiency factors drop below 1.0.
- Concrete impermeability properties are considerably improved due to pore refinement in biomass ash blended concretes compared to control concrete.
- Biomass ashes (except WAs) from a variety of agro-industrial by-products can be used as cement replacement materials with beneficial results in strength development and performance in chloride penetration.

It is hoped that the results of this study will pave the way for a more in depth evaluation of a wider range of biomass ashes on cement and concrete production.

ACKNOWLEDGEMENTS

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Abstract [Max 200 words]

The External Thermal Insulation Composite Systems with rendering (ETICS) are a building envelope technology widely used both in new construction interventions and in energy refurbishments. Installed ETICS display multiple failure modes, which are often the result of the repeated action of multiple agents inducing cyclic stresses and strains, thus fatigue. In this study, to predict the performance decay over time and to assess the durability, we coupled two numerical models: a hygrothermal model, computing the Heat and Moisture Transport (HMT) in porous media, coupled to a Thermo-Mechanical Finite Element Method model (TM-FEM). The transient temperature profile computed with the HMT model was the input for the TM-FEM simulations, with which we assessed the thermal stress over time, and the frequency of the events exceeding the critical stress thresholds, beyond which the base coat is subject to fatigue. As variables, we considered the moisture response of the base coat, and the optical properties of the finishing coat, in a set of Southern European climates. Especially we focused our attention on the frequency of thermal shocks and freezing-thawing cycles in HMT analysis as the main causes of cracks in case of temperature variation in a preliminary 3D TM-FEM model analysis.

1 INTRODUCTION

External Thermal Insulation Composite Systems with rendering (ETICS) are a very popular building envelope technology, which consist of an insulation board bonded onto a new or existing masonry or concrete wall, and mechanically fixed with anchors. The insulation board is then protected by a base coat, usually 3-4 mm thick, made of the cement mortar with resin used as adhesive, but reinforced with a glass fibre mesh. Onto the base coat is applied a 'key coat', or primer, and then the finishing, which is usually a thick (1.5-2 mm) top coat [1]. This technology is very common all over Europe, since its low cost and since it
allows to continue to use a building while it is being refurbished. However, failures of ETICS are widely documented [2][3].

After a previous analysis of the mainly failure modes which occur in ETICS (cracking, detachments, bulging, wrinkling, and biological growth) and thanks to a previous survey, we saw that cyclic thermal shocks, water absorption and sometimes freeze-thawing are, in Southern Europe climates, the most relevant mechanisms that cause degradation, loss in performances, and failures of ETICS [6]. Whether the relevance of their effects on ETICS is known less studied, instead, are both the intensity of the shocks, which shall be reproduced in the laboratory, and their frequency in different climate areas. In fact, intensity and frequency are fundamental knowledge to study the durability of objects exposed to cyclic stress. Moreover, among the different loads that ETICS bear, the stresses due to thermal variation are most of the time much higher than the ones due to gravity. Both night and day temperature changes and seasonal fluctuations cause thermal movements and, as a consequence of different material properties, stresses inside the insulation boards and in base and top coat. Although ETICS have been used for years, there is still a lack of understanding reasons and mechanisms of different failure cases and, even if there have been some attempts to study the failures, mainly by numerical simulation (especially by the Finite Element Method), most of these attempts didn’t succeeded because of the low availability of the necessary material properties, and especially of fracture mechanical properties [7].

This work concerns the analysis carried out by means of simultaneous Heat and Moisture Transport (HMT) numerical simulations of ETICS with mineral wool (MW) or expanded polystyrene (EPS), and different base and top coats. The aim is to highlight the different behaviour in terms of frequency and intensity of events that may be critical for durability, in a set of Southern European climates. Thus, the HMT analysis provides the temperature distribution through the layers, which is used as input data for the finite elements thermo-mechanical simulations (TM-FEM) that provide the stress-strain.

2 CALCULATION MODEL AND INPUT DATA FOR HMT

We performed a parametric study to assess the number of events (thermal shocks, water absorption and freeze-thaw) that cyclically repeated could jeopardize the service life of the system (i.e. about 35 years for an ETICS). To assess the intensity and frequency of these critical events, we used the software tool WUFI 5.2 (developed by the Fraunhofer Institute for Building Physics), [8] compliant with EN 15026 [9] benchmark test validation requirements. WUFI uses the finite control volumes method to resolve numerically the transient heat and moisture transport in building materials. Weather data for the selected representative cities for Southern Europe were obtained from the Meteonorm database. Solar absorbance (α) was varied between 0.10 and 0.90 (0.01 and 0.99 are used just as ideal lower and upper limits). The ETICS was modelled for two years oriented in the four cardinal directions. Just the data for the second year were analyzed, when the moisture content achieved its long-term trend.

3 HYGROTHERMAL ANALYSIS

3.1 Thermal shocks and moisture content

For the thermal shock analysis we considered the differences in surface temperature between two time steps (i.e. one hour), counting the sudden temperature variations over one year (Figure 1). We computed the thermal shock events combined with the moisture content
in terms of liquid water occurrence in the base-coat (more sensible to capillary water absorption) with a value greater than 50% of the free water saturation. This was assumed as the threshold above which there is a reduction of mechanical resistance (compressive and tensile strength), as presented also in [10]. With low water contents in the base coat, these events do not occur frequently in the Southern European climates that we analyzed. In general, the absolute number of thermal shock events per year is higher in Southern European contexts, such as Barcelona, than in Atlantic contexts, such as Nantes. Events comprising a sudden surface temperature variation coupled to a rain event are computed to be twice more frequent in Barcelona than in Nantes.

![Figure 1: Thermal shocks with temperature variation greater or equal to 20°C, Milan](image)

### 3.2 Freeze-thaw

Predicting the damage due to freeze-thaw cycles is a critical point due to two main factors:

- The critical temperature for which freezing occur in the material pore structure, because it is conditional on dissolved salt presence and on the pore dimension;
- The critical moisture content in the material, above which, if it is freezing, it will cause the damage due to the pressure generated by expansion in becoming solid and it depends also on the pore size distribution and interconnection.

We assumed the freezing temperature of water in the pores below -5°C, after the Literature on the freeze-thaw in concrete [11]. We counted the occurrences when the temperature in the base coat – the layer that is more exposed to freeze-thaw – drops below -5 °C, then rises above to 0 °C, and the water content is above the critical value (Figure 2), as done also in [12]. In our case the critical moisture thresholds are 70%, the 80% or the 90% of free water saturation. With low moisture contents in the base coat temperature not often lower than -5°C, the freeze-thaw cycles do not occur for the cities of Southern Europe with solar absorbance greater than 0.2, as showed for Milan. In a city further north, such as Zurich, they occur for absorbance up to 0.6.
3.3 Comparison between ETICS with EPS and MW

We modelled the hygrothermal response of two identical ETICS assemblies, except for the insulating material. In one case, we included mineral wool (MW), in the other case, we considered an expanded polystyrene (EPS) panel having the same thermal conductivity as the MW, but with all the other properties typical of EPS boards. MW ETICS with solar absorbance higher than 0.5-0.6 have a lower number of thermal shock events than the same ones with EPS (roughly 15% in average). These events occur in winter conditions, right after sunrise of when a façade a few instants before in the shadow is immediately sunlit. Right before the thermal shock, the base coat over a MW panel is actually moister than over EPS, and, thus, to produce a rapid surface temperature variation a phase change of the water in the base coat must occur, and that water has to be dried out by the sunbeams. The differences in the moisture contents in the base coat over EPS and over MW are no more than 10 kg m$^{-3}$, but this is sufficient to produce a different balance of extreme thermal shock events. This difference seems to be driven by the different water vapour resistance of EPS and MW (that of EPS more than 30-40 times that of MW). For an EPS-ETICS, the water vapour flowing through the wall assembly from the inside, during cold nights, condensates in the most external sections of the insulation panel, and once in liquid phase is stuck there since the closed pores structure of EPS. In case of MW-ETICS, water flows into the base coat, since the water vapour permeability of mineral wool is close to that of the air and itself is capable of hygroscopic moisture sorption getting in equilibrium with environmental conditions.

4 TM-FEM ANALYSIS

Since temperature variations are known to be one of the most common causes of ETICS failure [13] and the consequences of a wrong definition of them in a TM-FEM analysis are often underestimated. Although most of the studies in the Literature couple thermal and mechanical simulation in a single FEM analysis [14], it was chosen not to do so in order to take into account the (important) role of moisture in determining the temperature inside the ETICS. In this study, the temperature distribution obtained from HMT simulations is used as input of the TM-FEM analysis.
The definition of the geometrical model for the TM-FEM analysis raised some interesting questions. Even if the width and length of the insulation panels and the thickness of the layer are defined by shape and size of commercial products and by code of practice [15], the effects of deviation from the declared value due to (production or construction) tolerances and of different type of constraints have to be taken into account. A simple 1-D analysis has been made to understand the influence of mechanical properties of coat and insulation panel on the development of cracks on base (and top) coat. Figure 3 shows the main results of the analysis: regardless the type of insulation panel (here modelled as a layer of springs) a temperature increase cannot be the only cause of cracks (due to instability) on the base coat ($\sigma_{cr} = 6.1$ MPa, $\Delta T_{cr} = 576$ K) even if the base coat is already cracked (damaged perfect model, $\sigma_{cr} = 3.1$ MPa, $\Delta T_{cr} = 288$ K); a temperature drop may cause cracks on the base coat but only for a material with a (medium to) high Young modulus (Figure 3, Stress in the coat as a function its Young modulus $E$ [MPa] and of $\Delta T$ [K]). These results change if (construction and production) errors and imperfections are taken into account, for example a small difference in the thickness of two insulation panels placed side by side causes a huge decrease of the minimum temperature drop to have cracks (Figure 4). These preliminary results were confirmed by some later 3D FEM analyses where a model made of seven insulation layers (all of MW) with a cement based coat has been investigated using temperature distribution obtained from HMT analysis (in this case study the Milan climate has been used). If the model has no imperfections (due to errors in construction or to tolerances of materials) then there are no signs of possible cracks; if one insulation panel (the central one in this case study) has a different thickness (1 mm thicker in this case study) then displacements (Figure 5) and stresses (Figure 6) in the (base and top) coat show significant stress concentrations at the joints of flanked insulation panels.
Figure 4: Influence of imperfections on $\Delta T_{cr}$.

Figure 5: Displacements in x direction (thickness) [mm], for a positive temperature variation

Figure 6: Stresses z direction [MPa], for a negative temperature variation
In brief, cracks on the coat of ETICS will develop in the following case:

- the external face of the insulation panels does not belong to a perfect plane; a small step from one panel to the near one may be due even to a small tolerance in the thickness of the panel (manufacturing defects) or to defects in the support layer and it’s always present (gluing panels directly on a brick wall may not be a good idea, a plaster as regularization layer may be needed).
- the coat has a high Young (elastic) modulus (most of the coats on the market have a high elastic modulus).
- there are high temperature variations (between day and night and from one season to another); in most of the places where there is a high solar radiation there are likely to be high enough temperature variations, in sunny place beware of dark colours.

5 CONCLUSIONS

Considering thermal shock events, we noted a lower number of critical events for MW ETICS than for EPS ETICS, for solar absorbance higher than 50-60%. We impute this difference to the higher vapour permeability of MW compared to that of EPS, which allows for higher – although non-harmful – moisture content within the base coat over MW. After a cold night, a latent transformation occurs when the exterior surface is heated by the sunlight, and MW ETICS reach surface temperatures slightly lower than EPS ETICS. These considerations need to be verified with more extensive studies, and laboratory testing, because the approximations in material properties might have introduced systematic differences.

More interesting is that where thermal shocks are more frequent and potentially harmful, namely in Southern Europe, freeze-thaw does not seem to be an issue. Freeze-thaw cycles occur and eventually produce failures just in some case and for very low absorbances. However, due to soiling (i.e. dirt deposition) of the exterior surfaces, those very low solar absorbances are actually impossible in practice. From the results of the numerical sensitivity analysis we infer that with solar absorbances lower than 0.60 the risk of thermal shocks is remarkably limited, which is a lower limit to solar absorbance than that proposed in the European ETICS Association guideline. On the other hand, with solar absorbance higher than 0.20-0.30 the risk of freeze-thaw is also reduced in Southern European climates.

Considering the frequency of thermal shocks and of freeze-thaw, we conclude that for Southern European a base coat can be developed focusing more on resilience to thermal shocks than on resistance to freeze-thaw cycles, preferring a lower Young modulus, although with low capillary water absorption (which is critical for thermal shocks and freeze-thaw).

With the 3D FEM analysis, using the temperature distribution obtained by HMT simulations, we demonstrated that if the model has no imperfections (due to errors in construction or tolerances of materials) there are no cracks on the coat. The cracks will develop if the external face of the insulation panels is not perfectly plane, and the base coat has a high Young modulus and there are high temperature variations.

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MICROSTRUCTURE DEVELOPMENT IN SLAG-CEMENT SYSTEMS

E. Berodier (1), K. Scrivener (2)

(1) Laboratory of Construction Materials Ecole Polytechnique Fédér mal de Lausanne –
elise.berodier@epfl.ch
(2) Laboratory of Construction Materials Ecole Polytechnique Fédér mal de Lausanne –
karen.scrivener@epfl.ch

Abstract

The low permeability of the microstructure in slag-cement paste is regarded as the major reason of the better long-term durability of these systems. However it is still not clearly understood how the refinement of the microstructure is achieved.

This study reports on the development of the microstructure in slag-cement system. Ternary systems consisting of cement blended with quartz and slag were studied. The replacement of cement was kept constant and the proportion of slag and quartz were varied to isolate the effect of slag. Our results showed that slag products form in the water space. Once the capillary porosity becomes limited, the reaction slows down.

1 INTRODUCTION

Blended cements containing blast furnace slag are often recommended to improve durability in aggressive environments. Among the benefits of these blends are: improved compressive strength at long term and decrease the chloride ion permeability. These properties are strongly related to changes in the porosity of the paste. However the link between microstructure and durability is not well established for slag-cement systems. The interaction of the slag reaction with the hydration of the clinker phases complicates understanding the microstructure development in such systems. From thermodynamics models, we can predict the hydrates that will form and their volume fraction [1]. However such approaches give no indication of the spatial distribution of the hydrates.

The goal of this study was to analyse the development of microstructure in blended systems. The content of clinker was kept constant and the proportion of slag and quartz were varied to isolate the effect of slag.
2 EXPERIMENTAL DETAILS

Ordinary Portland cement (CEM I 52.5R) was mixed with quartz and slag. The chemical composition by XRF is shown in Table 1. All the powders had similar particle size distributions. To isolate the effect of slag, the content of the Portland cement was kept constant. All the systems were formulated to have a solid to liquid volume, the same as in a pure Portland paste at a water to cement ratio (by weight) of 0.4. The volume of the Portland cement was equivalent to 60% by weight of cement in a mixture with of plain Portland cement with a W/C = 0.4 (by weight). The remaining 40% Portland cement volume was replaced by an equivalent volume of slag or quartz so the water to solids by volume is always the same as it would be in a pure Portland cement at W/C = 0.4. The relative amounts of slag or quartz are expressed as the equivalent volume of Portland cement replaced. Full details of the mixture are given in table 2.

Table 1: Chemical composition of the materials

<table>
<thead>
<tr>
<th></th>
<th>Cement %w</th>
<th>Quartz %w</th>
<th>Slag %w</th>
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</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>19.25</td>
<td>97.91</td>
<td>34.09</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.68</td>
<td>1</td>
<td>19.87</td>
</tr>
<tr>
<td>CaO</td>
<td>63.23</td>
<td></td>
<td>33.01</td>
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<tr>
<td>MgO</td>
<td>2.05</td>
<td></td>
<td>9.73</td>
</tr>
<tr>
<td>Fe₂O₃</td>
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<td>Na₂O</td>
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<tr>
<td>K₂O</td>
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<tr>
<td>TiO₂</td>
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<td>0.9</td>
</tr>
<tr>
<td>SO₃</td>
<td>4.04</td>
<td></td>
<td>0.75</td>
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</table>

Table 2: Mixing design of the systems

<table>
<thead>
<tr>
<th>Systems</th>
<th>%weight</th>
<th>%vol</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Clinker</td>
<td>Slag</td>
</tr>
<tr>
<td>S cement</td>
<td>71.4</td>
<td>-</td>
</tr>
<tr>
<td>S-40Slag</td>
<td>44.2</td>
<td>26.3</td>
</tr>
<tr>
<td>S-30Slag-10Quart</td>
<td>44.5</td>
<td>19.9</td>
</tr>
<tr>
<td>S-20Slag-20Quart</td>
<td>44.8</td>
<td>13.3</td>
</tr>
<tr>
<td>S-10Slag-30Quart</td>
<td>45.1</td>
<td>6.7</td>
</tr>
<tr>
<td>S-40Quart</td>
<td>45.4</td>
<td>-</td>
</tr>
</tbody>
</table>

The hydration at 20°C was followed by calorimetry (TAM air Thermometrics) over 28 days. Ten grams of paste mixed externally were placed in the calorimeter.

For SEM Image analysis, a slice was cut from the cast paste. The hydration was stopped by isopropanol exchange to preserve the microstructure [2]. Once dried, the sample was prepared.
for BSE observation by epoxy resin impregnation (EPOTEK-301) and polished with diamond powders down to 1 μm and coated with about 15 nm of carbon coating.

The pore structure was studied by mercury intrusion porosimetry. Samples were dried in the same way as for SEM sample. All the analysed samples had similar weight for consistency. In the first step a pressure of 100 kPa was applied to intrude larger pores. Then, the pressure is increased to 400 MPa which accesses pores down to 2 nm.

3 RESULTS AND DISCUSSION

3.1 Effect of slag replacement on kinetics

Figure 1 shows the cumulative heat released over 28 days of hydration. The four curves are almost identical up to about 30 hours and then progressively diverge. The total heat reached at 28 days increases with slag content. Nevertheless the additional increase is smaller for the higher replacement levels i.e. from the system of 20% slag-20%quartz (see Figure 1(right). This suggests that slag reaction becomes limited at higher additions.

![Calorimetry curves](image)

Figure 1: (on the left) Calorimetry curves over 28 days (log scale) of 4 blended systems with increasing slag content. (on the right) the data at 28 days are reported as a function of the slag fraction

3.2 Microstructural changes with slag content

Figure 2 shows the changes of the microstructure with increasing the slag content. The microstructures becomes denser with increasing slag content. However, it can be seen that microstructure of the 20%slag-20%quartz system (Figure 2.d), 30%slag-10%quartz (Figure 2.b) and 40% Slag (Figure 2.a) are relatively similar. All are dense, the outer products fill almost all the space available between grains. These observations suggest that the reactivity of slag is limited by the space available to fill with outer products.
Figure 2: Micrographs (BSE mode) of blended pastes hydrated for 28 days of  
a) 40% Slag;  
b) 30% Slag 10% Quartz;  
c) 10% Slag 30% Quartz;  
d) 20% Slag 20% Quartz

3.3 **Effect of increasing water to solids ratio**

To explore the role of space a complementary system was studied: the 40% slag substitution with higher water/solids ratio to 0.6 instead of 0.4. Both cumulative curves are shown in Figure 3. From 30 to 150 hours both systems have similar kinetics, indicating that space is not limiting the reaction during this period. Then from 6 days, we see that the reaction slows down in the lower w/s system with less space, while it continues in the higher w/s system.

Figure 3: Cumulative heat released over 28 days (log scale). The increase of water/solids ratio increases slag reaction
The micrographs in Figure 4 show these two systems with different water/solids ratio. Large clumps of AFm phase are a notable feature in both systems. At higher water/solids = 0.6, these form in the spaces between particles, whereas in the system with w/s 0.4 these clumps occur in the shells of hydration product from small cement grains (Hadley grains), which would appear to be the only space available in this case. In addition, there is more evidence of fibrilar, less dense C-S-H in the high water to solids case.

![Micrographs of 40% Slag-cement systems with w/s 0.4 and w/s 0.6 after 28 days](image)

Figure 4: Micrographs of 40% Slag-cement systems with w/s 0.4 and w/s 0.6 after 28 days

4 CONCLUSIONS

In this study it is shown that the reactivity of slag is influenced in the long term by the space available. After 6 days the degree of slag reaction is limited at lower water to solids ratio 0.4. Mercury intrusion porosity and SEM analysis indicate that slag products form in the capillary porosity. Once the capillary porosity becomes limited, the reaction slows down.

These results indicate that the efficiency of the slag contribution to microstructure and property development varies as a function of the space available.

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A NUMERICAL STUDY OF THE INFLUENCE OF THE HYDRAULIC INTERFACE CONTACT ON THE HYGRIC PERFORMANCE OF A MULTI-LAYERED SYSTEM

E. Vereecken (1) and S. Roels (1)

(1) Building Physics Section, KU Leuven, Leuven – Evy.Vereecken@bwk.kuleuven.be; Staf.Roels@bwk.kuleuven.be

Abstract

In the current paper, the hydraulic contact interface in a multi-layered system consisting of a brick in combination with a capillary active interior insulation system is discussed. An overestimation of the inwards moisture flux can induce a high indoor relative humidity, mould growth, etc. In cases of capillary active interior insulation, moisture reaching the capillary active interior insulation could also induce a decrease in thermal resistance. Therefore, knowledge on the impact of the hydraulic interface is important to come to reliable hygrothermal simulations.

In a first part of this paper, the hydraulic contact interface is experimentally studied based on an imbibition experiment. Next, the influence of this hydraulic interface in cases of realistic climatic conditions is numerically studied. As found in a prior study on brick-mortar composites [1], the significant influence of the hydraulic interface on the moisture transport in an imbibition experiment was not found in cases of realistic climatic conditions.

1 INTRODUCTION

Most building elements are a composite of different material layers. Moisture transport in multi-layered building elements can – due to the presence of an interface contact – deviate from the moisture transport found for the combination of the single material elements. This deviation is projected in a deceleration of the liquid transport across the material interface. Hence, in order to achieve a reliable assessment of the durability of multi-layered systems, the influence of neglecting the interface resistance should be studied.

The hydraulic interface resistance between material layers is mostly investigated for a brick-mortar contact (e.g. [2],[3]). Minor or no attention is paid to the hydraulic interface resistance between the masonry wall and additional layers (e.g. (insulation) plasters/renders, capillary active insulation systems, etc.). Though, also for these systems it can be expected that a hydraulic interface resistance is present.
In the current paper, the moisture performance in a brick in combination with a capillary active interior insulation system, i.e. calcium silicate, is investigated. Calcium silicate is mostly glued to the wall construction. Hence, an interface resistance can be found between brick and glue mortar ($R_{IF1}$) as well as between glue mortar and calcium silicate ($R_{IF2}$).

In contrast to a brick-mortar composite, where liquid transport is mainly the result of a rain load or a rising damp flow, in cases of interior insulation also over-hygroscopic moisture obtained due to potential interstitial condensation can cause a liquid flow. Though, since the theoretical condensation plane is found at the interface between the glue mortar and the insulation material – and as a consequence no interface has to be bridged to reach the insulation layer – an interface resistance between the material layers is assumed to have no influence on the inwards moisture flux caused by interstitial condensation. A potential redistribution from the interstitial condensation towards the brick layer is assumed to include a minor risk as interstitial condensation is a slow process resulting in a small moisture amount. Hence, the potential influence of an interface resistance on the risk on damage patterns caused by interstitial condensation is questionable. Based on this reasoning, wind-driven rain remains the main moisture source. A rain load at the exterior surface could result in a moisture front reaching the warm side of the masonry wall. If the moisture could reach the capillary active interior insulation system, a decrease in thermal resistance occurs. In addition, due to the capillary forces, the moisture could be redistributed towards the room and could hence induce a high indoor relative humidity, mould growth, etc. Therefore, in the remainder of this section the main focus lays on the moisture flow from the brick layer (exposed to e.g. a rain load) towards the capillary active system.

The study on the hydraulic interface contact is in a first step performed based on an imbibition experiment. The experimental data are used in the inverse identification of the hydraulic interface resistance, which is implemented by use of a fictitious interface permeability $K_{IF}$:

$$K_{IF} = \frac{d}{R_{IF}} \quad (1)$$

with $d$ the thickness of the fictitious interface layer (1 µm) and $R_{IF}$ the interface resistance (m/s). The interface layer is assumed to have no capacity. Note however that the main reason for the determination of the interface resistances as performed in this paper is to investigate the magnitude of this resistance and its influence on the moisture inflow in a building assembly with a capillary active interior insulation system. The determination of the interface resistances should be considered as a preliminary study. A more precise determination of the interface resistances for realistic wet/dry cycles together with a physical explanation of the observed behaviour demands a more in depth investigation.

To end, the determined hydraulic interface is used in a numerical study on the influence of the hydraulic interface contacts in cases of realistic climatic conditions.

2 MATERIAL PROPERTIES

The liquid permeability and the corresponding pore volume distribution of the ceramic brick (CB), the calcium silicate (CaSi) and the cured glue mortar (GM) are given in Figure 1. Note that in the pore volume distributions only the pores assumed to be filled in a free water uptake experiment are included. The ceramic brick shows mainly a coarse porous structure ($10^{-6} - 10^{-5}$ m). Only a small number of pores corresponds to the hygroscopic range. The calcium silicate is mainly characterized by a unimodal fine pore system. For the glue mortar,
two pore subsystems can be clearly distinguished. A first system lays in the same range as the calcium silicate. A second system corresponds to even more hygroscopic (fine) pores.

3 IMBIBITION EXPERIMENT

In a first step, an imbibition experiment is performed on a series of composite samples. Samples with a perfect as well as with an imperfect interface contact are studied. In a next step, the interface resistances are determined by fitting the simulations of the imbibition experiments to the experimental findings.

3.1 Test setup

To investigate the importance of the hydraulic interface $R_{IF1}$ and $R_{IF2}$, in a first step the total moisture uptake during an imbibition experiment is experimentally determined for the contact types given in Table 1. To construct the samples, a glue mortar layer (0.26 liter H$_2$O for 1kg Calsitherm KP-glue) is applied between a 5 cm high brick layer and a 5 cm high calcium silicate layer (both stored at 50% RH and 20 °C). After curing (at 50% RH and 20 °C), the samples A, B and C (Table 1) are demounted by sawing at the interface(s) where a perfect contact is required. The final glue mortar joints have a thickness between 4 and 8 mm. Hereafter, a perfect hydraulic contact is obtained by applying a thin layer of kaolin clay between the material layers. To avoid a loss in contact between the kaolin clay and the material, the re-constructed samples are kept together by clamping screws (Figure 2). Since in practice a liquid flow from exterior is most likely, the brick layers are placed in contact with

Table 1: Types of interface contact for the different samples

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<tr>
<th>Interface between brick and glue mortar (IF1)</th>
<th>Interface between glue mortar and calcium silicate (IF2)</th>
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<tr>
<td>A Perfect hydraulic contact</td>
<td>Perfect hydraulic contact</td>
</tr>
<tr>
<td>B Perfect hydraulic contact</td>
<td>Imperfect (real) hydraulic contact</td>
</tr>
<tr>
<td>C Imperfect (real) hydraulic contact</td>
<td>Perfect hydraulic contact</td>
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<tr>
<td>D Imperfect (real) hydraulic contact</td>
<td>Imperfect (real) hydraulic contact</td>
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the water plane during the imbibition experiment. A one-dimensional moisture flow is obtained by taping the sides of the samples with plastic foil. This foil was a few centimeters higher than the sample height and closed at the top with a perforated foil. By doing so, evaporation at the top of the samples was attempted to slow down without avoiding air exfiltration out of the material. Note however, that due to the limited moisture flux out of the unsaturated sample, the perforation in the plastic foil and the duration of the experiment, a high relative humidity above the sample and the absence of evaporation are questionable.

Figure 2: Setup of the imbibition experiment for some of the re-constructed samples

3.2 Experimental results

Figure 3 shows the moisture uptake during the imbibition experiment. As can be seen, a real hydraulic contact (samples B, C, D) results in a slower water uptake. Moreover, those samples never reach the maximum total moisture content obtained in the sample with two perfect hydraulic contact interfaces (sample A). Capillary saturation of the calcium silicate layer is never reached. Note also the large spread in accumulated moisture inflow. This spread can be attributed to e.g. small deviations in the moisture properties of the brick and the glue mortar, to small differences in the thickness of the glue mortar layer and especially to a large

Figure 3: Total accumulated moisture content during the imbibition experiment.
spread in mismatching of the pore structures between the different materials and to potential small compaction pores between the material layers obtained during the preparation of the samples. In practice, an even larger spread in results can be expected. Moreover, other types of brick, glue mortar or insulation will result in other hydraulic interface contacts.

3.3 **Inverse identification of the hydraulic interface resistance**

To estimate the magnitude of the interface resistances, the imbibition experiments are simulated by use of HAMFEM [4]. Since the absence of evaporation at the top is questionable, the interface resistances are determined for a room relative humidity of 50%. The moisture surface transfer coefficient is set at $18.5 \times 10^{-9}$ s/m. No temperature influences are taken into account. The interface resistances $R_{IF1}$ and $R_{IF2}$ are determined based on the experimental results of the samples B and C. The interface resistance is implemented by an interface layer (1 µm) characterized by an interface permeability $K_{IF}$ (s) as defined in Eq.(1).

Firstly, the impact of a constant (not dependent on capillary pressure or moisture content) interface resistance is studied. Figure 4 shows for sample B the comparison between the experimental findings and the numerical results when including a constant interface resistance. A constant interface resistance is found to be inappropriate to simulate the moisture inflow. However, when only the start of the moisture uptake in the calcium silicate is of importance, a constant value can be considered.

Because of the poor agreement between the experimental and the numerical results when using a constant interface resistance, in a next step a variable interface resistance is proposed. Due to the large spread in results for the samples B and C (Figure 3), for both interface contacts both a small and a large interface resistance are determined. Additionally, it should be noted that at the start of the moisture accumulation in the calcium silicate, the moisture uptake for the samples of Type B was found to be similar to the moisture uptake found in the sample of Type A. This indicates the absence or negligibility of an interface resistance at low capillary pressure (note that the capillary pressure is given by a negative value, so at low moisture content) near the interface contact. Therefore, at low capillary pressure, the interface
permeability is assumed to be equal to the permeability of the glue mortar. At a later stage (and thus when a higher capillary pressure occurs near the interface), a larger interface resistance (and hence lower interface permeability) is required to obtain the decrease and finally the stop in moisture uptake as experimentally observed.

As an example, Figure 5 shows a comparison between the simulations and the experimental results for the samples with an interface resistance between the glue mortar and the calcium silicate (samples B) if a variable interface resistance – determined based on the assumptions mentioned above – is included. Similarly, based on the samples of Type C an interface resistance $R_{IF1}$ characterizing the imperfect contact between the brick layer and the glue mortar is determined by inverse identification. Figure 6 shows for interface 1 (brick-glue mortar) and 2 (glue mortar-calcium silicate) the interface permeability $K_{IF}$ which results in an

![Figure 5. Comparison between the measurements and the simulations in cases of a variable interface resistance for sample B.](image)

![Figure 6. Interface permeability (for a fictitious interface layer with a thickness equal to 1µm). IF1 = interface between brick and glue mortar, IF2 = interface between glue mortar and calcium silicate, S = small interface resistance, L = large interface resistance.](image)
acceptable agreement with the low and high moisture uptake found for the samples of Type B and C. The interface permeability is defined in function of the capillary pressure in the interface layer. As a reference, the liquid and vapour permeability of the glue mortar are given. In the hygroscopic range the interface permeability mainly coincides with the moisture permeability of the glue mortar. Note that an interface permeability equal or higher than the permeability of the glue mortar matches the absence of an interface resistance. In the over-hygroscopic range an interface resistance is included by – compared to the glue mortar – a lower moisture permeability. Starting from a capillary pressure equal to approximately \(-10^{5.8}\) Pa an even larger decrease in permeability is required to be in agreement with the experimental findings. Finally, the interface permeability is assumed to be equal to the vapour permeability of the glue mortar.

4 CLIMATIC CONDITIONS

In the current subsection the hygric performance of a wall assembly with a capillary active interior insulation system is numerically investigated in cases of (a) an implementation of a hydraulic interface resistance and (b) neglecting the hydraulic interface resistance. The numerical study is performed with HAMFEM [4]. The influence of the interface resistances is investigated for a wall assembly consisting of 19 cm brick layer, 4 mm glue mortar, 5 cm calcium silicate and 1 cm plaster.

To include the interface resistance, between the brick layer and the glue mortar and between the glue mortar and the calcium silicate a 1 µm interface layer is implemented. For the interface permeability, \(K_{\text{IF1}}(L)\) and \(K_{\text{IF2}}(L)\) as shown in Figure 6 are used to simulate the interface resistance between respectively the brick layer and the glue mortar and between the glue mortar and the calcium silicate. Note that the large interface resistances (= low \(K_{\text{IF}}\)) are used in the analysis, since these could result in the largest restriction of the inwards moisture transport. The analysis is performed for a north-west oriented wall, since for latter orientation a large wind-driven rain load is observed. The climate of Essen (Germany) is used. The indoor temperature and relative humidity are kept constant at respectively 20 °C and 50%.

The other boundary conditions are identical to the boundary conditions described in [1]. The annual maximum moisture content in function of the position is shown in Figure 7a. The implementation of a hydraulic interface resistance at both contact interfaces results in a higher maximum moisture content at the warm side of the masonry wall. The maximum moisture content in the glue mortar is slightly higher. Though, the interface permeability in the capillary pressure range between \(-10^{6.5}\) and \(-10^6\) Pa, as given in Figure 6, is too high to influence the maximum moisture content in the calcium silicate. To assess the time period exposed to a higher moisture content, Figure 7b shows the moisture content at the warm side of the masonry wall (19 cm from the outer surface). The influence of the interface resistance is only visible during the WDR peak in the beginning of June. The influence on the accumulated moisture content in the brick layer and in the calcium silicate layer was negligible (not shown). Based on the limited period exposed to an increased moisture content in combination with the negligible impact on the total accumulated moisture content for each material layer, it can be concluded that the determined interface resistances have only a minor influence on the hygrothermal performance of the wall assembly.
5 CONCLUSIONS

In this paper, the hydraulic interface contact was discussed for a brick layer in contact with a capillary active interior insulation system. It was found that an interface resistance should be implemented to enable an acceptable agreement between an imbibition experiment and the numerical results. A constant interface permeability – and hence interface resistance – was found to be inappropriate to simulate the moisture uptake. Therefore, a variable interface permeability was suggested. For the hygroscopic range no or a negligible interface resistance was found. Hence, the hydraulic interface contact tends to have no influence on vapour transport. In the over-hygroscopic range an interface resistance was included. When capillary saturation was almost reached an increase in interface resistance was required.

In cases of realistic climatic conditions, the interface resistance was found to have only a negligible influence on the hygrothermal performance of the wall assembly.

ACKNOWLEDGEMENTS

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REFERENCES

EVALUATION OF DAMAGE OF BUILDING FACADES IN BRASILIA

Elton Bauer (1), Eliane Kraus (2), Maria N. B. Silva (2) and Vanda A. G. Zanoni (2)

(1) University of Brasilia, Brasília – elbauerlem@gmail.com;

(2) University of Brasília, Brasília – kraus@unb.br; nazabsilva@gmail.com;
vandaz@terra.com.br

Abstract

Building facades undergo a natural durability reduction process due to age and exposure to atmospheric agents, however, building facades in Brasília have shown in recent decades marked signs of deterioration, which in some cases occur at an early stage. Thus, this study aims at carrying out a quantitative study on the influence of age and climatic agents on the deterioration processes. This paper presents a degradation survey methodology on 8 buildings through visual inspections and facade mapping in order to quantify the degradation level as a function of the solar radiation incidence. The results showed higher degradation levels on facades exposed to higher solar incidence as well as in older buildings. This study underscores the need for inspections to identify and quantify the damage in order to allow the assessment of the behavior and service life of facades exposed to severe insolation.

1 INTRODUCTION

Brasilia, the federal capital of Brazil, is a planned city and at just 53 years of age it is considered very young. Its facades have a very simple typology, usually employing rendering mortars or ceramic tile cladding, and often having a mixed use of both systems.

Ceramic tile cladding systems are vertical elements that compose, coat and protect the facades and even structural elements of the buildings, which are therefore highly exposed to the combined influence of rain, wind, insolation, biological agents and atmospheric pollutants [1][2][3]. This level of exposure triggers several kinds of degradation mechanisms, leading to a degradation process with loss of quality and performance more evident than in other elements of the building, which also implies high maintenance costs, an evolution from visual degradation to actual physical problems to the facade, and durability loss [4][5].

Pathological manifestations occurring to the facades are influenced and identified both by incidence rate and damage severity. Building facades in Brasilia have shown – at a greater intensity – ceramic tile detachment and crack failures and – to a lesser degree – an occurrence of grout failures, efflorescence, and sealing failures [6][7][8]. Brasilia's climate, with annual average temperatures considered high, exerts great influence on the degradation processes. Thermal variations generate pressures that act directly on the claddings, causing a differential thermal expansion of the cladding system and in each of its component parts (ceramic tile, grout, adhesive mortar, plaster and substrate) [7][9][10][11].

In order to relate facade degradation with climate related aspects and verify their influence on the degradation processes, data on the solar radiation incident on the facades of Brasilia coming from the north, east, south and west directions was used [12][13]. Solar radiations, considered under a special condition of maximum radiation on the facades, i.e. with no nebulosity, were obtained on specific days for all months of the year starting from the projection of the sun's apparent trajectories represented by Brasilia's solar chart. In this sense, solar radiation values do not represent monthly or annual figures, they represent instead a peculiarity in the case of facades in Brasilia and indicate only a trend of radiation intensities so as to guide the analysis of occurrences and degradation levels in facades.

This study consists of an analysis on the facade degradation caused by the pathologic manifestations of ceramic tile detachment, grout failures, cracks, efflorescence, and sealing failures in eight buildings of Brasilia. The selected buildings have ages ranging from 5-48 years with a total area of 21,158.00m². The facade damage survey is conducted according to a inspection methodology developed in the Laboratório de Ensaio de Materiais – LEM (Materials Testing Laboratory) of the University of Brasilia (UnB). The degradation analyses are carried out through a methodological approach previously developed in Portugal that aims to assess and quantify in detail the damage to the facades through their extent and overall degradation level (ODL) [3][14].

2 FACADE DEGRADATION STUDY METHODOLOGY

2.1 Survey and classification of the degradation incidence in facades

The inspections were conducted on facades with ceramic tile cladding. Visible damage (detachments, grout failures, cracks, efflorescence, and sealing failures) were mapped (Figure 1-a) based on inspections carried out during on-site surveys by the staff of the Materials Testing Laboratory (LEM) of the University of Brasilia [6][7][8]. The degradation of the facades is identified through the extent and severity of the damage, among which were:
detached ceramic tiles, grout failure, cracks, efflorescence and sealing failure [7][15][16].

Complementary to the methodology already developed by LEM-UnB an innovation is presented in the counting process of the pathological manifestations through a mesh overlaid on the mapping (Figure 1-b).

Figure 1: (a) Damage mapping scheme [16], and (b) mesh overlay on the facades analyzed.

Each mesh corresponds to a region of 0.25 m² damage, i.e. it allows to obtain not only the visible damaged area, but also a possible extension of the invisible damage, since the existence of a specific damage often implies a propagation region larger than the visible region, i.e. around the periphery of that damage.

2.2. Application of the Facade Degradation Calculation Routine

The calculation routine employed consists of evaluating the overall degradation level (ODL) based on the facade cladding systems with rendering mortar or ceramic tile cladding [2][3][14][17]. It is emphasized that this routine was adapted for facades with ceramic tile cladding, so as to provide continuity to the studies aimed at quantifying degradation on the facades of Brasilia [10]. We seek to verify the sensitivity of this routine to the identification and quantification of different degradation levels due to age and solar radiation incidence on building facades.

Maintenance and age are considered essential criteria for determining ODL's variation pattern as a function of time. Failures are analyzed both by considering the $k_{c,n}$ factor, risk level and repairing cost of the failures seen on the facades of Brasilia and by disregarding this factor. Thus, two ODL analysis models are established:

- Model 1 (M-1) - considers a weighting ($k_{c,n}$) for the cost of repairing the facades of Brasília (1);
- Model 2 (M-2) - considers a cost weighting of $k_{c,n} = 1.00$.

The ODL (1) establishes a direct relationship between the area of the facade affected by the defects and their defect level as a function of the total area of the facades analyzed.
ODL = \sum_{n} \left( A_n \cdot k_n \cdot k_{c,n} \right) / (A_r \cdot k) \tag{1}

Where:

- **ODL** - Overall degradation level (%);
- **A_n** - area of a facade affected by n defects (m²);
- **k_n** - n level of defects contained in the intervals (0.00, 0.02, 0.20, 2.00, 4.00) [3];
- **k** - constant, equivalent to the worst condition level (k = 4.00);
- **k_{c,n}** - the relative importance of defects detected in Brasilia;
- **A_r** - exposed facade surface (m²).

ODL is a parameter that measures in a simple fashion the damage level relative to a maximum reference damage expressed by the term \((A_r \cdot k)\). This parameter and its variables are adapted in this study for use with ceramic tile coated facades. Therefore, variables \(A_n\), \(k_n\), \(k\), \(k_{c,n}\) and \(A_r\) are adapted to reproduce the damage (ceramic detachment, grout failures, cracks, efflorescence, and sealing failures) on facades with ceramic tile cladding. The variable \(A_n\) corresponds to the area affected by the damage (ceramic detachment, grout failures, cracks, efflorescence, and sealing failures) and identified in the mapping. The damage level, represented by \(k_n\), ranges from 0.00 (no damage) to 4.00 (severe state of degradation).

The variable \(k_{c,n}\) (Table 1) varies according to the location of the samples analyzed and represents the weighting of the relative importance assigned to the repairing cost of each n damage type (where \(n_1\) = ceramic detachment, \(n_2\) = grout failure, \(n_3\) = cracks, \(n_4\) = efflorescence, \(n_5\) = sealing failure).

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<th>Weighting coefficient parameters</th>
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<td><strong>Condition levels</strong></td>
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<td>Repairing cost (Brasilia)</td>
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### 3 RESULTS

From the degradation survey in a sample of eight buildings, divided into seventy-one samples of facades or plumb bob, a degradation level was preliminarily obtained for each case of damage studied (ceramic detachment, grout failure, cracks, efflorescence, and sealing failure) as a function of age and solar directions (north, east, south and west) of the facades. The ODL analyses are conducted for M-1 and M-2 models that vary depending on whether the factor that relates to the repairing cost is taken into account.

The facades used in this case study have the following characteristics: 6-story residential buildings, a 21,158.00 m² total area of facades, reinforced concrete structures, ceramic block sealing and ceramic tile claddings.

Figure 2 shows the graph with the occurrences (percentage) of all defects (ceramic detachments, grout failures, cracks, efflorescence, and sealing failures) observed in the building facades.
It is observed that the percentage of ceramic tile detachment failures showed the highest results (71%) compared to other occurrences (grout failure, cracks, efflorescence, sealing failure), which corroborates previous studies on the facades of Brasilia [6][7][8]. Considering that ceramic tile detachment failures are usually associated with thermal variations, the high percentage obtained (Figure 3) highlights the incidence of solar radiation as a major cause for this failure.

Degradation level (ODL) results characterize the behavior of facades both in terms of age (Figure 4) and the directions of the facades (Figure 5). The ODL showed an increasing trend for both M-1 and M-2 models.

Figure 4 show that older facades have the highest levels of degradation. This is due to exposure to weather conditions, movement between ceramic cladding layers and the consequent natural aging process of the facades. It was also observed that the values for M-2, with damage weighting, also have higher degradation levels in relation to the M-1 model, which does not consider the repairing cost in the ODL calculation. This behavior shows the effect of weighting factor of type of pathological manifestation, i.e. the higher the value attributed to the repairing cost, the will be the ODL value.

The facades analyzed as a function of the solar radiation incidence (Figure 4) also show increased ODL due to greater exposure to solar radiation for the two analyzed models (M-1 and M-2).
The methodology used for determining the ODL allowed to accurately identify different degradation levels as a function of age (Figure 3) and solar radiation incidence (Figure 4) on
the facades. There was a considerable increase in ODL values for facades aged above 30 years (Figure 3). The same behavior was observed for the facades exposed to solar radiation (Figure 4). The higher the level of exposure of facades to solar radiation, the more advanced its stage of degradation.

Notably, there is a strong relationship between degradation levels and solar radiation incidence due to the high percentage of ceramic detachment failures observed in the facades, as well as crack and grout failures, also associated with the thermal effects arising from the solar radiation incidence.

4 CONCLUSIONS

The particularity of the residential building system of Brasília is worthy of notice. Based on the survey of the five facade degradation types (ceramic detachment, grout failure, cracks, efflorescence, and sealing failure) as a function of the age of the facades and solar orientation, it can be concluded that:

- The calculation routine is analyzed efficiently and shows an improvement in the assessment of degradation due to age and sun exposure. The pathological manifestations with the highest occurrence in facades were cracks and ceramic detachment.
- The pathological manifestations surveyed showed a strong relationship with the solar incidence.
- The ODL showed a progressive degradation of buildings with over 30 years of age, i.e., it showed a consistency in the calculation model for degradation and service life.
- ODL results in general proved the efficiency of its applicability and weighted analysis accuracy through damage level and repairing cost in quantifying the overall degradation level of facades.

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REFERENCES

Abstract

Expanding weather stripping foams are applied between various building component joints in the building envelope in order to achieve a satisfactory air tightness. A typical example is between a window and its joints towards the wall. During construction the distance may vary in these joints, therefore it is required that the weather stripping is able to expand up to a specified thickness. It is crucial that this expansion capability will be intact for a specified service life, or else the air tightness will be substantially jeopardized, which again may lead to a reduced energy efficiency of the building and also building damages caused by moisture ingestion. However, degradation and subsequent loss of expansion capability may occur in expanding weather stripping foams by their exposure to outdoor climate during many years, both directly exposed to solar radiation and wind-driven rain during the construction period and temperature ageing throughout several years. Furthermore, freezing/thawing cycles may also contribute to the deterioration.

In order to investigate their retention of expansion capability, the expanding weather stripping foams have been subjected to accelerated ageing by climate ageing and elevated temperature ageing. The durability and ageing of the expanding weather stripping foams have been studied and characterized by their expansion ability, chemical material analysis by Fourier transform infrared (FTIR) spectroscopy and structural analysis by scanning electron microscopy (SEM).
1 INTRODUCTION

Various building materials and components have to fulfil miscellaneous requirements with respect to durability. Hence, these building products also need to withstand different climate exposure factors throughout their lifetime, including both outdoor and indoor conditions. A core issue is to be able to fulfil specific functions by upholding key properties during the service life of the building products.

Accelerated climate ageing carried out in the laboratory is a versatile, powerful and time-saving method for investigation of the durability of building materials and components, also including newly developed material and solutions [1-2]. Performing a robustness assessment of these materials and solutions may also be found to be beneficial [3]. One key characterization tool is the Fourier transform infrared (FTIR) spectroscopical method which may reveal information about what chemical degradation reactions are occurring by analyzing the measured FTIR spectra and correlating changes in different absorbance peaks with changes in the chemical bonds due to the various ageing processes taking place [4-5]. The FTIR method may be utilized for various tasks and materials, e.g. polymers or plastics [6], wood [7-8], wood rot decay and fungus growth [9-10] and various wood impregnations and paints also including paints modified with nanoparticles [11-15]. For further information about the FTIR material characterization technique applied on miscellaneous materials it is referred to the available literature [16-25].

However, degradation processes may occur without any chemical reactions taking place and in such cases the FTIR technique will naturally not disclose any information about chemical changes as there are none. Nevertheless, applying FTIR spectroscopy to prove that there are no chemical reactions occurring may also be very valuable, often in combination with other material characterization tools like e.g. scanning electron microscopy (SEM). As a demonstration, we will witness and study such a case closer in the following.

In this work we are taking a closer look at the expanding weather stripping foam, which is intended for application between assorted building component joints in the building envelope in order to provide a satisfactory air tightness. A typical example is the joints between a window or door frame and the surrounding wall. The distance may vary in these joints during construction, thus it is required that the weather stripping is able to expand up to a specified thickness. Furthermore, this expansion capability has to be maintained and undamaged for a specified service life, otherwise the air tightness will be substantially jeopardized.

More specifically non-aged and aged samples of the expanding weather stripping foam will be subjected to material characterization by FTIR and SEM analysis, which hence will be attempted to correlate with any changes in the expansion capabilities of the weather stripping foam.

2 EXPERIMENTAL

Sample specimens of the expanding weather stripping foam were cut into suitable dimensions for the various ageing procedures and characterization investigations. Expanding weather stripping foam materials are intended for application between various building component joints in the building envelope as to provide a satisfactory air tightness, e.g. in the joints between a window frame and the surrounding wall. For the specific expanding weather stripping foam studied in this work, the normal expanded condition is 12 mm, i.e. the typical joint width for its intended application is also about 12 mm.
The expanding weather stripping foam samples have first been aged in a vertical climate simulator according to NT Build 495 [26] for 15 days (15 x 24 h). The samples were aged at a normal expanded condition of 12 mm. In this test apparatus the samples are subjected in turns to four different climate zones, that is, an ultraviolet (UV) and infrared (IR) irradiation zone (black panel temperature of 63ºC), a water spray zone (15 dm³/(m²h)), a freezing zone (-20ºC) and an ambient laboratory climate zone. The UVA and UVB intensities are averaged to respectively 30 W/m² and 3.4 W/m² for the investigations reported within this work. The exposure time is 1 hour in each climate zone in the above given sequence. For further details it is referred to the test method, i.e. NT Build 495 [26].

After undergoing accelerated climate ageing according NT Build 495 [26], a heat incubator (Termaks) has been employed in order to subject a selection of the samples furthermore to a constant air temperature of 70ºC for 90 days (90 x 24 h). The samples were aged at a normal expanded condition of 12 mm. Note that this temperature of 70ºC is at the upper end of the normally accepted high-end safe temperature range between 60ºC to 70ºC for accelerated ageing of polymers. The higher temperature of 70ºC is chosen in order to test the actual materials for as high temperature as 70ºC and to accelerate the temperature degradation as much as possible (Arrhenius equation).

The Fourier transform infrared (FTIR) material characterization (before and after ageing) was carried out with a Thermo Nicolet 8700 FTIR spectrometer with a Smart Orbit accessory, i.e. a horizontal attenuated total reflectance (ATR) accessory (single reflection) with a diamond crystal, in the wavelength range 4000 cm⁻¹ (2.5 µm) to 400 cm⁻¹ (25 µm) in an atmosphere with minimalized CO₂ and H₂O content through purging by a Parker Balston 74-5041 FTIR Purge Gas Generator. Each FTIR spectrum presented is based on a recording of 32 scans at a resolution of 4 cm⁻¹. In order to ensure satisfactory contact between the ATR diamond crystal and the sample, three or more FTIR spectra were recorded at various locations on the sample. The surfaces of the expanding weather stripping foam samples are relatively porous, which complicates accurate quantitative measurements (height of absorbance peaks) due to varying contact with the ATR crystal for the different samples. Air between sample and ATR crystal results in a weaker absorbance signal. Unless other conditions indicate otherwise (e.g. inhomogenities, impurities, etc.), the FTIR curves with the largest absorbance peaks represent the most correct measurements on one and the same sample with equal ageing time, and hence these curves are chosen as they are assumed to be the most correct ones. Qualitative measurements (location of absorbance peaks at wave numbers) do not represent a problem as long as the contact area is large enough to ensure a sufficient strong measurement signal. The FTIR spectra given in this work have not been ATR corrected, neither with respect to penetration depths nor absorbance band shifts, which both are dependent on the refractive indices of the sample and the ATR crystal (diamond in this case) and the angle of incident radiation. The penetration depth is in addition also dependent on the radiation wavelength, and increases with increasing wavelength (decreasing wave number). That is, non-corrected ATR spectra have much stronger absorbance bands at longer wavelengths (smaller wave numbers) than at shorter wavelengths (larger wave numbers). Note that it should always be stated if an ATR-FTIR spectrum has been ATR corrected or not, e.g. important during computerized database spectra comparison searches. As we in this study is solely comparing the ATR-FTIR spectra measured within this work, there is no need for performing any ATR corrections. Besides, the raw ATR-FTIR data in either transmittance or logarithmic absorbance mode are usually preferred. It should also be
noted that one often do not know the refractive indices of the samples which are measured, thus errors might be introduced in the ATR corrected spectra as the refractive index of the sample is an input parameter in the ATR correction.

The scanning electron microscopy (SEM) material characterization (before and after ageing) was conducted with a Hitachi Tabletop Microscope TM3000 equipped with a QUANTAX 70 energy dispersive x-ray spectrometer (EDS or EDXS) for qualitative and quantitative element analysis where elements in the range from boron (5B) to americium (95Am) may be detected.

The expansion capabilities of the weather stripping foam samples (before and after ageing) were determined by measuring the maximum expansion length and comparing the different samples. Forthcoming investigations will in addition attempt to measure the force or pressure exerted by the samples at a given expansion length for non-aged and aged samples.

3 RESULTS AND DISCUSSION

By visual inspection with the naked eye the expanding weather stripping foam samples looked similar before ageing and after both ageing procedures had been completed. However, the expansion capabilities had changed dramatically. The samples which had been subjected to only the short-term NT Build 495 [26] exposure for 15 days (15 x 24 h) were able to expand to 28 mm, which is similar to the expansion capability of the non-aged samples. That is, one may suspect that no ageing degradation has occurred, although the SEM analysis would at a later stage reveal a somewhat different story. The samples which had been subjected to both the short-term NT Build 495 [26] exposure for 15 days (15 x 24 h) and the long-term heat ageing for 90 days (90 x 24 h) was only able to expand to 15 mm, see comparison in Figure 1.

![Figure 1: Expansion capability of expanding weather stripping foam after ageing. The sample subjected to both the short-term NT Build 495 [26] and the long-term heat ageing is shown to the left, and the sample subjected to only the short-term NT Build 495 [26] exposure is shown to the right, thus demonstrating a large ageing-induced change in expansion capability.](image)

In order to investigate what chemical reactions and changes that may have occurred, FTIR spectra were recorded for both a non-aged, NT Build 495 [26] aged and combined short-term NT Build 495 [26] and long-term heat aged sample (Figure 2). For all three samples, the results revealed nearly identical FTIR spectra being qualitatively similar with some minor quantitative differences. That is, no chemical changes which would have been visible in the FTIR spectra could have taken place. Thus, it may be concluded that the FTIR spectra indicate that the expanding weather stripping foam has not chemically changed significantly
during the ageing. As to avoid surface effects and obtain a more realistic bulk value the FTIR measurement points were located approximately 5 mm from the exposed (e.g. UV) surface and into the bulk material.

![Figure 2: FTIR transmittance versus wave number between 4000-400 cm⁻¹ in the bulk of the expanding weather stripping foam in non-aged and aged conditions.](image)

The SEM analysis disclosed the difference in ageing between the samples as depicted in Figure 3, and revealed that the matrix constituting the pore structure in the expanding weather stripping foam had been only partly damaged by the short-term (15 days) NT Build 495 [26] exposure, whereas the weather stripping foam had been severely damaged by the sequential combined effect of the (i) short-term (15 days) NT Build 495 [26] and (ii) long-term (90 days) heat ageing.

![Figure 3: SEM photos (60 X magnification) of the expanding weather stripping foam, depicting the non-aged sample (left), sample subjected to only the short-term NT Build 495 [26] exposure (middle) and sample subjected to both the short-term NT Build 495 [26] and the long-term heat ageing (right).](image)

Furthermore, closer inspection at larger magnifications with the SEM revealed some crystals in the pore matrix structure of the expanding weather stripping foam, both after the
short-term NT Build 495 [26] exposure and the long-term heat ageing. Elemental analysis through EDS unveiled that these crystals contained calcium (Ca) as visualized in Figure 4. It was concluded that the origin of the calcium-containing crystals stemmed from the water (tap water) spray exposure during the accelerated climate ageing according to NT Build 495 [26]. Hence, this also demonstrates that care has to be taken with respect to the applied water source during accelerated climate ageing experiments.

![Figure 4: Elemental analysis through energy dispersive x-ray spectroscopy (EDS) to the left and SEM photos in the middle (3000 X) and to the right (1000 X) depicting the same area where the EDS was performed, revealing calcium-containing crystals in the pore matrix structure of the expanding weather stripping foam (after long-term heat ageing).](image)

As a first assumption it would have been natural to assume that the short-term (15 days) NT Build 495 [26] ageing with the UV exposure initiated a degradation process which then continued and was accelerated by the higher temperature during the long-term (90 days) heat ageing. However, such an assumption can not be correct as photodegradation processes involve chemical reactions with changes in chemical bonds which should have been clearly visible in the FTIR spectra, whereas Figure 2 demonstrates the opposite, i.e. that no chemical reactions have occurred within the bulk of the aged expanding weather stripping foams.

The expanding weather stripping foam is a porous material, and one may suspect that such a material might take up water during the water spray cycle in the climate ageing according to NT Build 495 [26]. This could especially be true if the material to some extent has a somewhat open-porous structure, whereupon the freezing cycle with the subsequent water-to-ice expansion will put a mechanical strain to the porous matrix in the samples. Thus, during a sufficient number of repeated freezing-thawing cycles a mechanical degradation may take place with a possible outcome as depicted in the middle SEM image in Figure 3. However, if this is the case for the short-term (15 days) NT Build 495 [26] ageing, it is still not clear what happens afterwards in the following long-term (90 days) heat ageing with severe damage to the porous matrix as shown in the SEM images in Figure 3 and the loss of expansion capability as demonstrated in Figure 1.

Further information explaining the results observed in the present study are expected to be obtained during forthcoming experiments which will include separate (stand-alone) long-term heat ageing with no initial climate ageing according to NT Build 495 [26]. Nevertheless, the accelerated ageing of the expanding weather stripping foam represents an interesting example which demonstrates that the powerful FTIR characterization tool may also be valuable in proving that no chemical reactions have actually occurred. Furthermore, the versatile SEM
characterization tool may give further insight to the ageing and degradation processes beyond what mere testing of mechanical properties may disclose. Thus, FTIR and SEM apparatuses should be an integral part of any laboratory handling ageing processes of building materials and components, which unfortunately is often not the case.

4 CONCLUSIONS

Expanding weather stripping foams have been subjected to accelerated ageing by climate ageing and elevated temperature ageing. The durability and ageing of the expanding weather stripping foams have been studied and characterized by their expansion ability, chemical material analysis by Fourier transform infrared (FTIR) spectroscopy and structural analysis by scanning electron microscopy (SEM). These investigations demonstrate that the powerful FTIR characterization tool may also be valuable in proving that no chemical reactions have actually occurred, whereas the versatile SEM characterization tool may give further insight to the ageing and degradation processes beyond what mere testing of mechanical properties may reveal. Hence, FTIR and SEM facilities should be an integral part of any laboratory dealing with ageing processes of building materials and components.

REFERENCES


Abstract
During rain events, the amount of wind driven rain (WDR) that hits a façade depends on the wind speed, horizontal rainfall intensity, orientation, building surroundings and building geometry. Some parts of the building, typically the corners, will be subjected to higher WDR intensities compared to the rest of the building. Depending on the material properties of the exterior surface of the wall and the WDR intensity, excess water may start running down the wall due to gravity. The type of flow (film flow, straight rivulet, meandering rivulet, droplets) is determined by the amount of runoff, which in turn will affect the flow velocity. In this paper a 2D discrete numerical model was developed to simulate the several flow types on a building surface, depending on the material properties and climatic conditions. The model was validated against measurements reported in literature, and verified against reported data from 1D Nusselt solutions.

1 INTRODUCTION
Rain deposition on facades is determined by raindrop distribution, relation of the wind direction to the façade, façade geometry, surroundings, wind speed and terminal velocity: the moment a raindrop collides with a surface the water will either splash or bounce away from the surface, adhere to the wall, be absorbed by the façade material, run down due to gravity or evaporate on the surface. When water runs down the wall, it may pick up dirt particles at the wall surface, transport these to lower parts of the building, and deposit these when the water flow runs dry. This process generates a specific fouling pattern on buildings, affected by the parameters that determine the rain impingement, climatic conditions, as well as the material properties of the different parts of the building. Next to that, runoff may also affect the hygrothermal response of building components, and may increase the risk for water ingress at specific locations.
Raindrop collisions were first studied by Engel [1] in characterizing damage phenomena due to the impact of “water drops” on the surfaces of airplane wings and fuselage. Mutchler and Hansen [2] carried out work on water drop impact on water surfaces (film) and reported higher splash size on a water film for increasing drop size, and the biggest splash was recorded for a water film of one-third drop diameter. It has been established by Couper [3] that runoff from a smooth gloss-painted panel varies according to rainfall intensity with typically about 100% runoff expected at low rainfall intensities (3.15 mm/h), 35% for moderate intensities (6.30 mm/h) and a little over 20% at high intensities (9.45 mm/h). More recently, Abuku et al. [4,5] developed a model on rain impingement on facades, primarily to render better input data for HAM-modeling of porous materials in building envelopes. Insufficient data were published to practically calculate the amount of water that is splashed or bounced away from the façade, water runoff was not analyzed.

A WDR runoff factor was proposed by Choi [6], similar to the runoff calculation according to Straube [18], but this does not take into account absorption and splash, and assumes a perfect film on the building surface. Several authors have described, analyzed and modeled droplet deposition, growth and drop avalanches on substrates [7-15]. Both the simplified models proposed by El Shimi et al. [16], Beijer [17] and Blocken and Carmeliet [18], as well as the FEM models by Kalimares [19], Blocken and Carmeliet [20] and Van den Brande et al. [21] assume perfect film flow without rivulets on facades, neglect raindrop splash and bounce effects, and omit film flow instability due to the viscous momentum imbalance which causes undulatory behavior, as discussed by Takeshi [22]. Literature reviews on water runoff studies [20, 23] conclude that most run-off models incorporate significant simplifications, most of which still needs to be validated (although there is a lack of well documented runoff experiments).

2 MODEL DEVELOPMENT

The numerical simulation model, in order to simulate run-off on a building facade, consists of two parts. The first part, called micro model, is created in order to reduce the needed computation time of the macro model and also to investigate the behaviour of water flow on small scale. The second part, which is called macro model, will estimate the quantity and the behaviour of the run-off of the whole facade.

2.1 Micro model

The absorption in porous materials was modeled by means of a steep-front absorption model [24], which assumes a constant influx until surface saturation, followed by a declining influx as a function of the absorption coefficient and time. For the purpose of evaluating peak runoff rates, the effect of evaporation was disregarded, and the splash effect was, in absence of applicable correlations in literature, simplified by assuming a fixed value. Catch ratios were derived from literature; the model allows to choose between the ISO 15927-3:2009 [25] approach, the model by Straube and Burnett [26], and catch ratio’s reported by Blocken [27], which vary with wind speed and horizontal rainfall intensity. In this model, the catch ratio distribution was assumed independent of the horizontal rainfall intensity (acceptable for intensities above 2mm/h), and a linear dependency of the catch ratio to wind speed (typically a slight overestimation for wind speeds below 4m/s). For the simulations a weather file is used, but the wind directions are then subdivided into circular sectors: e.g. for all wind
directions between -11.25° and 11.25° to the normal of a façade catch ratios for perpendicular wind flow are used.

Figure 1. Visualisation of interpolated catch ratio’s derived from [27] for 0° (left), 45° (middle) and 67.5° (right) incident wind angle and a wind speed of 10m/s.

In the micro model, the raindrop distribution by Best [28] is subsequently used to create a raindrop diameter distribution for the impinging raindrops. Based on the horizontal rainfall intensity, the WDR and raindrop diameter distribution are calculated and used to generate discrete raindrops that impinge on a grid with squares of 10mm by 10mm. Subsequently, the volume of water is stored as a fictional film thickness that is used in the model. Based on a force balance for a droplet adhered to a surface in its critical state condition a formula was developed to calculate when water will start flowing down [29]:

\[
\left( \frac{96}{\pi} \right)^{1/4} \left( \cos(\Theta - \cos(\Theta)) \right) \left( \cos(\Theta) \right)^{3/4} (1 - 3/2 \cos(\Theta) + 1/2 \cos^3(\Theta))
\]

Where \( \rho \) is the liquid density (kg/m³), \( g \) is gravity (m/s²), \( \alpha_c \) is the angle of the plane (0°= horizontal), \( \sigma \) is the surface tension of the liquid (N/m), \( V \) is the volume of the droplet (m³), and \( \Theta_A \) and \( \Theta_R \) are the advancing and receding contact angles respectively (°). For a range of materials a threshold film thickness was calculated, to be applied in homogeneous or heterogeneous façade configurations. Based on the Nusselt solution for the average film flow speed, the water transport from one control volume to the next is calculated:

\[
u = \frac{g \cdot h^2}{3 \cdot \nu}
\]

Where \( u \) is the flow speed (m/s), \( h \) is the film thickness (m), and \( \nu \) is the kinematic viscosity (m²/s). A trace film thickness of 0.0116mm was assumed to account for water droplets that stay behind on the wetted trail. This assumption was based on experiments on a PMMA sheet, using a counting algorithm with AUTOCAD and an arithmetic grid behind the PMMA. These tests showed a mean value of 0.0116mm with a standard deviation of 0.0008mm. Evidently, this will depend on the material properties and type of runoff film, but for simplification and due to a lack of more specific data, the assumption on the film trace thickness was applied for all conditions. Furthermore, rivulet formation was based on the meandering thresholds defined by Legrand-Piteira et al [30]. Then, the maximum rivulet thickness is calculated, based on the criterion of minimum total energy considering the kinetic energy of a two-dimensional velocity distribution of a rivulet [31]:

\[
\delta = \frac{(1 - \cos \theta_b)^{0.22}}{\left( \frac{g \cdot h^2}{15 \cdot \mu^2 \cdot \sigma} \right)^{1/2}}
\]
Where $\theta_0$ is the equilibrium surface contact angle (°) and $\mu$ is the dynamic viscosity (N.s/m²). Note that equation 1 relates to discrete droplet flow, equation 2 is valid for uniform film flow, and equation 3 is derived for rivulet flow. By combining equations 2 and 3 there is an undisputable inconsistency in the calculation method, but in absence of applicable correlations on rivulet flow speed, it still yields the best approach to calculate rivulet runoff. The meandering of the rivulets is included in this model. Consequently, a random roughness of the material surface is assumed. The implementation of roughness is a method to simulate the changes in direction of the flow for specific flow regimes. Water that is flowing down will follow the path with the least resistance. Therefore the grid element with the lowest roughness is consequently the preferred flow path. The effect of prewetting by preceding droplets is simulated by decreasing the roughness of a grid element $(i,j)$ when water flows off this element. Finally, when all possible droplet flow directions have been prewetted, it is assumed that the water will flow towards the adjacent grid element that holds the highest volume of water. Depending on the flow rate, different flow types may occur:

<table>
<thead>
<tr>
<th>Flow type</th>
<th>Transition flow rate [mm³/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Drops</td>
<td>$Q \leq 200$</td>
</tr>
<tr>
<td>Small straight</td>
<td>$200 &lt; Q \leq 470$</td>
</tr>
<tr>
<td>Meandering</td>
<td>$470 &gt; Q \leq 1330$</td>
</tr>
<tr>
<td>Large straight</td>
<td>$Q &gt; 1330$</td>
</tr>
</tbody>
</table>

*Table 1. Transition flow rates*

The direction of individual drops depends on the surface roughness, prewetting and presence of water droplets on the surrounding grid elements. Small straight and large straight always go straight down. Meandering is a complex phenomenon, where instabilities or upstream disturbances may cause sinuous structured flows [32], which in some case may be suppressed leading to braided structures [33]. Note that the flow speed will typically be underestimated because of the assumption that rivulets have the same width as the grid squares (which is an overestimation for small straight and meandering). Therefore the film thickness is underestimated and thus also the film speed. However, a sensitivity analysis shows that this effect is negligible on a building scale. By using a decreasing roughness due to prewetting, channeling of the flow will occur. For drops, small straight and meandering, the film thickness of the flow path increases for increasing flow rates, until the flow type shifts to a higher level. For the large straight, the rivulet starts to expand when the maximum film thickness (equation 3) is achieved. For large straights the model assumes symmetrical rivulets, with maximum film thickness for the central part, and a symmetrical lower flow rate in the outer grid elements. Note that the meandering rivulet type is not modelled correctly. Inadequate information is available to calculate meandering patterns, and it is unclear to what extent such type of flow type will occur during rain events, as the meandering is caused by a momentum imbalance of a closed system that will most likely be disturbed when hit by...
individual raindrops. Consequently, the meandering effect is limited to directional changes due to roughness and prewetting.

2.2 Macro model

As the micro model calculates the impingement of individual raindrops on a grid with cell dimensions of 10mm by 10mm, it cannot be applied to simulate extended periods of time and large surface areas. In the macro model, the grid size is set to 0.5 m by 0.5 m and the water can only flow straight down. The WDR intensity on the facade is determined by the different WDR assessment models. In order to transfer the runoff effects considered in the micro model towards the macro model, an analytical approach was adopted to calculate runoff rates. Simulations with the micro model showed that during a rain event the amount of water adhering to the wall quickly reaches a steady state condition, which indicates that the calculation of the average runoff of a surface of 0.5m by 0.5 can be simplified. For PMMA, sand lime brick and ceramic brick transfer functions were derived, depending on the horizontal rainfall intensity and temperature (affects the kinematic viscosity for flow speed, and threshold value for droplet runoff initiation).

![Figure 3. Macro model input interface (left), macro model output interface (right)](image)

3 COMPARISON WITH LITERATURE

Blocken and Carmeliet [18] have reported a description of a numerical model that is based on the Nusselt solution, which is a simplification for both statistically steady, developed films and for transient developing films. A mass balance between the impinging water and the absorption is made in order to obtain a uniform film with a particular film thickness. The used absorption model is Hall and Hoff, with an absorption coefficient of 0.006 kg/m²s⁻⁰.⁵. A simplified WDR intensity is implemented. To compare the model reported in this paper with the one reported by Blocken, the same WDR intensity is implemented in the program. The maximum value is 1.15 l/m²h at the top (=20 m). The WDR intensity diminishes linear to an intensity of 0.2 l/m²h at 9 m.

To compare both models, the cumulated runoff on the plane is simulated in the Macro model (and averaged to a 1D solution for comparison). The variation of Q along the height of the facade on different time steps can be plotted. Figure 4 provides the comparison of both models. On the left side of the figure, the results obtained by Blocken and Carmeliet are
provided. On the right side, the results of the Macro model are show for identical boundary conditions. The values provided in the macro model are slightly higher compared to those reported by Blocken and Carmeliet, but the difference is rather small. For example, after 60 minutes, the maximum cumulated runoff rate in the macro model is 3 L/m on a height of 12 meters. In the model of Blocken and Carmeliet, this value is 2.5 l/m, on a height of 14 meters. We can conclude that the runoff in the macro model is slightly higher than the values obtained by Blocken and Carmeliet. Comparison of film thickness and runoff ratio versus height show similar trends.

Comparison of macro model simulation results with measurements reported by Beijer [17] on a concrete panel wall, shows that the cumulated runoff shows good agreement for t=18 min and t=36 min, but the macro model overestimates the runoff for t=72 min. Note that the information reported by Beijer is incomplete for a reliable validation, please refer to [18] for a more elaborate comparison of the model reported by Blocken and Carmeliet with the measurements by Beijer. Additional measurements are required for an accurate validation of runoff models.

Figure 4. Cumulated runoff reported by Blocken and Carmeliet [18] (left) and results from the macro model (right).
4 CONCLUSIONS

A numerical model was developed to simulate runoff patterns on building facades. Comparison with other simulations models and measurements reported in literature shows reasonable agreement. A micro model provides a phenomenological approach towards simulating rivulet formation and runoff, whereas a transfer function was used to simulate runoff effects on a building scale. However, several assumptions are made in the model, in absence of physical correlations. More measurements are necessary to develop fundamental insights, and validation cases for runoff models. Subsequent steps in the model development are coupling with a HAM model, implementing water ingress through the façade, modelling heterogeneous configuration such as brick walls, windows and window sills to simulate fouling patterns.

5 REFERENCES

AI RTIGHTNESS OF THE WINDOW–WALL INTERFACE IN WOOD-FRAME WALLS

W. Bracke (1), L. Devos (1), S. Maertens (1), N. Van Den Bossche (1), A. Janssens (1),

(1) Department of Architecture and Urban Planning, Ghent University, Ghent, Belgium – Wolf.Bracke@ugent.be; Nathan.VanDenBossche@ugent.be; Arnold.Janssens@ugent.be

Abstract
Due to the increased focus on airtightness in the current building practice, a better understanding of air leakage rates through different building components and building interfaces is required. This paper investigates the airtightness of the window-wall interface in wood-frame constructions through a literature review and experimental study. Different wall setups, window installation methods and air sealing solutions were tested resulting in 20 different configurations. Air leakages rates ranging from 0.00 up to 3.66 m$^3$/h.m at 50 Pa are measured and recommended configurations and installation techniques are discussed.

The results show that a passive house level airtightness is achievable when using spray-in-place polyurethane foam or airtight membranes, if some general guidelines are followed.

1 INTRODUCTION
Over the last decades, a slow shift in building method has come to front in Belgium. As cavity-brick walls have become standard practice since the 1960’s, recently an increased use of wood-frame buildings was observed [1]. Not only the fast and dry building method, but also the possibility to use thick insulation packages has increased the market share to an estimated 10%, which is expected to keep growing in the next years.

Despite the increasing number of wood-frame buildings, very little research has been conducted on best practice methods for the Belgian construction practice and to what extent the construction method is suited for the local moderate maritime climate is not well understood. Poor craftsmanship by contractors without the appropriate experience have led to prejudices amongst building owners concerning water infiltration and consequent durability problems. Contrary to Scandinavian experience, early wood-frame constructions in Belgium typically showed a poor airtightness when subjected to pressurization tests. Furthermore, as a poor airtightness level can cause high pressure differences over the water sealing material, it can be one of the driving forces for rainwater infiltration. These pressure moderation principles are thoroughly explained by Straube [2]. Besides avoiding possible water infiltration problems, achieving a good airtightness level has many other advantages such as
limiting infiltration heat losses and draught, assuring the proper functioning of the ventilation system and enhancing the sound insulation of the building.

In Belgium, the implementation of the European Energy Performance of Building Directive (EPBD) has resulted in a calculation method for the total energy performance level of a building, for which maximum levels are imposed by the national and regional governments. Increasing environmental concerns, rising energy prices, and more stringent European regulations have led to a very important focus on energy performance in the building industry. The energy performance level includes thermal performance of building components, energy performance of technical installations, use of renewable energy, ventilation, … The airtightness of the building skin is also included in the energy performance level, but as Belgium lacks a history of airtight building due to its moderate climate, this is currently often disregarded and a default conservative value is assumed for the calculation.

The stricter building regulations have led to enhanced thermal performances of the building skin. As more insulation is used, the transmission heat losses have been reduced and the relative share of infiltration heat losses has increased significantly. Contrary to some other countries (e.g. Denmark, UK, The Netherlands), there is no mandatory airtightness limit in Belgium, except for passive houses (n50 < 0.6 ACH). However, performing an airtightness test should be encouraged, as in most cases this will result in a lower energy performance level as current building practice results in an air leakage rate below the conservative assumption in the calculation method.

As a result of the decreasing maximum energy performance levels, an increasing part of the newly built houses are tested for airtightness after completion by means of a pressurization test (2% of all new buildings were tested in 2008, 7% in 2010 and 22% in 2012) [3]. With more stringent energy performance levels, a growing number of pressurization tests are expected to be carried out.

To stimulate the implementation of airtight construction, more research is needed in airtight solutions for different building components and building envelope interfaces. As the air leakage of a building is always a sum of the air leakage through these different components, airtightness targets and practical recommendations for each component would be a practical tool for designers and constructors.

This paper analyses the airtightness of different air sealing solutions for the window-wall interface (WWI) in wood-frame walls. For this specific building envelope interface, airtightness targets for different building airtightness levels were derived by Van Den Bossche [4]. Based on statistical research on the geometry of 200 dwellings constructed in Belgium in the 1990’s, maximum air leakage rates for the WWI were derived for which 75% of the dwellings would reach an airtightness of either 0.6 ACH (passive house level) or 6.0 ACH (standard practice) [5]. Assuming no more than 10% of the overall air leakage rate may be attributed to the WWI, maximum values of respectively 0.33 m³/h.m and 3.30 m³/h.m at 50 Pa pressure difference were derived.

2 LITERATURE REVIEW

The air flow V (m³/h) through an opening is expressed as a power law and is a function of the applied pressure difference ΔP (Pa), flow coefficient C (m³.h.Paⁿ) and the flow exponent n (-) [6].

\[ \dot{V} = C \cdot \Delta P^n \] (1)
In table 1, an overview is provided of airtightness measurements on WWI found in literature. All results are expressed in these quantities and normalized to 50 Pa pressure difference. For converting equivalent leakage areas, the discharge coefficient $C_d$ was assumed to be 1, for the flow exponent an average value of 0.66 was used. When comparing the airtightness of the window-wall interface, the air flow through the interface is expressed per running meter window-wall connection. As workmanship quality can have a significant impact on the airtightness, some of the tests were executed multiple times and a low, mean and high value were reported. Not only results for wood-frame walls, but also for cavity brick walls are enlisted. (PW: Plywood, WB: Window Brackets, JL: Jamb Liner, (N)CC: (non) continuous corners, P: plaster, F_I: foil adhered on interior side of window frame, F_E: foil adhered on external side of window frame).

<table>
<thead>
<tr>
<th></th>
<th>m$^3$/h.m @ 50 Pa</th>
<th>Low</th>
<th>Mean</th>
<th>High</th>
</tr>
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<td><strong>No treatment</strong></td>
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<td>Proskiw</td>
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<td>4.87</td>
<td>4.97</td>
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<td>Van Den Bossche</td>
<td></td>
<td>-</td>
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<td>-</td>
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<tr>
<td><strong>Mineral wool</strong></td>
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<td>1.63</td>
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<td>Relander</td>
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Table 1: Airtightness test results for window-wall interface found in literature [7][8][9]
The absence of an air seal around the windows shows a large spread of air leakage rates, probably caused by the difference in gap size around the windows. Mineral wool, backer rod and self-expanding strips seem unreliable solutions when an ambitious airtightness level is desired. SP-PUR, airtight membranes and tape on the other hand resulted in leakage rates below 0.33 m$^3$/h.m at 50 Pa, thus seem to be good options when the passive house airtightness level is the target. Attention should be paid when applying SP-PUR in combination with window brackets, as leakage paths around these brackets can exist.

3 EXPERIMENTAL RESEARCH

3.1 Test method and error analysis

The airtightness of the window-wall interfaces was measured in a standard calibrated test rig according to NBN EN 12114 [10]. After three pulsations of 660 Pa, the airflow was measured at pressure differences of 50 – 100 – 150 – 200 – 250 – 300 – 450 – 600 Pa, both for positive and negative pressure. These measurement points are controlled for outliers using Chauvenet’s criterion [11]. Data were rejected if the total probability for one suspect value within the entire sample group was below 0.5. A power law function is fitted through the measurement results and the leakage is calculated for a 50 Pa pressure difference. To define the confidence interval for this calculated result, the following aspects were taken into account:

- Error on the air flow measurements: based on 40 measurements, a 95% confidence interval of 3.965% was derived [4]
- Errors on the corrections for reference conditions: based on Monte-Carlo analysis of 1000 simulations, this is limited to 0.14%
- Error on the linearization and ‘best fit to power law’ $\dot{V} = C.\Delta P^n$ according to an inverse $t$-distribution, which give the uncertainty of the flow coefficient C and flow exponent n.
- Error propagation in power law

$$\sigma_{\dot{V}}^2 = \sigma_C^2 \sigma_n^2 \left[ (\Delta P^n)^2 \cdot \frac{\sigma_C}{\sigma_n} + (C.\Delta P^n.\ln(\Delta P))^2 \cdot \frac{\sigma_n}{\sigma_C} + 2C.\Delta P^{2n}.\ln(\Delta P).r \right]$$

Where $r$ = pearson correlation coefficient. Based on 30 measurements, a conservative value of -0.5 was assumed because C and n are negatively correlated.

As a result of this process, errors at 50 Pa pressure difference usually lie in the order of magnitude of 10% of the mean value (variation coefficient), while conducting a single measurement at 50 Pa would result in an error of only 3.965%. When measurements on very airtight construction details are conducted, slightly deviating values at low pressure differences can lead to confidence intervals up to 50% or more.

The air leakage through the window-wall interface cannot be measured separately, but can be derived from subtracting the extraneous air leakage through the wall from the overall air leakage. The error on the leakage through the window-wall interface will thus be a combination of the error on the overall leakage and on the extraneous leakage. As the errors are primarily the result of the fitting of the power law, which is uncorrelated for different
measurements, the total error is calculated by adding in quadrature the errors of both measurements.

3.2 Test setup and results

3.2.1 Installation methods

The tests are executed on a full-scale setup built in a steel test rig. A non-operable wooden window measuring 123 cm wide and 155 cm high was installed in a wood-frame wall of 228 cm by 196 cm. The connection between the insulating glass unit and wooden window frame was taped off to avoid leakage through the window itself.

Two different wall types are tested: The first type (W1) consists of bituminous impregnated fibreboard at the exterior side. This board functions as a rain- and windscreen but cannot be regarded as an airtight layer. The airtightness is achieved by the OSB at the interior side of the wall. The window was installed in line with the structure of the wall itself. The second wall type (W2), relates to a different configuration where additional insulation is installed in the cavity between the interior wood-frame wall and the exterior leaf. The latter wall configuration has two main advantages: the continuous layer of insulation reduces the thermal bridge effect caused by the solid wood elements, and there is no need to increase the volume of wood in the wall when increasing the thermal resistance. For this wall, it was assumed that the window was installed in line with the continuous insulation layer in the cavity, and hence protrudes in respect to the OSB panels installed at the exterior side of the wooden frame. It should be noted that the use of OSB on the exterior side might lead to hygrothermal problems: as the OSB is a rather vapour-tight material, there is a risk for interstitial condensation inside the wall if there is no adequate vapour barrier on the inside.

Two common air-sealing methods are evaluated for the WWI: an airtight foil and spray-in-place polyurethane foam (PUR). For each case two window installation methods were tested: installation with window brackets (WB), and installation with surrounding plywood frame (PW). For W2, the use of a self-expanding strip (SES) was also evaluated.

Two types of airtight foils are tested. The first consists of a laminated polyester membrane and contains two self-adhesive strips (F2): one strip to adhere the foil to the side of the window or the plywood frame and one butyl-strip which was designed for a good adhesion on a brick wall. The question then rises whether this butyl-strip is also a good solution for adhesion on the OSB. For comparison, an airtight foil consisting of only one self-adhesive strip (F1) is installed. The strip is used to adhere the foil to the side of the window or plywood frame, and the foil is attached to the OSB by using a one-component silyl-terminated polymer sealant.

When attaching the foil to the side of the window or plywood frame, it might seem evident to start the foil at one the corners of the window or plywood frame but as corners are typically weak spots in the air sealing, it is recommended to start in the middle of one of the sides. The end part of the foil should overlap the beginning by at least 40 cm and the overlapping areas are glued together using a sealant prescribed by the manufacturer of the foil.

The installation of the 2D foil around the 3D corner requires some attention when applying the foil continuously over the corners (CC). As the perimeter of the wooden window is smaller than the perimeter of the window opening, the foil is folded at the corners to provide more length at the outer side. In this way, no tension is present at the corners and the risk of a loosening connection between foil and OSB or window frame can be avoided. For W2, the
corners were executed both continuously as non-continuously (NCC) by applying horizontal and vertical strips of foil in overlap for the latter case. The SP-PUR is a one-component low-expansion open cell foam with a high elastic recovery, and was applied with a foam applicator gun system. The foam manufacturer recommends spraying water before applying the foam, as a higher relative humidity promotes the adhesion and expansion of the foam. For W2, the difference between both dry (D) and wet (W) window-wall interface was examined.

Before each measurement, a reference measurement was conducted to determine the extraneous air leakage through the wood-frame wall and the test setup itself. For these measurements, the window area was sealed off using an airtight box. The box was pressed against the wall and the connection between both was made airtight using a closed-cell foamband, which was compressed between the plywood box and the wall. On average, a leakage rate of $0.19 \pm 0.02 \text{ m}^3/\text{h.m}$ at 50 Pa was found, which will show to be rather high compared to the air leakage rates through the window-wall interface itself. The effect of this extraneous air leakage is substantial and significantly increases the calculated confidence intervals in the measurement results.

### 3.2.2 Test descriptions and results: wall 1

Six configurations were tested. Figure 1 shows the corresponding window-wall interface details, which might clarify the description of the configurations.

A) A foil was continuously adhered to the side of the window before installation. Standard window brackets were screwed through the foil, onto the window, and the window was installed in the wood-frame wall. The foil was connected to the OSB using polymer sealant resulting in an air leakage rate of $0.03 \pm 0.07 \text{ m}^3/\text{h.m}$ at 50 Pa.

B) The use of a self-adhesive butyl strip instead of a polymer sealant is evaluated. During an overpressure test, leakage at the corners of the foil became evident, as the foil started ballooning due to high pressure differences. To what extent this effect was caused by premature failure due to the lack of firmly pressing during installation or an inadequate drying period of the butyl strip before testing is unclear. However, the continuous application of the butyl-strip around the corners requires a lot of attention and introduces a significant risk for installation errors.

Figure 1: Window-wall interface details wall 1
C) Before the application of the SP-PUR, standard window brackets were again used to mount the window. In Belgium, unlike other countries as Germany, windows are never mechanically fixed by bolting through the frame itself, as the installation may lead to large deformations of the window frame. The opening between the wood-frame and the window was filled with SP-PUR. As the depth of the wood-frame is limited to 80 mm, the window brackets are not completely covered with foam. Probably an adhesion problem exists between the SP-PUR and the metal brackets, as leakage paths were easily detectable.

D) A plywood frame was built around the window and used instead of mounting brackets to fasten the window to the wood-frame wall. The airtight foil was adhered to the plywood using the self-adhesive strip. The frame was installed, and put in the correct position in respect to the window opening by using small pieces of plywood at the sides. The foil was fixed to the OSB by using the sealant, as this was shown to be a more feasible solution than the butyl-strip. The test with plywood frame and foil (test A) showed a very similar air leakage value compared to the test with mounting brackets and foil. Note that additional tests (E) pointed out that in some cases air leakage might be found through the end face of the plywood. Small defects in de veneer layers of the plywood caused leakage paths which were responsible for an additional $0.12 \pm 0.09 \text{ m}^3/\text{h.m}$ at 50 Pa, thus it can be advised to apply airtight tape over the face ends of the plywood frame, or an adequate width of plywood to avoid continuous defects.

F) The last installation relied on SP-PUR for sealing the cavity between the plywood frame and the window opening. The plywood frame was again centred in the opening using plywood blocks. As these protruded into the cavity, the foam could not surround the blocks and during testing, leakage paths were detected, similar to the leakages around the window brackets.

Table 2: Test results wall 1

<table>
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<th>Average</th>
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</thead>
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<td>$V_{50}$</td>
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</table>

3.2.3 Test descriptions and results: wall 2

Fourteen configurations were tested. Figure 1 shows the corresponding window-wall interface details, which might clarify the description of the configurations.

In wall 2, a different type of window brackets was used, as the window was installed in line with the insulation in the cavity. As the insulation itself was not expected to have an important impact on the airtightness, it was omitted during testing.

G) A foil with two self-adhesive strips was applied to the side of the window and the OSB. The foil was folded at the corners, similar as in the configuration of wall 1, to provide a continuous airtight connection. The end parts of the foil were again placed in sufficient overlap and glued together. During the air leakage test, weak spots were again detected at the
corners where the butyl-strip was installed on the OSB. Fixing the foil at the corners using polymer sealant resulted in a significant lower air leakage rate (test H). Sealing the edges of the foil caused an additional decrease of the air leakage rate, but the effect was not statistical significant (test I).

Figure 2: Window-wall interface details wall 1

J) The foil was applied non-continuously; in four strips overlapping at the corners. This installation method turned out to be much more convenient, and can easily be executed by one person, contrary to the continuous installation. Next to that, a lower air leakage rate was measured compared to the continuous application without additional sealing.

K) A foil was connected with self-adhesive strips to the interior side of the window and to the wood-frame construction. In this way, no connection exists between the foil and the airtight OSB layer at the exterior side of the wall, so large leakages can be expected, as the foil does not cover the joint between the OSB and the wooden stud adjoining the window. Even though this installation method is not to be recommended and leads to very high air leakage rates, the effect of the sealed corners (test L) and the non-continuous installation (test M) showed again to have a significant impact.

N) The use of SP-PUR in combination with window brackets was analysed. To keep the test setup clean from PUR for further testing, thin pieces of plywood were connected on the side of the wood-frame and the side of the window. The connections between the pieces of plywood and the setup were made airtight using polymer sealant. When analysing the detail in Figure 2, the use of PUR might seem unrealistic for the installation of a window protruding into the cavity. However, in reality other building materials such as a window sill and insulation layers close off a cavity which can be filled using SP-PUR. For the first test, the SP_PUR was executed without wetting the cavity. This installation resulted in a considerable air leakage rate and again, the window brackets showed to be the weak spots. In a subsequent configuration, the window-wall joint was wetted using a plant spray before applying the foam
The effect of the wetting is remarkable as the air leakage rate dropped by 90%. Probably the SP-PUR did not completely fill the cavity between the window and wooden post, as the width of 50 mm is rather large. In practice, a maximum width of 40 mm is often cited, but there are no strict guidelines in respect to the maximum width that can be sealed with foam, as it is strongly affected by the relative humidity in the air. In this situation, the additional expansion of the foam due to the wetting may have had an important impact.

P) A plywood frame was screwed on the side of the window and connected airtight using a polymer sealant. The frame was installed in the correct position using small plywood blocks. A foil with two self-adhesive strips was connected to the plywood frame and the OSB, and additionally sealed around corners and edges using polymer sealant. This resulted in an air leakage rate of 0.08 $\text{m}^3/\text{h.m}$, which is very similar to the alternative installation with window brackets.

Q) The foil was again applied non-continuously at the interior side of the wall, connecting the end face of the plywood frame and the wood-frame construction. Contrary to tests K, L and M, airtight tape was placed over the connection between the wooden post and the OSB preventing air leakage through the wall. For the previous tests, only the PUR or airtightness foil was installed and the interior finish was omitted. For this case, the impact of an interior finish was also evaluated (test R). Gypsum fibreboard was placed over the end face of the plywood frame to decrease the tension on the airtight tape caused by ballooning of the foil when no interior finish was present. As the air leakage rate dropped from 0.25 to 0.16 $\text{m}^3/\text{h.m}$, the interior finish had a significant impact.

S) A self-expanding strip was applied in the cavity between plywood frame and the window opening. The cavity was 18 mm wide, whereas the SES application range was 10 – 20 mm. The SES was installed continuously, causing an incomplete filling at the corners. Consequently, very large airflow rates were observed during testing. Note that this confirms the findings of Relander, please refer to Table 1.

T) PUR was sprayed between the plywood and wood-frame. In this setup, the cavity was 35 mm wide and was wetted before the application of the foam, as previous tests confirmed this measure to be effective. Contrary to a similar test on wall 1, the plywood installations blocks did not protrude into the cavity and consequently it was possible to completely surround the blocks with foam. Attention was paid to the correct installation of the foam: the cavity was filled starting from the bottom and the foam gun was adjusted precisely to spray the correct amount of foam. This resulted in an almost completely airtight window-wall interface.

### Table 3: Test results wall 2

<table>
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<tr>
<td>I</td>
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</tr>
<tr>
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<td>0.23</td>
</tr>
<tr>
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<td>M</td>
<td>1.36</td>
<td>0.90</td>
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4 DISCUSSION

The air leakage rate of 20 window-wall interface configurations was measured both in positive and negative pressure. For the largest part of the solutions, a $V_{50}$-value lower than 0.33 m$^3$/h.m was measured, which means they can be used in passive house constructions without the risk of losing a significant share of the total allowed air leakage of 0.6 ACH [4].

The design of a continuous airtight layer proved to be essential: when the air sealing layers are not connected, large air losses were observed. The use of self-expanding strips as an airtightness solution is also not recommended.

In general, very airtight solutions can be achieved when using airtight foils or spray-in-place polyurethane foam. However, large relative differences between execution methods were observed.

Regarding the use of foils, the main leakage paths were obviously observed at the corners, where the continuous application caused considerable air leakage when no additional polymer sealant was used next to the butyl strip. The continuous application also proved to be an inconvenient technique, which is hard to execute properly. As a result, folds in the foil around the corners might cause problems when applying a plaster layer. However, as wood-frame constructions are seldom executed with a plaster layer over the – already airtight – OSB, this is more specifically a problem in brick walls.

Applying separate horizontal and vertical strips of foil was experienced to be a much easier installation technique. No tension or folds around the corners exists which can cause the previously discussed problems.

The use of sprayed polyurethane foam was found to be a more fool-proof air sealing method, though some recommendations should be followed. Most specifically, the wetting of the application area is important for a good expansion and adhesion of the foam. Especially in cold and dry circumstances this should not be disregarded. Furthermore, the filling of vertical cavities should be started from the bottom up to avoid a phenomenon called ‘cell collapse’ and the width of the cavity should not exceed 40 mm.

Though very good air leakage rates were measured in this research, the long-term performance of SP-PUR is still under investigation. As UV radiation deteriorates the foam quality, precautions should be taken during prolonged construction phases.
REFERENCES


A MODEL OF CARBONATION AND A TECHNIQUE TO DETERMINE THE DEGREE OF CARBONATION

Katja Fridh, Lars-Olof Nilsson

Division of Building Materials, Lund University, Lund, Sweden – katja.fridh@byggtek.lth.se; lars-olof.nilsson@byggtek.lth.se

Abstract
To be able to estimate the service life due to carbonation induced corrosion and the CO₂-uptake of different types of concrete is the degree of carbonation needed. This paper proposes and demonstrates a measuring technique together with a carbonation model. The carbonation model which reveals the importance of the degree of carbonation is based on carbonation as a moving boundary where the rate is dependent on the amount of CO₂ in the air, the diffusion coefficient of CO₂ in carbonated concrete and the amount of CaO which can be carbonated. The degree of carbonation is obtained by dissolution of concrete in HCl and monitoring the increase of pressure the release of CO₂ creates. This pressure is then translated through calibration curves to a CaCO₃ content. The liquid is then analyzed regarding the content of calcium and the degree of carbonation is the ratio of CaCO₃ to Ca. With this information it is possible to calculate the amount of CO₂ uptake and the progress of carbonation in concrete.

Keywords:
Degree of carbonation, carbonation model, measuring technique, CO₂-uptake
1 INTRODUCTION

Carbonation of concrete has historically been interesting due to its role in corrosion of reinforced concrete constructions. When the carbonation front reach the reinforcement is the pH lowered to levels that break the passivity of the steel bars and corrosion is possible. The simple measuring technique used worldwide is applying phenolphthalein on a newly cut surface and if the surface remains uncoloured is the concrete regarded as carbonated and if the surface become pink/purple is the concrete regarded as uncarbonated. The thickness of the uncoloured layer is referred to as the depth of carbonation. Typical results of this kind of measurement on outdoor reinforced concrete structures can be found in several investigations [1-3]. How the depth of carbonation varies with surrounding climate and concrete quality is also available and different kind of models has been presented [4-7]. What really is measured is a pH-change. From about pH 8 is the indication colour pink/purple and at lower pH is the indication colour transparent. This information is fully satisfactory when the risk of corrosion is the main object of the study.

A relatively new question is if it is possible to estimate how much carbon dioxide this carbonation of the concrete will take up during its service life [8-12]. The manufacturing of cement contributes with 5 % to the global production of carbon dioxide. In a life cycle analysis of the environmental impact of concrete should not only this emission of carbon dioxide be accounted for but also the uptake should be evaluated. Knowledge about the relation of depth of carbonation obtained by phenolphthalein measurement and the degree of carbonation in this depth is needed to be able to use previous obtained data and to be able to calculate the CO$_2$ uptake. This paper presents a measuring technique were it is possible to determine the degree of carbonation.

2 THE UPTAKE OF CO$_2$

2.1 Degree of carbonation

Concrete subjected to an environment containing carbon dioxide will begin to absorb it and during the reaction of CaO and CO$_2$ forming CaCO$_3$ and the excess CO$_2$ will continue further into the concrete and new CaCO$_3$ can be formed. Different constituents of the cement paste (C$_3$S, C$_2$S, C$_4$AF, C$_3$A, CSH) will be more or less willing to carbonate in different environments and different figures on the degree of carbonation is presented e.g.; 75 % [13] and also 100 % [8]. This numbers are theoretical assumptions and in this paper will a technique be presented where the degree of carbonation can be determined experimentally.

2.2 A model to calculated the CO$_2$ uptake

In a proposed model [14] is the important material properties for the carbonation process derived and the importance of the degree of carbonation is apparent.
The proposed model of carbonation is shown in Figure 1. The carbonation is regarded as a front moving inwards from the surface. The concentration of CO$_2$ is $c$ at the surface and zero at the front. The mass balance states that when the concentration of CO$_2$ is zero at the front all available CO$_2$ has to be consumed at the front. The flow $J$ (kg/m$^2$s) of carbon dioxide during a timestep $dt$ has to be bound into a unit of concrete (a, kg/m$^3$) and therefore extents the depth of carbonation $dx$ (m).

$$J_{CO_2} \cdot dt = a \cdot dx_{CO_3}$$  \hspace{1cm} (1)

The flow $J$ (kg/m$^2$s) can be described as

$$J_{CO_2} = D_{CO_2} \frac{\Delta c}{\Delta x} = D_{CO_2} \frac{c}{x_{CO_3}}$$  \hspace{1cm} (2)

where $D_{CO_2}$ is the diffusion coefficient of CO$_2$ in carbonated concrete (m$^2$/s), $c$ is the concentration of CO$_2$ in the air (kg/m$^3$) and $x_{CO_3}$ is the depth of carbonation (m). (2) then becomes

$$\frac{D_{CO_2} \cdot c}{x_{CO_3}} \cdot dt = a \cdot dx_{CO_3}$$  \hspace{1cm} (3)

Integration of both sides gives the depth of carbonation, $x_{CO_3}$

$$\int_0^{x_{CO_3}} \frac{D_{CO_2} \cdot c}{a} \cdot dt = \int_0^{x_{CO_3}} dx_{CO_3} \Rightarrow x_{CO_3} = \sqrt{\frac{2 \cdot D_{CO_2} \cdot c}{a} \cdot t}$$  \hspace{1cm} (4)

The depth of carbonation is found to be dependent of the diffusion coefficient of CO$_2$ in concrete ($D_{CO_3}$, m$^2$/s), the concentration of CO$_2$ in the air, ($c$, kg/m$^3$), the time ($t$, s) and the amount of CO$_2$ that is needed to move the carbonation front $dx$ (a, kg/m$^3$). The diffusion
coefficient is assumed to be constant in the section since the CO\textsubscript{2} always will flow through a carbonated layer. The variable a, is dependent on the following,

\[ a = C_{\text{con}} \cdot \frac{CaO}{C} \cdot \frac{(CaO)_{\text{co}_2}}{CaO} \cdot \frac{M_{\text{co}_2}}{M_{\text{ca}O}} \]  \hspace{1cm} (5)

\( C_{\text{con}} \) = mass of cement (kgcement/m\textsuperscript{3}concrete)

\( CaO/C \) = amount of CaO in the cement (kgCaO/kgcement)

\( (CaO)_{\text{co}_2}/CaO \) = part of CaO which carbonate (kg/kg) – degree of carbonation

\( M_{\text{co}_2} \) = Mole weight of CO\textsubscript{2} (kgCO\textsubscript{2}/kmol)

\( M_{\text{co}_2} \) = Mole weight of CaO (kgCaO/kmol)

The amount of CO\textsubscript{2} that is absorbed in the carbonated layer \( m_{\text{co}_2} \) (kg/m\textsuperscript{2}) is

\[ m_{\text{co}_2} = X_{\text{co}_2} \cdot a \]  \hspace{1cm} (6)

and (4) and (6) gives the final expression

\[ m_{\text{co}_2} = \sqrt{\frac{2 \cdot D_{\text{co}_2} \cdot c}{a} \cdot \sqrt{t} \cdot a} \]  \hspace{1cm} (7)

The model show all the material properties needed to calculate how much CO\textsubscript{2} that is absorbed into a certain concrete. The variable a, is obviously an important factor since it appears twice. Most of the material properties are known if the recipe of the concrete is known. But one is not well known, the degree of carbonation. This property has to be measured for different concrete in different climates, even indoors since the goal is to be able to calculate total amount of absorbed CO\textsubscript{2} in all concrete.

3 MEASURING THE DEGREE OF CARBONATION

This present technique was first developed by Nilsson and Möller and presented in a licentiate thesis [15]. The principle is simple. When carbonated concrete is dissolved in hydrochloric acid the CO\textsubscript{2} that has previously been bound by carbonation is released. If this is done in a gastight container, the pressure will rise. This pressure increase is measured and calibration measurements with pure calcium carbonate gives what amount of carbon dioxide the pressure increase caused by the sample corresponds to. A part of the acid solution is analysed by ICP-AES. With the amount of calcium and the amount of carbon dioxide, the degree of carbonation can be calculated.

\[ \text{degree of carbonation} = \frac{\text{amount of CO}_2}{\text{amount of Ca}} \]  \hspace{1cm} (8)
3.1 Equipment

Figure 2. The measuring device, before (left) during and after (right) a measurement.

The basic cylinder has the internal height 103 mm and a diameter of 100 mm.

3.2 Measuring procedure

For a measurement, 0.35 l of HCl (17.5% by weight) is poured into the airtight stainless steel vessel. Crushed concrete is then placed in the sample holder. The sample is chosen to contain as much paste as possible. The lid is closed with six screws and between the lid and the vessel an o-ring is placed. The valves are closed and the vessel is tilted, figure 2. The pressure after six minutes is noted and the vessel is then opened and a sample of the liquid is sent to analysis by ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy).

4 MEASUREMENTS

4.1 Calibration measurements

To be able to translate the pressure increase to amount of carbon dioxide calibration measurement with pure CaCO₃ was made, figure 3. These measurements were performed according to section 3.2. All the measured pressures from measurements on concrete where then translated with this calibration curve.
4.2 **Degree of carbonation in concrete**

Samples from an exposure site in Sweden, Smöjen and two Swedish office buildings (Kungsör and Limhamn) were analysed. The results are shown in Table 1 and figure 5.1. The depth of carbonation was determined by thin sections.

**Table 1: Degree of carbonation in samples from Smöjen and Kungsör.**

<table>
<thead>
<tr>
<th></th>
<th>Degree of Carbonation [molCO$_2$/molCa]</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Smöjen</strong></td>
<td></td>
</tr>
<tr>
<td>19310 - Surface</td>
<td>0.54</td>
</tr>
<tr>
<td>19310 – Center of carbonated layer</td>
<td>0.53</td>
</tr>
<tr>
<td>1866 - Surface</td>
<td>0.64</td>
</tr>
<tr>
<td>1866 - At the carbonation front</td>
<td>0.51</td>
</tr>
<tr>
<td><strong>Kungsör</strong></td>
<td></td>
</tr>
<tr>
<td>0-5 mm</td>
<td>0.67</td>
</tr>
<tr>
<td>6-11 mm</td>
<td>0.21</td>
</tr>
</tbody>
</table>
The concrete in samples 1866 was a w/c-ratio 0.60 and its binder was a CEM I. The depth of carbonation measured by thin section was 12 mm and two samples of 5 mm were sawn without adding water. The samples had been protected against rain.

The concrete in samples 19310 was a w/c-ratio 0.80 and its binder was a CEM I. The depth of carbonation measured by thin section was 23 mm and three samples of 5 mm were sawn without adding water. The samples had been protected against rain.

The concrete from Kungsör was from an 22 years old inner wall and its binder was a CEM I.

From the house in Limhamn a core was drilled through an inner slab, the core was split and phenolphthalein was applied. The carbonated layer was sliced up and crushed to obtain a profile on the degree of carbonation. The binder was a CEM I and the concrete was 44 years old.

Figure 4. Core from an inner slab and its profile of degree of carbonation.

5 DISCUSSION

The presented measuring technique is able to determine the degree of carbonation on relatively large samples of concrete (5g) which is important so the sample is representative. If the depth of carbonation is determined by e.g. thin section or phenolphthalein it is possible to determine a degree of carbonation profile for the carbonated layer. It was then found that the front was not really a front and that the concrete used in this measurement (CEM I) contained carbonates in the aggregates or in the cement or in both since the non-carbonated concrete also had a degree of carbonation which was fairly constant.
6 CONCLUSIONS

It is a very important task to able to assess all the environmental impacts of concrete. Quantifying the CO$_2$-uptake is one. To be able to do that it is important to determine the degree of carbonation and the depth of carbonation. The presented measuring technique and the proposed model is a way of accomplishing that.

ACKNOWLEDGEMENTS

The authors wish to thank the Swedish Consortium for financing concrete research for funding the studies presented in this paper.

REFERENCES

NONDESTRUCTIVE DETERMINATION OF CHLORIDE ION USING AG/AGCL ELECTRODE PREPARED BY ELECTROCHEMICAL ANODIZATION

F. Pargar (1), D.A. Koleva (1), O. Copuroglu (1), E.A.B Koenders (1) and K. van Breugel (1)

(1) Civil Engineering and Geosciences, Department Materials & Environment, Delft University of Technology, Stevinweg 1, 2628 CN Delft, The Netherlands

Abstract
The stability of the chloride ion selective electrode in highly alkaline solutions must be ensured; otherwise, the application in concrete is not feasible. A simple solution to this problem is to increase the amount of compact and uniform AgCl film on Ag wire by adjusting the current density and anodizing time. In this study the Ag/AgCl electrodes were prepared in four different current densities and the microstructures of the electrodes were compared and the potentiometric response of the electrodes to chloride in water, synthetic concrete pore solution and cement extracted solution was systematically studied.

1 INTRODUCTION

The analysis of chloride content in hardened cement-based materials is not an easy task. The traditional way of powder drilling, dissolution in acid and chloride analysis by titration (Volhard method) is a destructive and a time consuming method. The results are generally related to the total chloride content \cite{1}, whereas determination of free chloride needs to take into account additional phenomena within sample preparation (e.g. physical chloride binding within filtration).

Ag/AgCl electrodes are stable, easily prepared, and have long been used in electrochemistry as chloride ion selective or reference electrodes. Solid Ag/AgCl electrodes are also used as embeddable sensors in cement-based materials. They are sensitive to (primarily) chloride ions and ideally exhibit a certain electrochemical potential that depends on the chloride ion activity in the medium and on temperature. Due to the small dimensions of such chloride sensors they allow highly localized measurements, e.g. accuracy at the depth of the reinforcement, rather than average values over the comparatively large concrete volume under investigation, which is the case when using other techniques. Moreover, the chloride concentration can be monitored non-destructively and continuously \cite{2}.
The Ag|AgCl electrodes are based on two equilibriums: the electrochemical equilibrium involving formation of interfacial potential and the solubility equilibrium between the cation and its sparingly soluble salt. The electrochemical equilibrium is \(^3\):

\[
2\text{AgCl} + 2\text{OH}^- \rightleftharpoons \text{Ag}_2\text{O} + 2\text{Cl}^- + \text{H}_2\text{O}
\]  

(1)

And the solubility equilibrium is:

\[
\text{AgCl} \rightleftharpoons \text{Ag}^+ + \text{Cl}^-
\]  

(2)

Combining two equations above, the equation below can be written:

\[
\text{AgCl} + e^- \rightleftharpoons \text{Ag} + \text{Cl}^-
\]  

(3)

At 25 °C the solubility product of AgCl is \(K_{sp} = 1.8 \times 10^{-10}\). Because AgCl is relatively insoluble, it maintains the constant activity of silver ions at the electrode \(^3\).

When \(\text{Ag}^+\), \(\text{OH}^-\), \(\text{Cl}^-\) and \(\text{SO}_4^{2-}\) are presented together in the medium, thermodynamically favourable is the reaction of \(\text{Ag}^+\) with \(\text{Cl}^-\), followed by reactions, involving \(\text{OH}^-\) and \(\text{SO}_4^{2-}\). Associated with the lower concentration of \(\text{SO}_4^{2-}\) in the pore solution of hydrated cement-based materials (0.1-18.2 mmol/L) in comparison with \(\text{OH}^-\) (18-360 mmol/L), the reaction between \(\text{Ag}^+\) and \(\text{SO}_4^{2-}\) is negligible \(^4\).

At high pH values, the AgCl membrane becomes unstable and can be, partly or completely, converted into \(\text{Ag}_2\text{O}\). By continuously transforming the electrode surface into \(\text{Ag}_2\text{O}\)-covered one, the electrode acts as a pH sensor rather than a chloride sensor. The formed silver oxides or hydroxides do not necessarily adhere to the membrane surface \(^5\) and the activity of silver near the surface (and hence electrode potential response) is determined by exchange equilibrium \(^3\):

\[
2\text{AgCl} + 2\text{OH}^- \rightleftharpoons \text{Ag}_2\text{O} + 2\text{Cl}^- + \text{H}_2\text{O}
\]  

(4)

With respect to stability of the Ag|AgCl sensors, the microstructural properties of the AgCl layer or the Ag|AgCl interface are of great importance. For example, instability of the sensors is known to be related to the weak bonding between the AgCl layer and the Ag substrate, when sensors are produced at higher current density regimes; similarly, stability issues are at hand when a very thin AgCl layer forms under low current density regimes \(^6\). Therefore, an optimum choice of current regime is an important aspect in sensor preparation.

A vexing problem associated with the most reported thin-film Ag|AgCl electrodes has been their poor stability. Open circuit potentials are typically observed to be stable for a time period in the range of a few minutes to a few hours. Instability has generally been attributed to dissolution of the very thin layer (few nm) of sparingly soluble AgCl and the subsequently developed mixed potentials at the electrode|solution interface \(^7\). A simple solution to this problem, which does not require polymer coatings, is to increase the amount of silver chloride on the surface of silver.

Some authors report that no significant effect on the potential value of the sensor was induced by anodization current density of higher than 0.2 mA/cm\(^2\) \(^8\). Studies \(^9\) showed the influence of different current densities (0.2, 0.4, 0.6 mA/cm\(^2\)) on the stability of the electrodes in alkaline solution with 1 M chloride ions. The potential of the sensors were monitored over time and based on their stability, the optimum current density was determined. The results showed that the potential of the sensor anodized in regime of 0.4 mA/cm\(^2\) for 2 hours was stable over 3 months. The instability of the other sensors was assumed to be due to the thin
AgCl layer, formed under lower current density and a weak bonding between AgCl layers and Ag substrate at high current density.

In this paper the relationship between the potential of the sensors and the microstructure of the formed AgCl layer was studied. AgAgCl sensors were prepared in four different anodization regimes. Microstructural observations of the AgCl layer were correlated to potentiometric response and stability in chloride-containing model solutions.

2 EXPERIMENTS

Silver wires of 1 mm in diameter (99.99% purity) were cleaned for 2 hours in concentrated ammonia and immersed in distilled water overnight. Then, they were anodized for one hour in a 0.1M HCl solution at the current density of 0.5, 1, 2 and 4 mA/cm$^2$ (Table 1); platinum mesh served as a cathode. The colour of deposited AgCl layer at different current densities was brown-black except the sensor anodized at 4 mA/cm$^2$ which was greyish-white.

The electrodes were stored out of direct sunlight before further testing. The anodized silver wire was welded to a copper wire and the non-anodized and welded zones were protected with an epoxy resin.

In order to study the cross section of the Ag|AgCl sensors, the Ag wire was narrowed in a definite section prior to anodization to make it easier to break by stretching the sensor from two sides of the notch. Then the sensor was investigated using scanning electron microscope under SEM, energy dispersive spectroscopy (EDS).

Table 1: Example of construction of a table

<table>
<thead>
<tr>
<th>Regime</th>
<th>Current density (mA/cm$^2$)</th>
<th>Duration (hour)</th>
<th>Measured thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>0.5</td>
<td>1</td>
<td>6~10</td>
</tr>
<tr>
<td>II</td>
<td>1</td>
<td>1</td>
<td>12~17</td>
</tr>
<tr>
<td>III</td>
<td>2</td>
<td>1</td>
<td>~20</td>
</tr>
<tr>
<td>IV</td>
<td>4</td>
<td>1</td>
<td>~40</td>
</tr>
</tbody>
</table>

3 RESULTS

3.1 Microstructural study

Fig. 1 shows the morphology of the AgCl layers, formed on the Ag substrate at different current densities in 0.1 M HCl. As expected and also observed from the cross-sections (Fig.1) with increasing the current density the thickness of AgCl layer was increased from 6µm to 40µm (Table 1). At current densities of 0.5 and 1mA/cm$^2$, AgCl packed-piled particles were generated on the Ag substrate. At higher current density of 2mA/cm$^2$ small-unpacked AgCl particles were detected close to the silver substrate and the surface became a mosaic of complex particle shapes with some remaining particles. Further increasing the current density to 4mA/cm$^2$ did not bring new features to the top and cross section views though the morphology kept changing as the current increased.

SEM photos show that the AgCl layer is dense but contains channels between AgCl particles. According to the literature, these micro-channels are too narrow which are hardly accessible for chloride ions dissolved in the solution but easily accessible to water molecules and the porosity between neighbouring AgCl grains is less than 1% [10].
AgCl layer is through these narrow channels. Because the grain borders mainly appear at the crests while the central area surrounded by borders is usually lower than the borders, the layer looks like a “valley” (Fig. 1). Cross section views also reveal that the pores observed in the top view are not penetrating the AgCl layer but are mostly cavities on the surface of the AgCl layer. Therefore, as mentioned previously, at higher current density the deposited silver chloride is disordered and twisted in comparison with elongated-packed particles, formed at lower current densities. The complex morphology and formation of various grains can be, at least partly, related to the transport processes in the solution adjacent to the deposit surface, where surface diffusion may also be involved. The continuous change of the surface morphology of deposited AgCl was considered as the proof that silver is the charge carrier in silver chloride layer and deposition is at the AgCl|solution interface \[10\].

The cross section of the AgCl layer in regime I and II (0.5 and 1 mA/cm\(^2\)) shows the densely packed particles with the width of approx. 1 to 2 μm and height of more than 5 μm. In regimes III and IV, the piled AgCl particles cannot be considered individually and instead of one AgCl layer two different AgCl layers were detected. Small particles, separated from the twisted AgCl layer with a border line in between were observed in the proximity of the Ag substrate (Fig. 1b).

a) Top view (left) and cross section (right) - 0.5 mA/cm\(^2\)

b) Top view (left) and cross section (right) - 4 mA/cm\(^2\)

Figure 1: SEM top and cross-sectional views of AgCl deposited at different current densities

This two-layer appearance is possibly related to limitations and morphological changes with oxidation and reduction on the Ag surface, when thicker AgCl layer is present. At higher current density regimes, the AgCl particles in the vicinity of the Ag substrate are smaller,
whereas the inter-grain channels are more widened (Fig.2). This observation is in line with reported studies on the morphology of AgCl layers after reduction cycles only \[10\]. The above considerations are elaborated as follows: As the current density increases from 2 mA/cm\(^2\) to 4mA/cm\(^2\), the thickness of this reduced AgCl layer increases from about 5 μm to about 10 μm with the porosity of up to 60% \[10\]. Reduction in AgCl can be attributed to simultaneous effects of thick AgCl deposited over Ag substrate and high current density. Thicker AgCl can hinder silver transportation to the AgCl|solution interface where the new AgCl layer forms. By increasing the charge, the demand for silver at the AgCl|solution interface and the thick AgCl over the Ag substrate cause the AgCl close to the substrate to be reduced to silver. Therefore, it seems that by increasing the AgCl thickness, the AgCl close to the substrate reduces to account for Ag supply for the anodic process at the solution interface. This obviously changes the morphology of the deposited layer while the ionic resistivity increases.

On the other hand, EDS spectra at different points of the cross section of Ag/AgCl electrode anodized at 4mA/cm\(^2\) indicate the formation of AgCl on the silver substrate (Fig. 3).

![Figure 2: SEM photo taken from the AgCl layer close to substrate at regime IV](image)

![Figure 3: EDS results of cross section of Ag/AgCl electrode prepared in regime IV](image)

### 3.2 Tests in aqueous solutions

To study the sensitivity of different Ag/AgCl electrodes to various chloride concentrations and their stability in a short-term measurement, the potential of the electrodes as a function of
time. Potentiometry was performed for 7200 s at a frequency of 1 s\(^{-1}\) in cement extract solution with three different chloride concentrations.

The observations show (Fig. 4) that stable potential was achieved after 100s for the sensors prepared in regime I, whereas 3600 s were needed for sensors, prepared in regime IV. In the continuous measurement for 7200 s, the potential difference among Ag|AgCl electrodes in the solutions with 20 mM (not presented) and 44 mM chloride concentrations varies from about 50 mV at the beginning of the measurement to 7 mV and 4 mV respectively, after 2 hours. In contrast, for the 260 mM chloride solution, the potential difference among different sensors was less than 10 mV at the beginning and reached to 1 mV after 2 hours. Therefore, sensors stabilize faster as the chloride concentration increases from 44 mM to 260 mM. In other words, the sensors are more stable at higher chloride concentration which can be related to the morphology, microstructure and thickness of the AgCl layer over the silver substrate. The thicker and more complex the AgCl layer is, the longer is the time, needed for the Ag|AgCl electrodes to respond. Consequently, the AgCl morphology and microstructure affect the potential response of the sensors.

<table>
<thead>
<tr>
<th>b) 44mM chloride concentration</th>
<th>c) 260mM chloride concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Graph b) 44mM chloride concentration" /></td>
<td><img src="image" alt="Graph c) 260mM chloride concentration" /></td>
</tr>
</tbody>
</table>

Figure 4: Potential stability of the electrodes in cement extract solution

### 3.3 Calibration of the chloride sensor

The principle of linear relationship between the electrode potential of the Ag|AgCl electrode and the logarithm of Cl\(^-\) activity in solution is fundamentally following the Nernst law and equation; at a given temperature can be briefly described by the following equations (1):

\[
E = E_\text{Ag/AgCl}^0 + \frac{RT}{nF} \log [\text{Cl}^-] = -0.0214 - 0.05916 \log (a_{\text{Cl}^-}) \quad (\text{V vs SCE, } 25^\circ\text{C})
\]

The above equation shows the linear relationship between the electrode potential and the logarithm of the Cl\(^-\) activity in solutions. The differences between chloride activity and concentration were taken into account by using mean activity coefficients for sodium chloride \([11]\). By measuring the potential of Ag|AgCl electrode, the Cl\(^-\) ion activity in the solution can be calculated. The potentiometric responses of the sensor to chloride ion activities in different solutions are presented in Fig. 5 together with the values reported by other researchers. The minimum amount of chloride in cement extract solution is 11 mM which was measured by ion chromatography.
As can be seen in Fig. 5 with increasing the chloride concentration in different solutions, the potentials converge. Therefore, the effect of different solutions on the response of the sensor decreases as the chloride concentration increases. In this study the difference between the maximum and minimum potentials in different solutions is 16 mV at 16 mM chloride concentration decreasing to 2 mV in 1000 mM chloride solutions. Therefore, it is evident that the higher is the chloride concentration, the lower is the sensitivity and the higher is the accuracy of sensors’ response in different solutions.

According to the literature for the studied media [3], and in the pH range from 11.9 to 13.7, chloride concentrations below 2*10^-4 to 7*10^-3 molal cannot be accurately determined by potentiometric measurements, due to the OH^- interference. It was also shown [5] that in solutions with pH~13.7 the limit of chloride ion detection would be around 10 mM. Based on these results and considerations, curve fitting was performed for the response in solutions with chloride concentrations higher than 8 mM according to equation (2):

$$E (mV \text{ vs } SCE) = m_0 + m_1 \cdot \lg a_{Cl^-}$$  \hspace{1cm} (2)

The results of the linear regression performed for the three above mentioned solutions are summarized in Table 2. A good linear relation is found for the solutions in a range of chloride concentration from 8 mM to 1000 mM. The slope of the calibration curves varies from 49 to 58 mV/dec with an average of 54 mV.

Table 2. Slope of the calibration curve and results of the statistical analysis of the calibration of the sensor in simulated pore solutions, cement extract solution and distilled water.

<table>
<thead>
<tr>
<th>Solution</th>
<th>Slope (m1)</th>
<th>Axis (m0)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>cement extract solution</td>
<td>-49.73</td>
<td>-12.48</td>
<td>0.998</td>
</tr>
<tr>
<td>simulated pore solution</td>
<td>-58.11</td>
<td>-13.31</td>
<td>0.999</td>
</tr>
<tr>
<td>demi water</td>
<td>-55.39</td>
<td>-15.69</td>
<td>0.999</td>
</tr>
</tbody>
</table>
4 CONCLUSION

The following conclusions can be drawn from the present investigation:

- The AgCl layer microstructural properties vary and depend on the current densities, applied within the anodization regimes. Higher current densities result in thicker layers (as expected) but also increase complexity (e.g. more than one interface was observed).
- The thicker and more complex is the AgCl layer, more time is needed for the Ag/AgCl electrode to assume a stable potential response.
- The microstructural properties of the AgCl layer (the sensor respectively) affects the sensitivity especially at low chloride concentrations. The thicker and more complex is the AgCl layer; more time is needed for the electrode to respond.
- The chloride sensor prepared in 0.5 mA/cm\(^2\) regime showed excellent stability and sensitivity. The response follows the Nernst law with good linearity in the range from 8 to 1000 mM chloride concentration.
- At 1000 mM chloride concentration the effect of different solutions on the response of the sensor is negligible.

REFERENCES

MODIFICATION OF CELLULOSE-CEMENT COMPOSITES BY ADMIXTURE OF SUGAR CANE BAGASSE ASH

RODRIGUES, Conrado de Souza(1), ZUKOWSKI, Bartosz(2), FIGUEIREDO, Stefan Chaves(3) and SANTOS, Luciana Alvarenga(4)

(1) Departamento de Engenharia Civil (DEC), Centro Federal de Educação Tecnológica de Minas Gerais (CEFET-MG), Av Amazonas 7675, Belo Horizonte-MG, Brazil, 30510000, crodrigues@civil.cefetmg.

(2) Departamento de Engenharia Civil, COPPE/UFRJ, Av. Horácio Macedo 2030, Centro de Tecnologia, Bloco I-2000, Rio de Janeiro–RJ, 21941972, bzukowski87@gmail.com.

(3) DEC/CEFET-MG, Av Amazonas 7675, Belo Horizonte-MG, Brazil, 30510000, stefanccf@gmail.com.

(4) Departamento de Engenharia Civil, Centro Federal de Educação Tecnológica de Minas Gerais, Av. dos Imigrantes 1000, Varginha-MG, Brasil, 37022560, lucianasantos@varginha.cefetmg.br.

Abstract
The growing demand for cement products, as well as the idea of sustainable development supports the research of alternative materials that could be a partial substitute for cement. One of potential materials is fly ash and many other sources of pozzolanic materials, which application in cement-based materials have been well documented. This work presents the influence of sugar cane bagasse ash (SCBA) on the properties of cellulose-cement materials. Two types of SCBA, obtained by different processing, were investigated Bending strength tests and tests of physical properties were carried out on composites samples with two types of sugarcane bagasse ash. On the basis of the tests was claimed that the addition of sugar cane bagasse ash reduces the density of the composite, as well as increases porosity and water absorption. The increase of bending strength of composites was obtained after accelerated aging cycles. Obtained results show the possibility of application the sugar cane bagasse ash in the cellulose-cement composites according to the idea of sustainable development.

Keywords: sustainable development, cellulose-cement composite, eucalyptus fibers, sugar cane bagasse ash

1 INTRODUCTION

Cement and its composites are one of the most common materials in civil engineering. It is not a surprise that the last years were devoted to development of cement industry and research programs that can increase the properties of cement-based materials. On the other hand,
cement production is one of the main sources of industrial CO$_2$ and all environmental impacts related to it. That is a reason to procure the possibility of cement replacement to provide a material with better properties, lower cost and more friendly to the environment [1].

The replacement of asbestos fibres in cement composites brought many challenges, as asbestos fibers are strong, durable and compatible to the cement matrix. As a result, asbestos-cement composites present high strength, toughness and stability for long term use. Attempts to replace asbestos reinforcement by other fibres have shown that the simple replacement does not provide fibrocements with properties with properties fully compatible to those of the asbestos-cement.

When cellulose fibres are used as alternative reinforcement, durability is one of the issues not fully resolved. Due to a range of deterioration mechanisms, cellulose-reinforced fibrocements loose the strength and toughness obtained at short term. Changing the matrix composition by using highly reactive mineral admixtures is a common approach to enhance the long term performance of cellulose-cement composites. Reactive mineral admixtures have been successfully obtained from agricultural wastes processed as biomass for energy co-generation. This is the case of ashes resulted from the burning of rice husks and sugar cane bagasse. Depending on the burning conditions, the ash can present significant contents of amorphous silica, which can be reactive with cement. The pozzolanic reaction (between SiO$_2$ from the ashes and Ca(OH)$_2$ from hydrated cement) improves the structure of the matrix and the interfacial transition zone providing increased strength and stability of the cellulose-cement composites.

Besides the improvements in the composites’ behaviour, the use of mineral admixtures partially replacing cement has also a significant effect in decreasing the environmental impacts associated to these fibrocements, once less cement is used.

This work describes the influence of bagasse ash admixture on the properties of cellulose-cement composites. Because the sugar cane crops can be fertilized with potassium-based fertilizers its residue can be present in ash. This is why the influence of two types of ash (without and with chemical treatment to remove K$_2$O) was analyzed. The tests were carried at Federal Centre for Tech. Education of Minas Gerais (CEFET-MG) in Belo Horizonte (Brazil) and involved composites’ production and characterization (physical and mechanical parameters) before and after accelerated weathering in environmental chamber.

2 METHODOLOGY

2.1 Materials and mix proportions

The SCBA was obtained from sugar cane mill that process an average of one million tons of sugar cane per year for sugar and ethanol production. The bagasse is the main waste of the process and is used for energy co-generation. As the details on the burning process during the co-generation were not provided, this study was conducted using the bagasse collected in the mill and burned in laboratory scale under controlled condition. The referred SCBA-1 is sugar cane bagasse ash resulted by burning the bagasse for 3 hours at 650°C. The ash was grinded in a ceramic ball mill for 24h.

After burned, SCBA-1 presented 3.4% K$_2$O, which is above the limit for mineral admixtures to be incorporated in cement-based materials. Therefore, an ungrinded portion of SCBA-1 it was treated by acid attack (HCl) to reduce K$_2$O content (a process detailed in [8]).
Treated SCBA-1 was burned again at 650°C for 3 hours and grinded for 24 in a ceramic ball mill, resulting in SCBA-2. Figure 1 shows samples of the two SCBA used in this study.

Besides SCBA, the composites’ matrix was composed also by portland cement and carbonate filler. The portland used present low admixtures content (Brazilian CP-V), being compatible to American OPC type I or European CEM I. The carbonate filler was obtained by grinding limestone particles in a steel ball mill for 24 hours. The composition and physical parameters of all matrix raw materials are shown in Table 1.

Cellulose fibres obtained from Eucalyptus by the kraft pulping process were used as reinforcement in all composites.

Table 1 — Raw materials chemical composition and physical parameters

<table>
<thead>
<tr>
<th>(%)</th>
<th>SCBA-1</th>
<th>SCBA-2</th>
<th>OPC</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>40.08</td>
<td>37.46</td>
<td>21.92</td>
</tr>
<tr>
<td>TiO₂</td>
<td>2.21</td>
<td>2.61</td>
<td>0.18</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>31.46</td>
<td>35.06</td>
<td>5.15</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>12.91</td>
<td>15.36</td>
<td>2.05</td>
</tr>
<tr>
<td>CaO</td>
<td>4.45</td>
<td>1.25</td>
<td>64.42</td>
</tr>
<tr>
<td>MgO</td>
<td>1.05</td>
<td>0.66</td>
<td>0.85</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.11</td>
<td>0.01</td>
<td>-</td>
</tr>
<tr>
<td>K₂O</td>
<td>3.40</td>
<td>1.42</td>
<td>0.89</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.72</td>
<td>0.47</td>
<td>0.30</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.51</td>
<td>0.30</td>
<td>-</td>
</tr>
<tr>
<td>Cl</td>
<td>0.01</td>
<td>0.62</td>
<td>-</td>
</tr>
<tr>
<td>C (LOI)</td>
<td>2.03</td>
<td>1.21</td>
<td>4.21</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Physical parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>S&lt;sub&gt;BET&lt;/sub&gt; (m²/g)</td>
</tr>
<tr>
<td>Density (g/cm³)</td>
</tr>
<tr>
<td>Average particle size (µm)</td>
</tr>
</tbody>
</table>
2.2 Composites’ formulation and samples preparation

As the focus of this research was on the effects of SCBA incorporation, it was considered a reference composite based on previous studies [2, 6]. This was produced with a matrix composed by 50% Portland cement and 50% carbonate filler (weight basis) and a fixed reinforcement rate of 5% (volume basis). The SCBA was incorporated as a partial replacement of both cement and filler. After previous research on rice husk ash in fibrocements [6] a 30% total SCBA incorporation was applied, replacing 15% of cement and 15% of the carbonate filler, as in Table 2.

<table>
<thead>
<tr>
<th>Fiber [%]</th>
<th>Cement [%]</th>
<th>Filler [%]</th>
<th>SCBA [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>REF 5.0</td>
<td>50.0</td>
<td>50.0</td>
<td>0.0</td>
</tr>
<tr>
<td>SCBA1 5.0</td>
<td>35.0</td>
<td>35.0</td>
<td>30.0</td>
</tr>
<tr>
<td>SCBA2 5.0</td>
<td>35.0</td>
<td>35.0</td>
<td>30.0</td>
</tr>
</tbody>
</table>

There were made 3 sets of samples. Two sets were made with admixture of bagasse sugarcane ash, cement, neutral filler and eucalyptus fiber and one reference set. Each set contains 14 plates (160x160x6.5mm) which were cut for smaller samples (160x40x6.5mm) and then used for tests.

The composites were made according to Hatschek process adapted to the laboratory scale. The composition of samples was based on previous research on the influence of fly ash admixture on cement cellulose composites [2]. The tests were according to methods presented in RILEM [3].

Fiber from the paper industry can contain a significant humidity, to ensure the proper amount of fiber in the sample (5% by volume) humidity was checked before samples production. Part of the fiber was dried by 6 hours at 100 ºC and the humidity was calculated to 7% and the total mass of fiber equal 13,42g for plate.

The plates were produced by slurry mix-vacuum dewatering-compaction method. It starts with the fibers being mixed with water for 3 minutes at a 1000 rotation/min to reduce the possibility of flocculation. Then, the cement, filler and ash were add and continuously mixed for the next 3 minutes at 2100 rotations/min. The resulted mix presented about 20% of dry solids, by mass. There was used a form connected to container and the pump to remove the water from samples by under pressure. The air from container was removed by pump. Next the slurry was placed in form and manually compacted to remove part of water. The rest of the water was removed by under pressure. Then plates were compacted in mechanical press by 5 minutes with the pressure raise up to 3.2 MPa and for the next 5 minutes kept under this pressure. Plates were cured by 21 days in plastic bag and separated by the wet paper tissue to keep humidity.

After 21 days plates were taken out of plastic bag and cut into smaller samples (40x160mm) which were used for the tests. Samples were placed in the environmental chamber for accelerated aging cycles: 3 hour under water in 25 ºC to fully saturation, next 18 hours of drying at 60 ºC and relative humidity of 0% to completely dry state and 3 hours at 25 ºC and relative humidity of 0%. After 0, 5 and 25 cycles 12 samples of each set were taken out and for the next 48 hours prepared to tests in dry and saturated state.
2.3 Testing methods

For the mechanical tests, the squared plates were sawed in three samples of 40mmX160mm each, which were used for the three point bending tests. The samples were tested under two moisture conditions: in equilibrium (stabilization at 50% R.H. and 23°C for five days before the test) and saturated (samples soaked in water 24h before the test). The loading were applied at a constant 0.5mm/min and the mechanical parameters were obtained according to RILEM [3]. Physical properties like density, water absorption and porosity were obtained by ordinary Archimedes approach (weighting dry and saturated samples).

3 RESULTS AND DISCUSSION

Figures 2, 3 and 4 show the effects of 25 ageing cycles on reference, SCBA-1 and SCBA-2 composites, respectively. REF samples had their bending strength significantly decreased after 25 cycles; the same aspect occur with the strain energy absorption capacity (figure 2). Therefore, the varying temperature and moisture along with the weathering cycles provided the changes in materials properties compatible to those observed in fibrocements under service conditions.

![Figure 2: Bending strength and strain energy absorption of REF](image1)

![Figure 3: Bending strength and strain energy absorption of SCBA-1](image2)
When SCBA-1 was used partially replacing cement and the carbonate filler, the bending strength decreased in non-aged composites (0 cycles), Figure 3. Comparing Tables 3 and 4 it is possible to note that the incorporation of SCBA increased the composite porosity, which also can contribute to the decreased strength. However, during the cycles there is an increase in bending strength and the drop in specific energy observed in REF composites is not present in SCBA-1 composites. The increase in strength is probably due to the thermal activation of the pozzolanic effect of SCBA-1, creating novel hydration products and contributing to composites’ strength. Moreover, the increased strength was not followed by a decrease in composites’ porosity, which remained constant in SCBA-1 samples.

The effects of SCBA-2 incorporation (Figure 4), partially replacing cement and carbonate filler, resulted in a similar effect to that observed in SCBA-1 samples. The more noticeable difference is that SCBA-2 composites do not present a much lower strength at 0 cycles, comparing to REF samples. So the use of 30% SCBA-2 resulted in composites with mechanical properties compatible to REF composites at short term, but with enhanced mechanical behaviour after the ageing cycles (higher strength and toughness). Also, SCBA-2 composites present higher porosity than REF samples.

The data on Tables 3 to 5 show that SCBA-1 and SCBA-2 have similar effects when incorporated in cement composites. Comparing to REF, SCBA composites have higher porosity and water absorption, with no noticeable differences between SCBA-1 and SCBA-2 in this case. Unfortunately, it was not possible to determine how the pore structures are different for the three materials, which would help to better establish why a higher strength was achieved in SCBA composites even at a higher overall porosity.

Table 3 — Physical properties of reference samples (REF)

<table>
<thead>
<tr>
<th>Cycles</th>
<th>Density [g/cm³]</th>
<th>Absorption [%]</th>
<th>Porosity [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.70 ± 0.05</td>
<td>17.88 ± 1.77</td>
<td>30.26 ± 2.46</td>
</tr>
<tr>
<td>5</td>
<td>1.75 ± 0.04</td>
<td>15.59 ± 1.04</td>
<td>27.24 ± 1.20</td>
</tr>
<tr>
<td>25</td>
<td>1.71 ± 0.05</td>
<td>16.09 ± 0.88</td>
<td>27.51 ± 0.87</td>
</tr>
</tbody>
</table>
4 CONCLUSION

In this study, it was investigated the effects of two different SCBA on the mechanical and physical properties of cellulose-cement composites. Although SCBA presented lower silica content than other agricultural residues (especially rice husk ash) it was possible to observe improvements in the composites’ behaviour due to their incorporation. The strength properties of composites with SCBA-1 and SCBA-2 indicate that both ashes presented pozzolanic activity with cement, especially when comparing the mechanical properties along with the ageing cycles. In this case, SCBA-1 resulted in composites with lower strength than REF composites, but while the strength of REF samples decreased with the ageing cycles, SCBA-1 composites increased their bending strength after 25 cycles. It seems that the reactivity between SCBA-1 and cement was activated by the higher temperatures used in the cycles. On the other hand, SCBA-2 presented bending strength compatible to that of REF composites, even before the accelerated ageing; the values increasing slightly during the cycles. Besides that, both composites incorporating SCBA presented higher porosity and water absorption than REF samples. So, it seems that the mineral admixtures does not function as a filler, although the pozzolanic activity seemed to overcome the deleterious effect of higher porosity on strength.

ACKNOWLEDGEMENTS

Authors would like to thank CAPES, FAPEMIG, CNPq and CEFET-MG for the financial support.

REFERENCES


ACCELERATED AGEING AND DURABILITY OF DOUBLE-GLAZED SEALED INSULATING WINDOW PANES

Silje Kathrin Asphaug (1), Bjørn Petter Jelle (1,2), Lars Gullbrekken (1) and Sivert Uvsløkk (1)

(1) SINTEF Building and Infrastructure, Department of Materials and Structures, NO-7465 Trondheim, Norway – silje.asphaug@sintef.no; bjorn.petter.jelle@sintef.no; lars.gullbrekken@sintef.no; sivert.uvslokk@sintef.no

(2) Norwegian University of Science and Technology (NTNU), Department of Civil and Transport Engineering, NO-7491 Trondheim, Norway – bjorn.petter.jelle@ntnu.no

Abstract

A crucial property for double-glazed sealed insulating window panes is to maintain their thermal insulating properties and thus low U-values by keeping their low-conductance noble gas concentrations within the sealed glazing units, i.e. not admitting air into the sealed window panes. However, degradation and thus subsequent reduction or loss of low-conductance gas concentration may occur in the sealed glazing units by their exposure to outdoor climate during many years, e.g. by being exposed to solar radiation, wind, moisture, wind-driven rain, elevated temperatures and freezing/thawing cycles.

The choice of spacers in the sealed window panes is important with respect to be able to keep as low thermal transport through the window panes as possible, i.e. low U-value. In addition, the type of spacers may also influence their durability and resistance towards ageing, which hence may be characterized by the low-conductance noble gas concentration, e.g. argon, krypton or xenon, versus elapsed time in a long-term perspective.

Thus, several double-glazed sealed insulating window panes, with aluminium spacers and super spacers, have been subjected to accelerated ageing by climate ageing and elevated temperature ageing. The durability and ageing of the sealed window panes have been studied and characterized by their spacer type and gas concentration as function of accelerated ageing time.

1 INTRODUCTION

Energy-efficient insulating glass (IG) units are increasingly used in buildings although there is limited knowledge in the field of durability. The durability is critical to window energy performance. Seal failure leads to the loss of insulating gas, e.g. argon, krypton and xenon, and hence reduces the U-value of the glass unit. Windows account for about 30 to
50% of thermal transmission losses through building envelopes, and knowledge about durability is therefore highly important in terms of energy performance in buildings. To the authors knowledge there are few studies in the literature investigating ageing of insulating glass units with different spacers and the gas concentration reductions impact on the energy performance.

Several studies address various aspects with window glazing, e.g. fenestration technologies of both today and tomorrow [1], solar radiation glazing factors including also electrochromic windows [2], self-cleaning effect for glazing products [3, 4], miscellaneous energy aspects of windows and window frames [5, 6], and a state-of-the-art review and future perspectives on window spacers and edge seals in insulating glass units [7]. In general, investigating the durability of building materials and components, also newly developed ones, e.g. by carrying out accelerated climate ageing in the laboratory, is of major importance [8, 9]. Thus, performing a robustness assessment of these materials and components may also be found to be beneficial [10].

More specifically on insulating glass units, Christensen [11] has conducted an analysis of gas-filled units on the Danish marked. The gas concentration of a range of units from different producers was tested. Gas concentrations from 66 to 94% were measured. The life expectancy of four types of gas-filled units (Ar + SF6) was also tested. One selection was exposed for 1 week and one selection was exposed for 5 weeks of accelerated ageing according to DS 1094.4 [12]. One selection was aged by heating at 70°C for 60 days. Analyses showed no difference in gas concentration for the selection exposed to accelerated aging according to DS 1094.4, but the selection aged by heating had an average diffusion loss of 4%.

Measurements and literature described by Olsson-Jonsson [13] showed that gas-filled units are generally gastight with little gas leakage and that double-sealed units are more gastight than single-sealed units. It was also found that the amount of gas-fill in newly manufactured units vary widely, e.g. about 90% gas-fill in most cases but very low in some cases. Polysulphide was found to be the most gastight sealant, and silicone sealant had the poorest performance.

Insulating glass units are exposed to a variety of environmental factors, such as wind loads, working loads, temperature and atmospheric pressure fluctuations, solar radiation, water and water vapour. Insulating glass units, including their life expectancy, have been studied by Wolf [14, 15] and Wolf and Waters [16]. Among the findings it was stated that the resistance of the edge seal to water vapour diffusion was determined almost exclusively by the low water vapour permeability of the polyisobutene (PIB) primary seal. The water vapour permeability of the secondary insulating glass sealant played only a subordinate role. However, the viscoelastic properties of the secondary insulating glass sealants, particularly the tensile stress and elastic recovery, was of great importance as these properties affected the primary seals' ability to function.

Correlation between field studies and laboratory tests of the performance of insulating glass units in buildings has been investigated by Lingnell and Spetz in a twenty-five year study [17]. The insulating glass units in the field studies were located in different cities and climates in USA. Furthermore, several other studies have investigated mechanical stress and stress due to temperature differences [18-20].

In this work double-glazed sealed insulating window panes filled with argon gas, with both super spacers and aluminium spacers, have been subjected to accelerated weathering over
extended time periods to determine whether the window pane units can withstand high
temperatures, ultraviolet (UV) radiation, water spray and freezing/thawing cycles without
failure. One aspect to be investigated is also whether the new super spacers are as good as the
traditional aluminium spacers in terms of maintaining the gas concentration. Hence, the
objective of this study is to investigate ageing of sealed window panes with different spacers
and the impact of gas concentration decrease on the windows thermal performance.

2 EXPERIMENTAL

2.1 Sample materials
This study includes 18 insulating double glass window pane units filled with argon gas, 9
with super spacers and 9 with aluminium spacers as depicted and described in Figure 1 and
Table 1. All the windows are produced by Friva AS.

![Figure 1: Window pane (left) with super spacer (middle) and aluminium spacer (right).](image)

Table 1: Properties of the two window pane types.

<table>
<thead>
<tr>
<th></th>
<th>Super spacer</th>
<th>Aluminium spacer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Producer</td>
<td>Friva AS</td>
<td>Friva AS</td>
</tr>
<tr>
<td>Name</td>
<td>Isoler 4-16 Arg-4ES3</td>
<td>Isoler 4-16 Alu Arg-4ES3</td>
</tr>
<tr>
<td>Size (mm)</td>
<td>350x500</td>
<td>350x500</td>
</tr>
<tr>
<td>Thickness window pane (mm)</td>
<td>24.2</td>
<td>25</td>
</tr>
<tr>
<td>Glass thickness (mm)</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Energy coating</td>
<td>S3</td>
<td>S3</td>
</tr>
</tbody>
</table>

2.2 Ageing methods
The glass units are divided into three groups, see Table 2. Each group consists of 3 window
panes with super spacer and three with aluminium spacer.
Table 2: Test groups, each group consists of 6 window panes, 3 window panes with super spacer and 3 with aluminium spacer.

<table>
<thead>
<tr>
<th>Group</th>
<th>Ageing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Vertical climate simulator</td>
</tr>
<tr>
<td>2</td>
<td>Two weeks in vertical climate simulator than moved to the heating chamber (60°C)</td>
</tr>
<tr>
<td>3</td>
<td>Heating chamber (60°C)</td>
</tr>
</tbody>
</table>

The insulating window pane samples in Group 1 have been aged in a vertical climate simulator (Figure 2) according to NT Build 495 [21] for up to 365 days (365 x 24 h). In this test apparatus the samples are subjected in turns to four different climate zones, that is, an ultraviolet (UV) and infrared (IR) irradiation zone (black panel temperature of 63°C), a water spray zone (15 dm³/(m²h)), a freezing zone (-20°C) and an ambient laboratory climate zone. The UVA and UVB intensities are averaged to respectively 30 W/m² and 3.4 W/m² for the investigations reported within this work. The exposure time is 1 hour in each climate zone in the above given sequence. For further details it is referred to the test method, i.e. NT Build 495 [21].

A heating chamber (Termaks) (Figure 2) has been employed in order to subject the insulating window pane samples in Group 3 to a constant air temperature of 60°C for up to 365 days (365 x 24 h). Note that this temperature of 60°C is at the lower (possible safe) end of the normally accepted high-end safe temperature range between 60 to 70°C for accelerated ageing of polymers (e.g. gaskets and sealants). In order to test the actual samples for as high safe temperature as possible, and to accelerate the temperature degradation as much as possible (Arrhenius equation), as high safe temperature as possible is chosen, while ensuring that the temperature is not as high (unsafe), that it will induce any degradation reactions that will not occur during natural ageing.

The window pane samples in Group 2 have been aged for two weeks in the vertical climate simulator and then moved to the heating chamber.

Figure 2: Accelerated ageing of window panes in vertical climate simulator (left) and heating chamber (right).

2.3 Measurements

The window panes were measured according to the schedule in Table 3. Each measured value is an average of 5 measurements. Group 2 was moved from the climate simulator to the
heating chamber after 2 weeks. Due to potential damages caused by the movement, Group 2 was measured 2 times in addition, one day after the movement and 4 weeks after.

Table 3: Interval of measurements.

<table>
<thead>
<tr>
<th>Weeks:</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>2+1 day</th>
<th>3</th>
<th>4</th>
<th>6</th>
<th>8</th>
<th>8 to 52</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date/</td>
<td>28.01</td>
<td>04.02</td>
<td>11.02</td>
<td>12.02</td>
<td>18.02</td>
<td>25.02</td>
<td>11.03</td>
<td>25.02</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>V</td>
<td>V</td>
<td>V</td>
<td>Not measured</td>
<td>V</td>
<td>V</td>
<td>Not measured</td>
<td>V</td>
<td>Every 4th week</td>
</tr>
<tr>
<td>2</td>
<td>V</td>
<td>V</td>
<td>V</td>
<td>Moved</td>
<td>V</td>
<td>V</td>
<td>V</td>
<td>V</td>
<td>Every 4th week</td>
</tr>
<tr>
<td>3</td>
<td>V</td>
<td>V</td>
<td>V</td>
<td>Not measured</td>
<td>V</td>
<td>V</td>
<td>Not measured</td>
<td>V</td>
<td>Every 4th week</td>
</tr>
</tbody>
</table>

To determine the gas concentration in the window panes, a device called Gasglass 1002, Sparklike was used (Figure 3). This instrument can only be used on units filled with argon gas. It works by creating a high-voltage spark inside a sealed glazing unit, and then analyzing the resulting light spectrum to produce a direct display of the percentage of argon in the gas filled unit. The method is non-destructive and very suitable for measurements during an ageing procedure.

Figure 3: The argon gas concentration is measured using Gasglass 1002 Sparklike.

The temperature of the window panes must be between 15 and 35°C in order for the measurements to be valid. The heating chamber was therefore turned off and the door left open for at least two hours before the window panes where measured. The window panes were taken out of the chamber, placed horizontally on a table for gas concentration measurements, then placed back in the chamber. The window panes in the climate simulator were measured as located in the wall construction.

The accuracy of the Gasglass for a 4-12-4Low-E IG unit is stated to be within the following specifications; 100 % - 90 % ± 1 %, 90 % - 85 % ± 1.5 % and 85 % - 80 % ± 2 %. Between 80 % and 0 % the accuracy is expected to be within ± 5 % but not guaranteed. Lowest measurable concentration is approximately 50 %.
3 RESULTS AND DISCUSSION

The window panes have been subjected to accelerated ageing for 350 days (50 weeks). Figure 4 shows the reduction in argon gas concentration during the 50 weeks of exposure for all of the 18 window panes, depicting both super spacers and aluminium spacers. Figure 5 shows the same results as Figure 4, but to make the results more clear the values are shifted so that all window panes start from the same value of gas concentration, i.e. 92%. Figure 5 clearly shows how the results separate in three directions. At the top there are a group of windows that show little to no gas leakage. Among them are both aluminium spacers and super spacers, all from ageing in the climate simulator (Group 1). Below the results separate in two groups, all of which are from ageing in the heating chamber (Group 2 and 3). Of these the upper graphs are window panes with super spacer and the lower graphs are window panes with aluminium spacer. The aluminium spacer shows more gas leakage than the super spacer.

Figure 4: Gas concentration versus time of the window panes subjected to accelerated ageing

Figure 5: Gas concentration versus time of the window panes subjected to accelerated ageing. The values in figure 4 are shifted so that all window panes start from the same value of gas concentration, i.e. 92%.
Figure 6 shows how the U-value increases with decreased gas concentration. The decrease in gas concentration may be directly correlated to the U-value of the windows which hence will lead to a larger thermal loss through the building envelope. Such calculations will be carried out in future work for different building and climates.

![Figure 6: Increasing U-value with decreasing gas concentration, double (upper graph) and triple layer glazing (lower graph) with low emissivity coating.](image)

Without further investigating the structure of the spacers and the materials which are used in the primary and secondary edge seal, it may be hard to conclude what is causing the gas to leak out from the window panes. Opposed to the heating chamber, the climate simulator only subjects the window panes to high temperatures every fourth hour. Because there is no reduction in gas concentration for Group 1 (climate simulator) but significant reduction in Group 2 and 3 (heating chamber), an explanation might be that the diffusion resistance of the sealant in the spacers decreases due to the increased temperature. This may be part of an explanation to why the window panes with aluminium spacers experience a larger loss of argon gas than the ones with super spacer. The area of the sealant between the spacer and the glass, in which the gas may diffuse through is larger in an aluminium spacer than in a super spacer. Another explanation may be, when aged at high temperatures, the sealant degrades and hence reduces the diffusion resistance causing the gas to diffuse more rapidly. This would however cause the graphs in Figure 5 to increase more rapidly during time, and not linearly as depicted so far.

4 CONCLUSIONS

The argon gas concentration in insulating glass units has been measured over time in which they are subjected to different types of accelerated ageing. The decrease in gas concentration may be directly correlated to an increase in U-value for the windows which hence will lead to a large thermal loss through the building envelope.

REFERENCES

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Abstract
This research evaluated the physical properties that influence in the durability of cementitious materials, such as tortuosity and sorptivity. The tortuosity is an important parameter that defines the permeability and the configuration of the flow channels through which can flow a fluid.

Due to the technical difficulties of the determination of the tortuosity, the electrical measurements are proposed and they were performed in mortars after 28 days of age. The mortars were contaminated with heavy metals, nitrates of Cr$^{3+}$, Pb$^{2+}$, and Zn$^{2+}$ in order to simulate cementitious materials produced with contaminated wastes. After that, a test of capillary absorption was carried out in these contaminated mortars. Thus, also was evaluated the influence of heavy metals in the physical properties of cementitious materials.

The results showed that the contaminants influence differently in the capacity of water capillary absorption (sorptivity) and in the configuration of the pores (tortuosity). Chromium decreased and zinc increased the sorptivity of the mortars. These data of sorptivity reflected in the values of tortuosity that were less with the addition of the heavy metals in the mortars. Therefore, the heavy metals may alter the durability of cementitious materials.

1 INTRODUCTION
The water capillary absorption is a method utilized by others researches for evaluate the durability and performance the mortars [1]. This test reflect physical properties how the porosity, the tortuosity and permeability. Thus, the knowledge of these properties is powered tool to assess the durability of the building materials. The tortuosity is viewed as a property of the porous medium that can be measured from the 3D micro-geometry of the pore structure [2]. In other hand, news studies were carried out about the determination of the tortuosity through electrical methods [3]. Also, the tortuosity is an important parameter in the transport...
proprieties and may difficult the leaching of the heavy metals and its measurement is important for optimizing the materials when waste materials are incorporated on the cementitious matrices [4]. The main interest of the present paper is to assess the electrical impedance of the mortars contaminated with heavy metals (chromium, lead and zinc) and related to these durability properties as the tortuosity, porosity and water absorption.

2 MATERIALS AND METHODS

2.1 Materials
Two mortar samples were prepared with cement Portland CPV – ARI, the water to cement ratio was 0.50 (W/C = 0.50) and the cement to sand ratio was 1:2 (by weight). The heavy metals were added in the mortars through of nitrates (Cr (NO$_3$)$_3$.9H$_2$O; Pb (NO$_3$)$_2$; Zn (NO$_3$)$_2$.6H$_2$O). The contamination concentrations were 20, 40, 80 and 160 ppm of heavy metal by the weight of Portland cement.

2.2 Methods
In the measurements of impedance spectroscopy it was used a frequency range of 200 kHz to 2 MHz and the test was carried out in the mortars with 28 days of age and after water capillary absorption. The results of impedance spectroscopy are Nyquist plots that show the imaginary part of electrical impedance (electrical reactance) and the real part (electrical resistance). When the electrical reactance is zero and the value of the electrical resistance is maximal, this electrical resistance is named “bulk resistance” that refers to total electrical resistance of the mortars.

The water absorption was carried out in the mortars with 28 days of age. Through this procedure, the sorptivity (S) is determined. The sorptivity is the slope of straight of plot $t^{1/2} \times i$, where $t$ is the time of water absorption and $i$ is the water content absorbed by section surface of the samples (cm$^3$/cm²). After water absorption test, the impedance spectroscopy was carried out in the mortars saturated. The mortars saturation was ensured with a greater contact time between samples and water, after the end of absorption test. This second measurement was used to find the tortuosity ($\tau$) of mortars, given by Equation 1:

$$\tau = F \Phi$$  \hspace{1cm} (1)

$F$ is a formation factor, the relationship between electrical resistivity of mortar ($\rho_{mortar}$) and electrical resistivity of pore water ($\rho_{porewater}$); and $\Phi$ is the porosity of mortars. The porosity was determined by Brazilian standard NBR 9778 (2005). The formation factor was determined by relation between the electrical resistivity of mortars with 28 days of age and the measurement of electrical resistivity after water saturation. This relationship refers to presence of the pore water after saturation and the Equation 1 became modified as showed below (Equation 2):

$$\tau = \frac{\rho_{28days}}{\rho_{mortarsaturated}} \times \phi$$  \hspace{1cm} (2)
3 RESULTS AND DISCUSSION

3.1 Porosity of mortars

The open porosity of mortars varied between 15 and 20 % (Fig. 1). The porosities of all contaminated mortars decreased in relation to reference mortar (without contamination). In accordance with our results of porosity, apparently, the zinc increased the porosity of mortars or the pores size diameters how was observed by [5]. One explanation of the authors for the decreasing of the porosity is that the hydration products are precipitated on the pores and after of chemical interaction of these compounds with the heavy metals, increases the free space into the pores.

![Figure 1: Porosity of the mortars.](image1)

3.2 Impedance Spectroscopy

The mortars contaminated with Cr\textsuperscript{3+}, in the concentrations of 40 and 80 ppm, showed electrical resistances higher than the electrical resistances of the samples Cr 20 and Cr 160. This is due greater porosity values of the samples Cr 40 e Cr 80. In accordance with [6], the conductivity of the mortars is attributed to the electrolyte filling pores. Then, when the mortars are not saturated, higher porosities results in higher electrical resistances, whereas the air that fills the pores not conduct the electrical current. Figure 2 represents the plot of electrical resistance (Z') and electrical capacitance, a component of electrical reactance (Z'') of the mortars contaminated with chromium at frequency range of 200 kHz to 2MHz.

![Figure 2: Electrical Impedance of mortars contaminated with Cr\textsuperscript{3+}.](image2)

The mortar contaminated with 40 ppm of lead nitrate showed the highest electrical resistance (Fig. 3) due greater porosity (Fig. 1). Also, due to its higher molecular density, the lead is available to adsorb onto C-S-H coatings formed around hydrating cement clinker particles [7]. The interface regions are related to electrical capacitance of the cementitious materials, originated to C–S–H gel phase [8]. As the lead is adsorbed on the surface of the C-S-H particles, this capacitance became reduced. The decrease of the capacitance is showed in
the Fig. 3. Comparing the plot of Fig. 2 with the plot showed in the Figure 3, the chromium affected mainly the electrical resistance ($Z'$) and the lead (as it not react chemically with the cementitious compounds) affect the electrical capacitance ($Z''$).

The zinc contamination decreased the bulk electrical resistance of the mortars at 28 days of age (Fig. 4). The decrease in the electrical resistance, Fig. 4, can be attributed to the formation of new compounds as $\text{CaZn}_2(\text{OH})_6 \cdot 2\text{H}_2\text{O}$ [9], the calcium ferrite and mainly crystals as a complex zinc aluminate [10]. These compounds mentioned promote the conduction of the electrical current and consequently, decreased the electrical resistance of the contaminated mortars.

3.3 Water Capillary Absorption

Water capillary absorption measurements were started at 28. The results are showed in the Fig. 5, Fig. 6 and Fig. 7. Many researches have showed the interaction of chromium with the cementitious matrices ([11], [12], and [13]). This interaction that exists between the $\text{Cr}^{3+}$ and the cement Portland can result on formation of new hydrates cementitious compounds (like $\text{Cr(OH)}_4$, $\text{Cr(H}_2\text{O)}_6^{3+}$) that decrease the porosity. Therefore, the water capillary absorption capacity of the mortars contaminated with chromium also decreases, as it can be showed in the Fig. 5.
Previous researches show that the addition of lead in the cementitious matrices doesn’t change significantly the porosity of these, but, this addition results in the porous of smaller diameter [14]. If the porous mortars have a smaller diameter, the capillary pressure is larger and the water absorption will be higher [15]. This decreasing on the porous diameter possibly caused the higher water absorption of mortars contaminated with lead (Fig. 6).

![Figure 6: Water capillary absorbed by mortars contaminated with Pb$^{2+}$.](image)

It can be seen in the Figure 7 that the mortars contaminated with zinc have higher water absorption. The zinc addition caused an increase in the pore volume in relation to mortar without the addition as can be seen in the paper published by [16]. This increase of the porosity promoted the increase on the water absorption as can be seen in the Fig. 7.

![Figure 7: Water capillary absorbed by mortars contaminated with Zn$^{2+}$.](image)

Through of water capillary absorption test, it was determined the sorptivity of the mortars contaminated with heavy metals (Table 1). The sorptivity is related to water absorption capacity of the mortars during the time. In general, we can say that chromium decreased the sorptivity of the contaminated mortars and the heavy metals lead and zinc increased the sorptivity.

Table 1: Sorptivity ($S$) measured in (g.cm$^{-2}$.s$^{1/2}$) of the mortars.

<table>
<thead>
<tr>
<th>Samples</th>
<th>$S$</th>
<th>Samples</th>
<th>$S$</th>
<th>Samples</th>
<th>$S$</th>
<th>Samples</th>
<th>$S$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ref.</td>
<td>0.0015</td>
<td>Cr 20</td>
<td>0.0012</td>
<td>Pb 20</td>
<td>0.0017</td>
<td>Zn 20</td>
<td>0.0017</td>
</tr>
<tr>
<td>Cr 40</td>
<td>0.0009</td>
<td>Pb 40</td>
<td>0.0017</td>
<td>Zn 40</td>
<td>0.0011</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr 80</td>
<td>0.0017</td>
<td>Pb 80</td>
<td>0.0020</td>
<td>Zn 80</td>
<td>0.0021</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cr 160</td>
<td>0.0010</td>
<td>Pb 160</td>
<td>0.0011</td>
<td>Zn 160</td>
<td>0.0019</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The mortar contaminated with 40 ppm of chromium showed the lower sorptivity and Zn 80 the higher value of sorptivity. The mortar Cr 40 showed the lowest sorptivity and a higher porosity. In the other words, it corresponds to interference of the other factors in the water absorption capacity of the mortars as the pores tortuosity and diameter size.

Afterward to water capillary absorption test, the mortars were maintained wrapped with parafilm to prevent drying, during eight days. After this period the mortars were submitted to electrical impedance measurements. These measurements are necessary for the determination of the formation factor used to calculate the tortuosity. Figure 8 presents the impedance spectrums of the mortars contaminated with chromium. It was possible to identify a decrease of reactance and resistance values, in relation to 28 days of curing.

![Figure 8: Impedance spectrums of the mortars with chromium after to water absorption test.](image1.png)

In the mortars contaminated with lead (Fig. 9), the sample Pb 160 showed the greater electrical resistance and the mortar Pb 40 showed the lower electrical resistance. And in the 28 days of age, these mortars showed a contrary performance. This fact may be associated the greater ions polarization in the water presence (after water absorption test).

![Figure 9: Impedance spectrums of the mortars with lead after to water absorption test.](image2.png)

All the mortars contaminated with zinc (Fig. 10) showed electrical resistance total close to reference mortar. The values of electrical resistance and reactance were lower after the absorption test than the values measured at 28 days of curing and this tendency was verified in the mortars contaminated with chromium and lead.
3.4 Tortuosity

In general, the addition of chromium, lead and zinc in the mortars decreased the tortuosity of the porous network (Table 2). Only the samples Cr 80 and Zn 160 showed a greater tortuosity than the reference mortar, without contamination. The higher tortuosity of sample Cr 80 may be explained through of your behavior verified on the water absorption test (Fig. 5). In the begging of the test the water absorption by mortar Cr 80 was very fast and after one minute, the absorption decreased with the time. This fast absorption resulted in a higher sorptivity of this sample. Nevertheless, an increase of the sorptivity not reflects a loss tortuosity. There are other factors that may influence the tortuosity [2].

Table 2: Tortuosity of the contaminated mortars.

<table>
<thead>
<tr>
<th>Mortars</th>
<th>( \tau )</th>
<th>Mortars</th>
<th>( \tau )</th>
<th>Mortars</th>
<th>( \tau )</th>
<th>Mortars</th>
<th>( \tau )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ref.</td>
<td>99.239</td>
<td>Cr 20</td>
<td>85.221</td>
<td>Pb 20</td>
<td>80.993</td>
<td>Zn 20</td>
<td>72.527</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cr 40</td>
<td>71.905</td>
<td>Pb 40</td>
<td>53.025</td>
<td>Zn 40</td>
<td>69.860</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cr 80</td>
<td>137.280</td>
<td>Pb 80</td>
<td>78.721</td>
<td>Zn 80</td>
<td>75.562</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Cr 160</td>
<td>93.043</td>
<td>Pb 160</td>
<td>92.431</td>
<td>Zn 160</td>
<td>101.668</td>
</tr>
</tbody>
</table>

4 CONCLUSIONS

- The impedance spectrums may to reflect in the microstructure changes of the mortars and the water absorbed quantified that decreased substantially the electrical resistances. The electrical test showed a variation of electrical capacitance in accordance with the C-S-H gel formation, how occurred in the mortars contaminated with lead. In the mortars contaminated with zinc, the mortars presented electrical resistances and reactances much lower than the reference mortar, without contamination.
- It can be observed that chromium decreased the sorptivity of the contaminated mortars and the heavy metals lead and zinc increased. The curves of water absorbed volume at time were more expressive for the understanding of the results of electrical impedance after the water absorption test.
- Tortuosity obtained by electrical tests was consistent with international literature and the addition of heavy metals decreased the tortuosity of the cement mortars.
- As the heavy metals affect the sorptivity and the tortuosity of the mortars, they also affect the durability of these cementitious materials.
ACKNOWLEDGEMENTS

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REFERENCES


THE INFLUENCE OF WETTING PRIOR AND ADDITION OF FINES IN PRODUCED MORTARS WITH RECYCLED AGGREGATE

Lidiane Fernanda Jochem(1), Janaíde Cavalcante Rocha(1), Malik Cheriaf(1)

(1) Department of Civil Engineering, Federal University of Santa Catarina – UFSC, Florianopolis – SC, Brazil – lidijochem@gmail.com; janaide.rocha@ufsc.br; malik.cheriaf@gmail.com

Abstract

The construction and demolition waste have an important mineral portion which can be benefited for producing recycled aggregate for use in engineering works. The fine fraction, for a long time, was disregarded, because it was believed to have properties with low quality. However, the fine fraction can be improved through appropriate process showing big potential for use in technological substitution to natural aggregate, contributing to the development of sustainable materials. In this study verified the effect of wetting prior of particle size composition, using recycled aggregate dry and wet previously, and setting the maximum level of added fine fraction (less than 150 µm) that mortars support in a composition with fine sand (0.15/1.2 mm). Five different compositions have been defined, varying the amount of fine and cement. Were determined the physical properties of aggregates, then the mortars were prepared and evaluated the properties in fresh and hardened state. The results showed that wetting prior of the aggregate, to addition of fines and cement content affected/improved properties of mortars. Since the fine added in the mortars aid in the plasticity of the mix, durability and prevented the appearance of cracks.

1 INTRODUCTION

The construction and demolition waste (CDW) are derived from construction activities, reform and demolition, according CONAMA Resolution [1]. These wastes are from origin, stages of construction and construction processes different, consequently the aggregates generated have great variability of the quality, composition and characteristics, however presents an important mineral portion which can be benefited for the production of recycled aggregates.

The growing demand in construction causes the increase occurring of waste of CDW, which being searched for the most varied applications, including, for coating mortars. However to dissipate the use of CDW in engineering works are needed parameters that
control the production and application of these mortars, reducing the variability of properties of aggregates.

[2] have shown in their studies that the fine fraction (particles below 4.8 mm) represents about 50% of the weight of the crushed CDW in coarse recycled aggregates production.

For a long time, the fine fraction was disregarded or used as a road pavement base because it was believed to have low quality properties [3]. However, recent studies have shown that fine fraction properties are not that different from coarse fraction properties, and can even be improved with appropriate processing [4].

Other studies have been carried out on crushed red bricks used as a partial replacement of natural sand in mortar production [3] and [5]. The use of fine recycled aggregate in coating mortar induces considerable shrinkage due to the high level of absorption of these aggregates and the pre-wetting could contribute to reducing the cracking problems.

2 MATERIAL AND METHODS

The recycled aggregate from construction and demolition used in this study was obtained from a processing plant located in the state of São Paulo, Brazil. The cement used was Portland cement (CPII-F 32). The fine material (<0.15 mm) added to the mortars was the fines of the own recycled aggregate (which was composed of crushed mortar and concrete).

In order to start the tests, all aggregates were prepared by removing the fine particles adhered by placing the aggregate in a 150 μm sieve and washing under tap water. This procedure was carried out for all of the aggregates used in order to ensure the percentage of fines incorporated in the mortar.

For the composition of the mortar mix, a fine grain-size distribution curve (D/d) (1.2/0.15 mm) was defined and five different compositions, varying the amount of fines from 0 to 24% (in relation to aggregate), that is, the ratio by weight ranged from 1:0:7.5 to 1:2:5.5 (cement:fines:aggregate). The water/cement ratio was 1.82.

The mortar mixes were prepared using the dry recycled (CDW) or previously wetted (CDW-W) aggregates. The amount of water used to wet the aggregate was determined from the product of the aggregate and the percentage absorption of the grains, obtained in a water capillary absorption test performed in the granulometric curve. The water used to saturate the aggregate was discounted of the total quantity of water in the composition.

The tests were carried out according to standard, however, for tests that do not have standard were used experimental procedures cited in the bibliography. The mechanical tests were carried out on 50 x 100 mm cylindrical samples.

For the characterization of the fine material (less than 150 μm) of the recycled aggregates, the chemical composition was determined by energy dispersive X-ray spectrometry (EDS) and the loss on ignition was carried out in 900°C.

The water absorption test carried out by capillary was based on the procedure settled at the ValoRes-UFSC laboratory [6, 7].

The elastic modulus test was carried out on 50x 100 mm cylindrical samples, adapted from the Brazilian Standard [8].
3 RESULTS

3.1 Characteristics of aggregates

The granulometric composition was obtained from a reference curve formed by: 80 %, 50 % and 30 % of the material dimensions: 0.6 mm, 0.3 mm and 0.15 mm. With a maximum aggregate size of 1.2 mm. The fraction less than 0.15 mm was added in proportions of 0, 6, 12, 18 and 24 % in the mixtures the mortars, and the fineness modulus in each composition with addition of fines was, respectively, 2.03, 1.84, 1.66, 1.49, 1.33.

The specific gravity of granulometric curve (D/d: 1.2/ 0.15 mm) and the fine fraction (<0.15 mm) are, respectively, 2.57 g/cm³ and 2.60 g/cm³, with unit mass of 1.15 g/cm³ and 1.01 g/cm³ and void ratio expressed in 55 % and 61 %. The absorption by immersion and capillary absorption, for 24 hours, was 13.5 % and 14.8 % of granulometric curve and the fine fraction (<0.15 mm) was 46 % and 42 %.

The laser grain size distribution of fine fraction has 100 % of particles smaller 0.08 mm, 50% of particles have a diameter less than 0.04 mm and 20 % of particles are less than 0.008 mm.

The chemical analysis carried out on the 150 µm fraction of the recycled aggregate showed the presence of CaO (40.8 %), SiO₂ (20.4 %), Al₂O₃ (9.4 %), Fe₂O₃ (3.2 %), K₂O (1.84 %), and other compounds (<1 %) and the loss on ignition was 23 %. The high calcium oxide content and loss on ignition are due to the presence of the cementitious matrix or hydrated cement from the recycled aggregate.

The area surface specific in the fine fraction determined using the blaine permability method was 2300 cm²/g, which is consistent with the value found by [9] for waste from the crushing mortars. And for CPII F-32, the value found was 3070 cm²/g.

3.2 Characteristics of the mortars in the fresh state

Table 1 shows the values of specific gravity and workability of mortars in the fresh state.

<table>
<thead>
<tr>
<th>Mortars/ trace (by weight)</th>
<th>Fines content (%)</th>
<th>Specific gravity (g/cm³)</th>
<th>Flow (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1: 0: 7.5</td>
<td>0</td>
<td>1.53</td>
<td>179.7</td>
</tr>
<tr>
<td>1: 0.5: 7</td>
<td>6</td>
<td>1.54</td>
<td>187.5</td>
</tr>
<tr>
<td>1: 1: 6.5</td>
<td>12</td>
<td>1.64</td>
<td>183.4</td>
</tr>
<tr>
<td>1: 1.5: 6</td>
<td>18</td>
<td>1.58</td>
<td>198.0</td>
</tr>
<tr>
<td>1: 2: 5.5</td>
<td>24</td>
<td>1.66</td>
<td>198.9</td>
</tr>
<tr>
<td>CDW</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1: 0: 7.5</td>
<td>0</td>
<td>1.30</td>
<td>170.1</td>
</tr>
<tr>
<td>1: 0.5: 7</td>
<td>6</td>
<td>1.34</td>
<td>175.2</td>
</tr>
<tr>
<td>1: 1: 6.5</td>
<td>12</td>
<td>1.41</td>
<td>173.5</td>
</tr>
<tr>
<td>1: 1.5: 6</td>
<td>18</td>
<td>1.53</td>
<td>171.0</td>
</tr>
<tr>
<td>1: 2: 5.5</td>
<td>24</td>
<td>1.51</td>
<td>166.4</td>
</tr>
</tbody>
</table>

W

612
According to the classification of [10] the mortars are classified with normal mortar and can be used in coating, because the specific gravity were within the limits of 2.30 and 1.40 g/cm³. However the mortars with CDW-W 0 % and 6 % specific gravity were below this value, as shown in Table 1.

Table 1 gives the values for the flow of the mortars. The addition of fines increased the workability, but in mortar with 12 % of fine with CDW-W decreased the workability due the saturation of fines in the mix.

### 3.3 Characteristic of the mortars in the hardened state

Table 2 shows the values compressive strength, flexural strength, modulus of elasticity carried out after 28 days of curing and the cement consumption.

<table>
<thead>
<tr>
<th>Mortars</th>
<th>Compressive strength (MPa)</th>
<th>Flexural strength (MPa)</th>
<th>Modulus of elasticity (GPa)</th>
<th>Cement consumption (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trace (by weight)</td>
<td>Fines (%)</td>
<td>Average</td>
<td>S.D.</td>
<td>Average</td>
</tr>
<tr>
<td>CDW</td>
<td>1: 0: 7.5</td>
<td>0</td>
<td>0.86</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>1: 0.5: 7</td>
<td>6</td>
<td>1.86</td>
<td>0.22</td>
</tr>
<tr>
<td></td>
<td>1: 1: 6.5</td>
<td>12</td>
<td>3.51</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td>1: 1.5: 6</td>
<td>18</td>
<td>3.98</td>
<td>0.16</td>
</tr>
<tr>
<td></td>
<td>1: 2: 5.5</td>
<td>24</td>
<td>4.75</td>
<td>0.19</td>
</tr>
<tr>
<td>CDW-W</td>
<td>1: 0: 7.5</td>
<td>0</td>
<td>0.00</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1: 0.5: 7</td>
<td>6</td>
<td>0.00</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1: 1: 6.5</td>
<td>12</td>
<td>0.00</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>1: 1.5: 6</td>
<td>18</td>
<td>2.61</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>1: 2: 5.5</td>
<td>24</td>
<td>3.07</td>
<td>0.02</td>
</tr>
</tbody>
</table>

In the compressive strength and flexural strength of mortars realizes a tendency to increase resistance with increased fines, as shown in Table 2. The mortars with CDW presented values of strength higher than mortars with CDW-W.

The values of modulus of elasticity found for the CDW and CDW-W were almost constant, except in sample without fines that reduced, due to lack of material less than 0.15 mm which helps in filling the pores, making the material more dense and thus more resistant. The values founded by [11] for the modulus of elasticity of mortars containing 0% and 20% recycled aggregate from the crushing concrete, are higher than those found in this study, this fact is justified because the research was used a trace 1: 4 without the addition of fine, with only the fine material own, w/c ratio less and low addition of recycled aggregate (Table 2)
The mortars CDW-W reduced cement consumption compared with mortars CDW. Increasing the quantity of fines in the mortars increased the cement consumption in the mortar CDW and CDW-W (Table 2).

The values of specific gravity, water absorption by immersion, open porosity, capillary absorption and wetting angle of mortars are showed in Table 3. The prior wetting of the mortars increased the specific gravity in the all traces.

<table>
<thead>
<tr>
<th>Trace (by weight)</th>
<th>Fines (%)</th>
<th>Specific gravity (g/cm³)</th>
<th>Absorption by immersion (%)</th>
<th>Open porosity (%)</th>
<th>Capillary absorption (%)</th>
<th>Wetting angle (º)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CDW</td>
<td>1: 0: 7.5</td>
<td>0</td>
<td>1.43</td>
<td>38.0</td>
<td>50.0</td>
<td>5.1</td>
</tr>
<tr>
<td></td>
<td>1: 0.5: 7</td>
<td>6</td>
<td>1.50</td>
<td>38.0</td>
<td>50.0</td>
<td>5.2</td>
</tr>
<tr>
<td></td>
<td>1: 1: 6.5</td>
<td>12</td>
<td>1.61</td>
<td>31.4</td>
<td>44.9</td>
<td>4.5</td>
</tr>
<tr>
<td></td>
<td>1: 1.5: 6</td>
<td>18</td>
<td>1.60</td>
<td>32.6</td>
<td>45.5</td>
<td>4.7</td>
</tr>
<tr>
<td></td>
<td>1: 2: 5.5</td>
<td>24</td>
<td>1.62</td>
<td>29.2</td>
<td>42.0</td>
<td>4.8</td>
</tr>
<tr>
<td>CDW-W</td>
<td>1: 0: 7.5</td>
<td>0</td>
<td>1.25</td>
<td>49.4</td>
<td>56.6</td>
<td>11.2</td>
</tr>
<tr>
<td></td>
<td>1: 0.5: 7</td>
<td>6</td>
<td>1.29</td>
<td>48.8</td>
<td>56.3</td>
<td>17.0</td>
</tr>
<tr>
<td></td>
<td>1: 1: 6.5</td>
<td>12</td>
<td>1.36</td>
<td>43.5</td>
<td>53.6</td>
<td>5.6</td>
</tr>
<tr>
<td></td>
<td>1: 1.5: 6</td>
<td>18</td>
<td>1.47</td>
<td>40.1</td>
<td>51.5</td>
<td>3.3</td>
</tr>
<tr>
<td></td>
<td>1: 2: 5.5</td>
<td>24</td>
<td>1.44</td>
<td>39.1</td>
<td>50.4</td>
<td>3.2</td>
</tr>
</tbody>
</table>

It is observed that the bigger the open porosity (interconnected pore) bigger is the absorption. The mortars with recycled aggregates absorb more water and consequently have a greater open porosity [12]. The increased amount of fine reduced absorption by immersion and porosity in mortars CDW and CDW-W.

Respect to capillary absorption in the mortars with CDW no tendency was observed with addition of fine, leaving the capillary absorption constant value. In Table 3 the addition of fines in the mortars with CDW-W reduced capillary absorption. The reduction in absorption is due to internal cure of the samples, over time the water contained inside the grains will be released and assists in hydration of the cement, resulting in a low porosity and permeability. However, the increase in capillary absorption occurred in the mortars with 0 and 6 % of fines, because these mortars shown the high porosity and permeability, being the surface of the sample with visible pores.

In Table 3, we can see that almost all of wetting angles were below 90º, which means that mortars have good wettability [13]. The smaller the wetting angle is more wetting the material, that is the greater the potential for absorption of the mortar. When compared traces of CDW with CDW-W, it can be seen that the samples have previously wetted showed angles similar to CDW, varying with greater intensity when compared to the mortar with 12 and 18
% fines, in this case, when the aggregate was saturated before the preparation of mortars reduced the potential for absorption of the samples.

4 CONCLUSIONS

The addition of fines (<150 µm) in mortars aids in improving the properties, due to the effect of filler and the higher porosity of the fine, allowing it to a better connection with the cement paste. This fact is reinforced by the mortars without fines did not show the best results, realizing the importance of the addition of the fines to mortars.

The wetting prior of recycled aggregate, so that it does not absorb the water needed the mix, practically all the properties worsened, improving only wetting angle and reduced the cement consumption and capillary absorption (18 e 24 % of fines).

Thus, it is concluded that the mortars produced with recycled aggregate and fines show characteristics desirable for their use as mortars.

REFERENCES

MOULD RISK ASSESSMENT FOR THERMAL BRIDGES: WHAT IS THE IMPACT OF THE MOULD PREDICTION MODEL?

E. Vereecken (1) and S. Roels (1)

(1) Building Physics Section, KU Leuven, Leuven – Evy.Vereecken@bwk.kuleuven.be; Staf.Roels@bwk.kuleuven.be

Abstract
Whereas previously the temperature ratio was commonly used as criterion to predict the mould growth risk during the design stage, nowadays other – more advanced – mould prediction models are available. These models include the main influence factors for mould growth: the surface temperature and relative humidity. Other factors, e.g. the substrate and the mould species, are only rarely included. In addition, the criterion which indicates mould risk differs for each model.

In this paper, a comparison between the different mould prediction models is made. To do so, the relative humidity and temperature courses found at the interior surface of three thermal bridges are used as an input in the mould prediction models. For some conditions it was shown that a different mould risk conclusion was drawn depending on the prediction model.

1 INTRODUCTION
To evaluate the durability of materials to mould growth and hence to minimise potential mould damage in buildings, reliable mould prediction models are necessary. While previously commonly the temperature ratio was used as criterion to predict mould growth risk during the design stage, nowadays more advanced mould prediction models are available, e.g. the VTT model [1], isopleth systems [2], the biohygrothermal model [2], etc. These models include the main influencing factors for mould growth: the surface temperature (T) and relative humidity (RH). Other factors, e.g. the substrate and the mould species, are included in only some of the existing models. In addition, most models are developed based on steady-state experiments and the criterion which indicates the mould risk differs for each model. Consequently, a different conclusion may be drawn depending on the used model.

Vereecken and Roels [3] showed widely varying results for the different mould prediction models commonly used in building simulation programs. In the latter study, however, fictitious cycles in temperature and relative humidity were analysed. The question arises if a similar discrepancy can be found for real varying climatic conditions. Thereto, in the current paper, the different mould prediction models are applied on simulated boundary conditions for
three thermal bridges. The results are also compared to the conclusions obtained based on the temperature ratio. Additionally, the impact of interior insulation on the mould risk is evaluated. However, due to the lack of knowledge concerning the reliability of the current mould prediction models, the latter evaluation serves mainly as a qualitative comparison.

2 MOULD PREDICTION MODELS

In the current section, the different existing mould prediction models and mould risk criteria are briefly reiterated. A more in depth description and some remarks on the different prediction models can be found in [3].

2.1 Temperature ratio

To minimise the mould risk, IEA-Annex 14 [4] proposes a design criteria based on the temperature ratio:

\[ \tau = \frac{T_{s,\text{min}} - T_e}{T_i - T_e} \]  

with \( T_{s,\text{min}} \) (K) the minimum indoor surface temperature and \( T_i \) and \( T_e \) respectively the inside and outside temperature (K). A temperature ratio of 0.7 is proposed as criterion, related to an acceptable mould risk of 5%. A lower ratio introduces an unacceptable high mould risk.

2.2 VTT model

The VTT model is an empirical mould prediction model, in which the growth development is expressed by the mould index \( M \) [1]. This index ranges from 0 (no mould growth) to 6 (the mould growth covers nearly 100% of the surface) and is often used as a design criterion. A mould index equal to 1 is often defined as the maximum tolerable value, since from that moment the germination process is assumed to start. The VTT model is based on regression analysis of a set of measured data [1], based on which the influence of temperature, relative humidity, surface, exposure time and dry periods is included in the VTT model. A first VTT model was based on lab experiments on pine and spruce sapwood. The calculation procedure can be found in [1][3]. Recently, the VTT model is expanded to other materials [5].

2.3 Isopleth models

Isopleth curves separate favourable from unfavourable T- and RH-conditions for mould growth. A first model that uses isopleths is the ESP-r model [6][7]. In this model, the mould fungi found in buildings are subdivided in 6 categories with respect to relative humidity and temperature: Highly xerophilic, Xerophilic, Moderately xerophilic, Moderately hydrophilic, Hydrophilic and Highly hydrophilic. For each of these categories a growth limit curve defined by a third-order polynomial function is determined based on an analysis of published data. When the RH- and T-combination exceeds such a curve, mould growth of the matching fungi will occur. The main disadvantage of this model is that the exposure time is not taken into account. A single exceed of the isopleths is set equal to mould formation.

Sedlbauer [2] subdivided the mould species and materials found in buildings in a set of classes. For each class, he developed isopleths indicating the time till germination and the mould growth rate. The conditions below which no spore germination or growth will occur are indicated by the LIM (Lowest Isopleth for Mould)-curve. Note that the isopleths are defined based on stationary laboratory experiments. Hence, an interim drying out of the spores cannot be taken into account.
To take into account the temperature and relative humidity at previous time steps, Moon [8] established the mould germination graph method. In this method, each curve in the isopleth system is indicated by a certain required exposure time for initiation of mould germination. For each curve, the associated accumulated exposure time can be recorded. When the accumulated exposure time for a group is equal or larger than its required exposure time, mould growth can start. Consequently, using this method also fluctuating conditions can be studied. However, although a delay during unfavourable conditions is included, a possible drying out of the spores is not considered in this model.

2.4 Biohygrothermal model

To make a more reliable prediction of the mould risk possible in cases of transient conditions, Sedlbauer extended his isopleth model with the biohygrothermal model [2]. In this model, the moisture balance of a spore, which has a certain osmotic potential and which can consequently absorb water from the environment dependent on the transient boundary conditions, is calculated. This means that even an interim drying out of the fungus spores can be considered. The spore is supposed to have germinated when a certain moisture content – the critical moisture content – is reached. The critical moisture content can be determined based on the moisture retention curve of the spore and the critical relative humidity found in the germination LIM-curve.

2.5 Mould index in WUFI-Bio

Since the mould growth in millimeters as determined by the isopleth system and by the biohygrothermal model is not a reasonable unit, Krus et al. [9] developed a conversion function based on which the mould growth in millimeters can be transformed in the mould index. More details can be found in [3].

2.6 General remark about the mould risk criteria

In the different models, also different definitions for the start of mould growth can be found. For instance, in the VTT model a mould index equal to 1 is defined as the start of germination and consequently as the value above which a mould risk exists. In the biohygrothermal model a spore moisture content higher than the critical moisture content indicates the start of germination. However, to evaluate the mould risk, in the latter model the mould growth per year is used to evaluate the mould risk [10]. To do so, a ‘signal light’ defines the risk. More than 200 mm mould growth per year is indicated by a ‘red light’, which is not acceptable. Less than 50 mm mould growth per year is indicated by a ‘green light’ and is usually acceptable. Between those two cases an additional evaluation is necessary, which is indicated by a ‘yellow light’.

3 APPLICATION

The analysis in the current study is performed for three thermal bridges, i.e. junctions between an exterior and an interior wall with a different level of insulation (Figure 1a,c,e). The indoor temperature and relative humidity is assumed to be constant and is set at respectively 20 °C and 50%. For the other boundary conditions, the material properties and the numerical model the reader is referred to Vereecken [11]. Figure 1b,d,f shows for the different configurations the numerically predicted temperature and relative humidity obtained in the corner in function of the time.
4 MOULD GROWTH ASSESSMENT

The mould growth evaluation is performed based on hourly data for the predicted surface temperature and surface relative humidity in the interior corner. Figure 2 shows the critical isopleths used in the ESP-r model together with the simulated hourly T- and RH-combinations in the corner of the three configurations. For Configuration 1 and 2, the influence of wind-driven rain is visible. When interior insulation is applied at both the exterior and the interior wall (Configuration 3), no mould risk is predicted. In the latter case, wind-driven rain does not influence the results.

Figure 3 shows the simulated hourly T- and RH-combinations as obtained in the corner of the different configurations exposed to wind-driven rain together with Sedlbauer’s isopleth system for category I. For Configuration 1 and 2, the LIM-curve is exceeded. For Configuration 3, no mould risk is predicted.
Figure 2. Simulated RH-T combinations and critical ESP-r isopleths.

Figure 3. Simulated RH-T combinations and Sedlbauer’s isopleth system for substrate category I: a,b) germination isopleths, c,d) growth rate isopleths.

Figure 4 shows an overview of the maximum mould intensity. As shown in Figure 4a, the difference between the maximum mould growth determined by Moon’s germination graph method (Sedlbauer’s isopleths, linear interpolation between the growth curves) and by the biohygrothermal model (based on the WUFI-Bio software [10] wherein the initial spore RH is set at 50%) is less pronounced than found in [3]. This can be attributed to the lower number of periods with unfavourable conditions in cases of the real varying conditions. The longer time span of these periods with unfavourable conditions is of minor importance. A comparison between the mould index determined by transforming the results of the biohygrothermal model (WUFI-Bio M) and the mould index obtained based on the VTT model is shown in Figure 4b. For the VTT model, the results obtained by both the original model and the updated model (sensitivity class s) are shown. The application of the original model is, however, theoretical since the finishing layers in this study are no wooden substrates. Where the VTT model implements a decline of the mould index during the periods with unfavourable conditions, in WUFI-Bio the mould index remains constant during these
periods. This may result in different mould risk conclusions, as shown by a comparison between the WUFI-Bio mould index and the VTT mould index for class s determined for Configuration 2.

Figure 4. Maximum mould intensity: a) mould growth, b) mould index.

Figure 5 shows the time till mould germination occurs. The large difference between the time till germination obtained based on a mould index equal or larger than 1 (VTT original, VTT class s, WUFI-Bio M) and the criteria used in combination with the other mould prediction models ($w_{\text{spore}} \geq w_{\text{crit}}$, exceed of isopleth, ...) is clearly pronounced. For Configuration 3 not exposed to wind-driven rain, also a large difference between the germination time obtained with Sedlbauer’s isopleth system and the biohygrothermal model is observed. The reason for this is the use of the LIM-curve for the determination of the critical spore moisture content.

Figure 5. Time till germination. For the ESP-r model, the results in cases of a moderately xerophilic (MX) species are shown. For Configuration 4 (not shown), no mould risk occurs.
Table 1 gives for the three configurations an overview of the obtained mould risk conclusions. For Configuration 3, nor the temperature ratio nor the mould prediction models indicate a mould risk. For Configuration 2 and 3, different conclusions are obtained. Based on the temperature ratio, the configurations are labeled as building details with an acceptable low mould risk. Due to the standardised determination procedure of the temperature ratio, the wind-driven rain has no influence on these results.

When using the VTT model, for the configurations not exposed to wind-driven rain no mould risk is found due to the decline in mould index during unfavourable conditions. When the configurations are exposed to wind-driven rain, for Configuration 2 a mould risk is predicted if the original VTT model is used.

When using Moon’s germination graph method, germination is expected to start for Configuration 2 and 3. Though, if the mould risk is evaluated based on the mould growth per year in combination with the ‘signal light’- criterion [10], for the configurations not exposed to wind-driven rain other conclusions are found. In the biohygrothermal model germination occurs if the spore moisture content is higher than the critical moisture content. This is the case for Configuration 2 and 3, independently of the wind-driven rain exposed to. Also here, evaluating the mould growth based on the ‘signal rule’-rule results for Configurations 2 and 3 not exposed to wind-driven rain in other conclusions than obtained when germination is used as a limit state. Based on the mould index determined in WUFI-Bio only for Configuration 2 and 3 exposed to wind-driven rain a mould risk is predicted.

Table 1. Mould risk conclusions (GERM = germination, ‘SL’ = ’Signal light’-rule).

<table>
<thead>
<tr>
<th>Configuration Model</th>
<th>1 Without WDR</th>
<th>2 Without WDR</th>
<th>3 Without WDR</th>
<th>1 With WDR</th>
<th>2 With WDR</th>
<th>3 With WDR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature ratio</td>
<td>No (0.72)</td>
<td>No (0.77)</td>
<td>No (0.92)</td>
<td>No (0.72)</td>
<td>No (0.77)</td>
<td>No (0.92)</td>
</tr>
<tr>
<td>VTT original</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>VTT class s</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>ESP-r</td>
<td>Yes if xerophilic</td>
<td>Yes if xerophilic</td>
<td>No</td>
<td>Yes</td>
<td>Yes if xerophilic</td>
<td>No</td>
</tr>
<tr>
<td>Moon’s germination graph method with linear interpolation (Sedlbauer I)</td>
<td>GERM: Yes ‘SL’: Extra analysis</td>
<td>GERM: Yes ‘SL’: Extra analysis</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Biohygrothermal model</td>
<td>&gt; wcrit: Yes ‘SL’: Extra analysis required</td>
<td>&gt; wcrit: Yes ‘SL’: Extra analysis required</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>WUFI-Bio M</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
</tr>
</tbody>
</table>
5 CONCLUSIONS

A mould risk assessment was performed for three thermal bridges. Where the temperature ratio indicated the configurations as acceptable building details, some, but not all, of the mould prediction models indicated a mould risk. As also found in [3], the different evaluation criteria may result in different conclusions. Though, the differences between the different models are less pronounced. The number of periods with unfavourable conditions influences the magnitude of the difference between the results obtained based on Moon’s germination graph method and the biohygrothermal model.

Additionally, the influence of interior insulation and wind-driven rain was visible. When interior insulation was applied at both the exterior and the interior wall, no mould risk was predicted by any of the models. The mould intensity and the time till germination after applying interior insulation at solely the exterior wall was lower than determined for the non-insulated thermal bridge. Wind-driven rain was found to induce an increased mould risk, while this factor is not included in the temperature ratio.

ACKNOWLEDGEMENTS

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REFERENCES

A METHOD FOR BLIND VALIDATION OF HYGROTHERMAL CALCULATION TOOLS

S. O. Mundt-Petersen (1), L.-E. Harderup (2)

(1) Department of Building Physics, Lund University, Lund, Sweden – solof.mundt_peterersen@byggtek.lth.se; lars-erik.harderup@byggtek.lth.se

Abstract
The use of hygrothermal calculation tools for dimensional purposes in the design phase is common. However, used tools are usually not blindly verified in real, on-site conditions.

This paper presents a method for verification of hygrothermal calculation tools for real-life situations. In the method calculations become carried out before the measurement results are known, i.e. by blind calculations, which afterwards are compared with measurements.

The main conclusion is that blind validations are reliable since intentional or unintentional adjustments of calculated results, to obtain better correlations to the measured values, are impossible. Other positive effect is that information about how the user perceives the tool could be found since the blind calculations are similar to the designer situation in daily work.

1 INTRODUCTION

1.1 Background
Laws and regulations require that energy need, indoor climate conditions and moisture safety should be estimated and analysed during the design phase, before a house is built [1]. There are several available calculation tools that could fulfil this purpose, such as IDA ICE, VIP-Energy, DEROB, WUFI, DELPHIN, HAM-tools etc. [2, 3, 4].

Known calculation tools are based on physical laws using established equations calculating the relationship between different factors affecting energy need, indoor climate or moisture distribution. After complete calculation the tools return an answer of calculated parameters, such as energy need, temperature, moisture distribution, indoor climate or moisture transport.

In general, known calculation tools are verified according to standards, BESTEST (Building Energy Simulation TEST) or other methods which contains comparative-, analytical- and empirical validation process [5, 6, 7, 8, 9]. A comparative validation is based on calculation results compared with results from other equivalent tools. In an analytical validation the results become controlled, often in different parts, to reference results taken from established equations. An empirical validation include laboratory-, field- or on-site field
Some validation methods show weaknesses in the empirical validation process [9]. In many situations there is a lack of available experimental empirical investigations [10].

Lack of complete knowledge and reliable equations complicate analytical and numerical methods in some situations, such as moisture transport in materials. Simplifications have been made where there was a lack of knowledge, in parts with minor influence on the results or parts of less importance. There are hygrothermal calculation tools that besides physical laws and established equations focus on empirical validations in laboratory and field test including initial boundary conditions and surrounding climate boundary data [10, 11, 12, 13]. One tested tool showed good correlations in a real situation with real conditions [14, 15].

Compared to other areas, such as medicine or drug-, foods and drinks-, animal food, cosmetics, safety-, pesticides and chemicals development and testing used validations were based on the framework of Good Laboratory Practice, GLP. Principles for GLP are governed by law in compliance with EU directive, which include inspection programs [16]. In science, several of those areas were treated using different kind of blind or double-blind tests in order to reach safe and reliable results. GPL requirements for the computerized systems consist of documentation, training, back up, security etc. However, GLP also introduce the term “acceptance testing”. This means that computerized systems should be tested in its anticipated operating environment to determine whether the system is acceptable for operational use [17].

In the construction industry, hygrothermal calculation tools are used in the design process for dimensional purposes, i.e. estimating the outcome in unknown future on-site conditions. The users are not aware of, and could not estimate, precise future real upcoming outcome in designed building, depending on surrounding factors such as, climate, material properties, human behaviour etc. Hygrothermal calculation tools for dimensional purposes in the design phase are therefore in a context carried out blind, i.e. the user could only guess future results.

Empirical validations are mentioned in standards for hygrothermal calculation tools, but it is not specified if they should be carried out in laboratory, field tests or real on-site conditions. Developer also point out the problem with lack of empirical, especially on-site, tests [10]. Furthermore, standards do not mention that tests should be blind, as the case when using the tools in reality. Non-blind tests make it possible for the developer to consciously or unconsciously adjust calculated results to get better correlation with expected or wanted results. In a context this means that standards for verification of hygrothermal calculation tools do not fulfill the requirements within GLP since the calculation tools not become blindly verified and not tested in real on-site situations. Interior factors within the calculation tool or exterior factors, such as a varied vegetation shading, that are impossible to take into account in a tool might affect the calculation results on a significant manner.

Standards for validation of hygrothermal calculation tools do not consider limitations in usability. Some reports indicate that simplified and inappropriate input data is responsible to more deviations from real, wanted or expected values than defects in the calculation tool [18]. Lack of correct applied input data may depend on unsuitable users, poor usability or both.

This paper presents a blind method to validate hygrothermal calculation tools under real on-site conditions, or laboratory situations, including possible influences of usability using real climate boundary conditions.

1.2 Aim

The aim of this study was to present a blind method in a real context, including simulation of real situations for the users, in order to validate hygrothermal calculation tools in a reliable
manner. The purpose was also to show examples of important findings by using blind method and other positive effects that could be reached using blind validation methods.

1.3 Limitations

This study does not deal with developer sensitive tests. The software development process for the hygrothermal calculation tools, such as equations, numerical technique etc., was not considered in detailed. The examples in the paper were limited to a blind case study carried out in wooden framed houses in northern European climate conditions.

2 BLIND METHOD

A blind method means that calculations are made without knowing the results from the measurements. By definition the blind comparisons in this paper could also be called single-blind. Notice that the method used is not double-blind since it is possible to guess the measured results before the calculations are made. Examples where the single-blind method has been used are an empirical study where the unknown values consist of measured values.

Using blind calculations the situation become equivalent to when the designers calculate energy need, indoor climate or heat- and moisture transport before a house is built. Below presented blind method is rather simple and it makes you think why it is not more often used.

2.1 Ambient factors

In order to reach reliable output running the blind validation of hygrothermal tools, there was a need to take into account a number of ambient factors mentioned below.

1. Calculator - The person that runs the calculations in evaluated tool.
   To get a reliable situation the blind calculations should be carried out by a person (calculator) that are independent from the developer. The calculator should be a possible user and his level of knowledge corresponding to the lowest limit for a user.
2. Test objects, i.e. houses - Houses where calculations are carried out to validate the tool.
3. Measurements equipment - Measurement equipment to collect data to be compared with blind calculated values.
4. Measurement collector - The person that collects measurement data and keeps it hidden from the calculator until the calculation were finished and reported.
   The measurement collector should be independent from the developer.

2.2 The blind validation method – step by step

This section presents a step by step “manual” how a blind validation can be done. Especially important factors that have to be fulfilled are found in step 11-16.

1. Determination of test objects, i.e. houses.
   The test houses in which measurements and blind calculations are carried out should be representative for cases where evaluated tool aims to be used. Since the outdoor climate conditions will influence the results it is recommended that test houses are located in different regions, i.e. north-, east-, coast- and inland climate conditions [15].
2. Determination of positions to be used for validation in selected houses and envelopes.
   Depending on the purpose of the validation different positions could be chosen. The positions should be selected in a manner that they cover the purpose of the validation, i.e. all parameters included in the validated tool should be included. Each position should be motivated for the specific case. Relevant in- and outdoor climate boundary
conditions should be continuously measured. Other factors, which might affect the results, such as two-dimensional effects in one-dimensional calculations, should be documented and if possible be included in the measurements.

3. Calibration and testing of measurement equipment.
   Calibrate measuring sensors and control measurement equipment before mounting.

4. Installation of measurement sensors in chosen positions.
   Each position where measurement sensors are mounted should be documented, preferably both with drawings and photos. Chosen positions can be known for the calculator. Sensors could be mounted in the construction phase to study the influence of initial incidents during construction. Possibilities for a second calibration should be taken into account when mounting sensors, especially in occupied houses.

5. Monitor the construction phase.
   In order to notice possible deviations between the drawings and the real conditions in studied positions the construction phases in studied houses might be monitored. Factors and boundary conditions that affect the results, such as a high level of construction moisture etc. should be noticed and if possible applied in upcoming calculation model.

6. Start and maintain measurements.
   Measurements preferably start as soon as possible, even before the houses are completely built and occupied. The measurement collector controls that the measurements start and independently stores all results inaccessible to the calculator. The measurement collector should control the quality of the measurements during the measurement period and if necessary ensure that measurement equipment is repaired.

7. Design and calculation model.
   When measurements are carried out, calculation models of studied positions are made. Calculation models are based on drawings and photos etc. from the construction phase and sensor mounting with the intention of reflecting real conditions as well as possible. Parameters, boundary conditions etc. should be documented. Deviations from reality, such as one-dimensional calculations in a two-dimensional situation should be noticed.

8. Perform test calculations.
   As a part of a real situation in the design phase, test calculations of made calculation models are made, preferably using similar climate data. The reliability of the results should be evaluated and needed adjustments in calculation model should be made [19].

   As a part of a real situation in the design phase a sensitivity test of the parameters that might have a high influence on the results should be made [19]. The sensitivity test could be made as a parametric study and should be documented.

10. Receive real in- and outdoor climate boundary conditions.
    When the measuring period is completed, real in- and outdoor climate boundary conditions are received. Real climate boundary conditions are needed in calculation models to obtain calculated results that are comparable to on-site measurements [15].

11. Calculations for validation.
    When real in- and outdoor climate boundary conditions are received (10) these are used in calculations for each studied position, without knowing the measured results.

12. Calculator deliver calculation results to the measurement collector.
    After completed blind calculations (11), the results are sent to measurement collector.

After calculation results have been received (12), the measurement collector compiles measured results and if possible makes a second calibration of measurement results.

14. Measurement collector sends measurement results to the calculator.
   When the measurement collector receives the blindly calculated results (12) from the calculator the previously inaccessible measurements are open and sent to the calculator.

15. Calculator compare measured results with calculated values.
   When received previously inaccessible measured values (14), measured and calculated values are compared and published together. This must be done regardless of possible correlation or deviations. The comparison also could be carried out by a third person.

16. Control of published measured and blindly calculated values to original values.
   The measurement collector check that published values (15) are consistent with originally sent (14) and received (12) values, i.e. control that no adjustments are made.

17. Analyse and report deviations between measured and blindly calculated results.
   Possible reasons for deviations are analysed, documented and if possible explained.

3 EXAMPLES AND COMMENTS OF RESULTS FROM BLIND COMPARISONS

In this section some examples of results from a blind validation of a hygrothermal calculation tool are presented and briefly commented in a context to above presented method. The results were presented using the Folos 2D visual mold chart [20]. A simple figure of the conditions for each presented figure was given together with each chart. A complete description of studied cases could be found in separate reports and papers [14, 15, 21].

3.1 Influence of air flow in the air gap behind a cladding

Comparison of measured (black), blindly calculated (turquoise) and non-blind calculated (brown) temperature and relative humidity (RH) in a wood framed wall.

![Figure 1: Comparison of measured (black), blindly calculated (turquoise) and non-blind calculated (brown), with adjusted air flow in the air gap, temperature and relative humidity in a point located outside the vapor barrier.](image)
The results are taken from a paper in which more detailed information can be found [15, 21]. The difference between the blind and non-blind calculations was that the air flow in the air gap behind the cladding was increased. If the validation in Figure 1 was non-blind and/or performed by the developer it is probable that only the good results in the second case are presented. This would have hidden the important information that the air flow is of high importance for the moisture conditions in the wall and to obtain reliable results.

### 3.2 Deviations in relative humidity caused by differences in temperature

Comparison of measured and blind calculated temperature and RH in a wood framed wall. The results are taken from a paper in which more detailed information can be found [14, 15].

![Figure 2: Comparisons of measured and calculated RH and temperature in the exterior part of a wall. Calculated RH (turquoise) and measured RH (black). Calculated temperature (yellow) and measured temperature (dark blue). RH\(_{\text{crit}}\) derived from the calculated temperature (red). Calculated RH > RH\(_{\text{crit}}\) (light brown) and measured RH > RH\(_{\text{crit}}\) (purple). Periods with artificial climate boundary data (green).](image)

The lower measured relative humidity in Figure 2 depends on a higher measured temperature. I.e. the vapor content is the same but different measured and calculated temperatures give different vapor contents at saturation. However, minor adjustments of precise studied point in the calculation model gives a higher calculated temperature and later on create better correlation between measured and blindly calculated values, which might have been the case with non-blind calculation carried out by the user. Besides that it might be an imprecise location of studied position in the calculation model the divergence between measured and blind calculated values might depend on a thinner insulation thickness caused by the measuring sensor size or the air space around the measurement sensor [22]. Furthermore, in this case there were not any possibilities for a second calibration [15].

### 3.3 Differences in measured and calculated amplitude

Figure 2 also show that measured RH and temperature have higher amplitude compared to calculated values. This could be adjusted for in the calculation model by decreasing the heat
and moisture capacity for the insulation material. However, the most probable reason for the higher amplitude in measured values is the air gap which surrounds the sensors [22].

4 DISCUSSION

The main reasons for blind validations are that they give a higher reliability to evaluated tool. A blind evaluated tool also creates reliability for upcoming parametric studies using the same tool. Using blind validations reduces the focus on only reaching correlation to measurements or other results, into finding and explains important factors affecting the results; understand how the tool works both theoretically and by the users. After the house is built it is important to investigate which significant factors that is important for deviations to calculate values. For researchers it is also the deviations that lead to new findings and discoveries.

Blind validations indicate if and during what conditions evaluated tool is possible to use in reality. In commercial hygrothermal calculation tools there are also financial purposes which may create reasons to adjust calculation results. It is helpful to point out useful boundary conditions and other input data, such as relevant air flows in façade air gaps, as showed in Figure 1. Recommendation of reliable input data might also limit the problem with inaccurately applied input data to reach a wanted, but incorrect result. Unconscious applied defect input data could also ruin the results from a good calculation tool [18].

Blind on-site validations give a direction on sensitivities to be expected dependent on factors that not is possible to control, such as in- and outdoor climate and occupant behaviour. Studies show that those factors might have a significant influence on the results [15, 18].

In general, houses of today become evaluated when steady state conditions are reached. By implementing the validation in the construction phase the influence of possible incidents during the construction phase that can affect the house during several years might be visible.

It should be mentioned that non-blind comparisons might be needed in the development phase of calculation tools. However, blind methods force developers, researchers or testers to reflect in a kind of trial and error situation during the validations process. By practicing this it can be assumed that the persons gaining experience, which may be valuable in future work.

5 CONCLUSIONS

Hygrothermal calculation tools used in the design phase, before a house is built, ought to be blindly verified and without being influenced by the program developer. This can be fulfilled by carrying out the calculations before the results of the measurement are known, i.e. by blind calculations, which afterwards are compared with measurements.

Blind validations are reliable since intentional or unintentional adjustments of calculated results, to obtain better correlations to the measured values, are impossible. There are also other positive effects since the blind calculations are similar to the designer situation before a house is built. This provides important information about how the user perceives the tool and trains users, researchers and developer to make accurate estimations and improvements.

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METHODS FOR COMPENSATE LACK OF CLIMATE BONDARY DATA

S. O. Mundt-Petersen (1), P. Wallentén (1)

(1) Department of Building Physics, Lund University, Lund, Sweden – solof.mundt_peterersen@byggtek.lth.se; petter.wallenten@byggtek.lth.se

Abstract

Laws and regulations require that energy needs, indoor climate and moisture safety should be estimated and analyzed during the design phase, before a house is built. Available tools to predict the hygrothermal conditions need, in general, hourly exterior and interior climate boundary conditions. Available climate data sometimes consist of periods with incomplete or lack of data.

The aim or this paper is to present methods in order to supplement periods with lack of data and methods in order to create diffuse, direct and long wave radiation. Presented methods to supplement and create climate data was tested in a case study in a wood frame roof.

The importance of using reliable climate boundary conditions in order to estimate the hygrothermal conditions before a house was build is presented. It is shown that real climate boundary conditions need to be used to reach correlation between measured and calculated hygrothermal conditions. The need for reliable climate boundary conditions for dimensional purposes and that WUFI 5.2 can be used in a reliable manner when calculating hygrothermal conditions in wood frame roofs located in Northern European climate are shown.

1 INTRODUCTION

1.1 Background

New laws and regulations during the last years require that energy need, indoor climate conditions and moisture safety should be estimated and analyzed during the design phase, before a house is build [1]. Today there are several energy, heat- and moisture calculation tools that are used in order to fulfil the requirements by estimating needed factors in the design phase, such as IDA ICE, VIP-Energy, DEROB, WUFI, DELPHIN, HAM-tools etc. [2, 3, 4].

In general, the calculation tools calculate hourly values for energy need, indoor climate conditions or heat- and moisture transport using numerical solutions. In order to obtain reliable results hourly climate boundary conditions are needed as input since climate conditions varies over time, sometimes with high variations between different hours [2, 3]. Most energy, heat- and moisture calculation tools include default climate boundary conditions to be used in the evaluation process. These exterior default climate boundary conditions, in one way or another, are mainly based on hourly climate measurements carried out by national metrological and
hydrological institutes [5, 6, 7]. Hourly values are needed in order to determinate dimensional effects, such as highest energy effect need for heating or highest temporary moisture load.

The content of default exterior climate boundary conditions varies between different calculation tools and between different climate files. In some cases real exterior climate data from one single year can be used. In other cases some kind of mean climate was created based on real climate conditions from several years [4, 6, 7]. Exterior climate data can also be created using software climate tools [8]. Interior hourly climate boundary conditions can be created using standards based on used exterior climate data [9, 10] or dependent on limits for required indoor climate conditions [1, 6]. Measured or created climate data for specific projects, can be used as climate boundary conditions [6, 7, 3, 11].

Working with measured climate data from Swedish meteorological and hydrological institute, SMHI, or own measured interior or exterior climate data to be used as boundary climate data conditions in specific projects it was found that almost all measurements include periods with impaired or lack of data. Periods with lack of data depends on measurement problems and varies in length from single hours to longer periods up to months. Some cases lack measured short- and long wave radiation. In order to get complete climate boundary conditions when there are periods with lack of data, there is a need to develop methods to compensate for this lack of data.

1.2 Aim

The purpose of this paper is to present a method that can be used in order to compensate for periods with lack of climate data. The paper also aims to present methods that could be used in order to create climate data for direct and diffuse short wave radiation and long wave radiation.

The intention is further to compare measured and calculated temperature and relative humidity (RH), in a case study, where calculated values were carried out using real but inadequate climate boundary data that had been supplemented and radiation climate data that have been created using presented methods.

1.3 Limitations

This paper does not consider any material properties or material boundary conditions. The paper focus on coupled heat- and moisture transport in wood frame constructions, i.e. the methods for compensating lack of climate data were tested in a wood frame roof. The calculation model is only briefly described and a detailed description is found in separate reports [11, 12].

2 METHODS

The methods are divided in three parts; methods for supplementing periods with lack of climate data, a method for creating long- and a method for creating shortwave radiation.

Hygrothermal test calculations was then carried out using default boundary climate conditions [13] and real supplemented climate data [11, 12] with and without created long- and shortwave radiation. The calculations were carried out in a roof using the hygrothermal calculation tool WUFI 5.2. Calculated temperature and RH were then compared to measured values in studied position. Comparisons between measured and calculated values were made using the Folos 2D visual mold chart [14]. Periods when mold growth becomes possible was based on LIM I [15].

2.1 Methods supplementing lack of in- and outdoor climate boundary conditions

The methods for supplementing periods with impaired or lack of climate data were simple and varied depending on climate parameters and the length of periods with lack of data. Primarily, if other reliable climate data was available at the same time when lacks of data occurred this data
was used as supplement for the lack of data. If no other reliable climate data was available, shorter periods less than one week with lack of hourly exterior temperature, RH and radiation climate boundary data were supplemented with the previous day hourly climate data value. The last daily temperature and RH data were repeated up to seven days. Longer periods than one week with lack of hourly climate boundary data were supplemented with hourly climate data from the same hour and day previous year.

2.2 Method inventing diffuse- and direct solar radiation

The presented method calculates diffuse radiation based on known global radiation in a simple way. Diffuse solar radiation was estimated as a function of the global radiation. With a high amount of global radiation, Wh/m², the method estimated that a low percentage of the global radiation consists of diffuse radiation. The lower the amount of global radiation, Wh/m², the method estimates that a higher percentage of the global radiation consists of diffuse radiation. During nights without global radiation, i.e. global radiation < 2 Wh/m², the diffuse radiation was 0 Wh/m². The percentage of global radiation that assumes to consist of diffuse radiation changes stepwise between different amounts of global radiation as shown in Table 1.

The method was based on experience from measured global- and diffuse radiation in the Swedish city Växjö during 1996. The choice of period and city depends on the problem to find a period and location when measured diffuse and global radiations were available at the same time. The relationship between measured global and diffuse solar radiation and estimated diffuse solar radiation based on measured global solar radiation for Växjö 1996 were presented in Figure 1.

Table 1: Estimated diffuse radiation based on a percentage on the amount of global radiation.

<table>
<thead>
<tr>
<th>Global rad. (Wh/m²)</th>
<th>% diffuse of global rad. (%)</th>
<th>Diffuse rad. (Wh/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 – 1.9</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2 – 99.9</td>
<td>100</td>
<td>2 – 99.9</td>
</tr>
<tr>
<td>100 – 119.9</td>
<td>99</td>
<td>99 – 118.7</td>
</tr>
<tr>
<td>120 – 159.9</td>
<td>88</td>
<td>105.6 – 140.7</td>
</tr>
<tr>
<td>160 – 199.9</td>
<td>77</td>
<td>123 – 154.9</td>
</tr>
<tr>
<td>200 – 299.9</td>
<td>66</td>
<td>132 – 198.9</td>
</tr>
<tr>
<td>300 – 399.9</td>
<td>55</td>
<td>165 – 220.9</td>
</tr>
<tr>
<td>400 – 499.9</td>
<td>44</td>
<td>176 – 220.9</td>
</tr>
<tr>
<td>500 – 699.9</td>
<td>33</td>
<td>165 – 231.9</td>
</tr>
<tr>
<td>700 –</td>
<td>22</td>
<td>154</td>
</tr>
</tbody>
</table>

Figure 1: Relationship between measured global and diffuse solar radiation from the Swedish city Växjö 1996 and estimated diffuse solar radiation using the radiation and percentages in table 1.

2.3 Method for creating long wave radiation - Clearness index method

Presented method was called the clearness index method and is a relatively simple method for calculating the long wave radiation from the sky. The long wave radiation from the sky ($L_w$) is typically measured in W/m² or Wh/m³h. The measuring instrument is some kind of pyrgeometer, e.g. a Hukseflux IR02 [16]. To make it clear that this radiation does not originate from the sun it is sometimes called the atmospheric long wave radiation. The long wave radiation is of the order
200-400 W/m² [17, 18] and varies on a daily and seasonal basis. The long wave radiation is seldom measured and must therefore be estimated based on other parameters. The most important parameters are the outdoor temperature, the RH and the cloudiness of the sky [19]. Many formulas exist and only a brief description of the chosen methods and formulas will be given. When formulating algorithms based on other meteorological data it is natural to start with Stefan-Boltzmann equation for the thermal radiation from a surface with the temperature $T$ (K):

$$ L_w = \varepsilon \cdot \sigma \cdot T^4 $$  \hspace{1cm} (1)

where $\varepsilon$ is the emissivity of the surface and $\sigma$ is the Stefan-Boltzmann constant. Many authors have formulated algorithms for $L_w$ mainly based on:

- The outdoor temperature: $T_o$ (K)
- The moisture content in air described by: outdoor vapor pressure $p_o$ (kPa) or dewpoint $T_d$ (K) or perceptible water $w$ (mm)
- Some kind of cloudiness index: $c_i$ (-)
- The atmospheric pressure at the ground (more seldom).

Presented method is based on the formula above (1). The outdoor temperature is used with an estimation of the sky emissivity. The emissivity is calculated from hourly values of outdoor and dew point temperature together with a 24 hours average calculated clearness index of the sky:

$$ \varepsilon_{sky} = 1.5357 + 0.5981 \left( \frac{T_d}{100} \right) - 0.5687 \left( \frac{T_o}{273.15} \right) - 0.2799 K_0 $$  \hspace{1cm} (2)

$$ L_w = \varepsilon_{sky} \cdot \sigma \cdot T_o^4 $$  \hspace{1cm} (3)

Parameters were estimated from 9 years hourly data from four Swedish cities [19]. Clearness index $K_0$ (-) was defined as quota between measured global solar radiation on a horizontal surface on ground $I_G$ (Wh/m²h) and extra-terrestrial solar radiation $H_0$, calculated over 24h average data.

$$ K_0 = \frac{I_G}{H_0} $$  \hspace{1cm} (4)

$H_0$ is the daily average extraterrestrial radiation in (Wh/m²h) [20]:

$$ H_0 = \frac{I_{SC}}{\pi} \cdot \left[ 1 + 0.033 \cos \left( 2\pi \frac{d_n}{365} \right) \right] \times (\cos(\varphi) \cos(\delta) \sin(\omega_s) + \omega_s \sin(\varphi) \sin(\delta)) $$  \hspace{1cm} (5)

where $\varphi$ (rad) is the latitude, $\delta$ (rad) is the solar declination, $\omega_s$ (rad) is the sunset hour angle, $I_{SC}$ is the solar constant (1367 Wh/m²h) and $d_n$ is the day number (1-365).

$$ \delta = 0.4093 \cdot \sin \left( 2\pi \frac{2844 + d_n}{365} \right) $$  \hspace{1cm} (6)

$$ \omega_s = \cos^{-1} \left( -\tan(\varphi) \tan(\delta) \right) $$  \hspace{1cm} (7)

3 MATERIALS – CASE STUDY TEST

Hygrothermal calculations with default and real supplemented exterior climate and different climate radiation models and comparisons to measured values were carried out in a wood framed roof construction. Studied position was located on the inside of the tongued and grooved wood in a 500 mm wide cold attic space. The house is a single family house located in the Swedish municipality Upplands-Bro 40 km west of Stockholm. A brief description of the cold attic with studied position and used calculation model [11, 12] are presented in Figure 2.
Roof from the outside:
Tiles, part of the outer Sd value
1 mm PVC roof membrane, Sd = 15 m
22 mm Massive wood – Spruce radial
70 mm Air gap
50 mm Air gap
70 mm Air gap
460 mm Mineral insulation, $\lambda = 0.037$ W/mK, including roof beams
1 mm Vapor retarder, SD = 50 m
30 mm Air gap, no ACH
13 mm Gypsum board

Figure 2: Drawing and WUFI calculation model showing the studied position [11, 12].

4 RESULTS

Five different cases with calculated temperature and RH were compared to measured values. The results from each case were analysed and also compared with each other in Folos 2D visual mold chart. Vertical lines on the top indicate a short period with replaced climate boundary data and horizontal lines indicates longer periods with lack of climate boundary data [11]. Specific replaced climate boundary condition for each hour was presented by in a separate report [12].

Figure 3 presents a comparison between measured and calculated temperature and RH using default climate boundary conditions [10]. The cases are: with and without explicit long wave radiation balance in WUFI 5.2.

Figure 3: Comparisons between measured (black) and calculated temperature and RH using default climate boundary conditions and with (turquoise) and without (red) explicit long wave radiation balance in WUFI 5.2. RH_{crit} (green) dependent on calculated temperature including explicit long wave radiation.

Figure 4 presents a comparison between measured and calculated temperature and RH using real, but supplementary, climate boundary conditions and invented diffuse solar radiation. The cases were: with and without explicit long wave radiation balance in WUFI 5.2.
Figure 4: Comparisons between measured (black) and calculated temperature and RH using real, but supplementary, climate boundary conditions and invented diffuse solar radiation and with (turquoise) and without (red) explicit long wave radiation balance in WUFI 5.2. RH_{crit} (green) dependent on calculated temperature including explicit long wave radiation.

Figure 5 presents a comparison between measured and calculated temperature and RH using real, but supplementary, climate boundary conditions and invented diffuse solar radiation with above presented Clearness Index radiation method (CI rad) calculating long wave radiation.

Figure 5: Comparisons between measured (black) and calculated temperature and RH using real, but supplementary, climate boundary conditions and invented diffuse solar radiation and using WUFI 5.2 explicit radiation balance (turquoise) and invented long wave radiation, CI rad, method (red). RH_{crit} (green) dependent on calculated temperature for WUFI 5.2 explicit radiation balance.
5 ANALYSIS AND DISCUSSION

As expected, calculated values using WUFI 5.2 default climate boundary conditions do not correlate to measured values, as shown in figure 3. Although there are clear deviations between measured and calculated values in June and July, as shown in figure 3, greater deviation between measured and calculated values were expected. This since previous research shows higher differences between different years [3, 11]. Other studies show that the Swedish default boundary conditions in WUFI 5.2 seems to report a lower risk of mold growth compare to real climate conditions [21]. The results show a slightly higher amount of risk using WUFI 5.2 default climate boundary conditions compare to measured values. The reason could be that the 2009 exterior climate creates a lower risk of mold growth compared to other years, such as 2011 [11].

In general there was a good correlation between the measured and calculated values when real, but supplemented, climate boundary conditions were used and long wave radiation was included in the calculations, as shown in figure 4 and 5. Detailed analysis indicates that shorter periods with supplemented climate boundary conditions do not highly influence the results. However, previous studies indicate that longer periods of replaced temperature and relative humidity, which highly affect the calculations results compare to other climate parameters, influence the correlation between calculated and real measured values [11].

As expected, calculated values using long wave radiation show higher relative humidity caused by a lower temperature and further on a higher risk of mold growth, as shown in figure 3 and 4. The correlations in length and amount of critical conditions comparing measured and calculated values, including long wave radiation, as shown in figure 4 and 5, show that long wave radiation should be taken into account in the calculations in order to predict the risk of mold and moisture related damages. The correlation between measured and calculated values, using two different methods for creating long wave radiation, as shown in figure 5, indicate that the clearness index method creates slightly higher RH in studied position during the winter.

All calculations show higher amplitude in temperature and relative humidity in calculated values compare to measured values. The higher amplitude in relative humidity probably depends on the variations in calculated temperature, probably caused since tiles, and its heat capacity, do not become included in the exterior roof assembly in the calculation model.

6 CONCLUSIONS

The results show that WUFI 5.2 could be used in order to make reliable hygrothermal calculations of the climate in attics in wood framed roofs in northern European climates. Real climate boundary conditions must be used in order to reach correlation to measured values. The results indicate that mean climate conditions not could be used without further analysis for dimensional purposes.

Used methods in order to supplement periods with lack of climate data and to create diffuse, direct and long wave radiation were successfully used in order to create climate boundary conditions to be used in the calculations. Long wave radiation has to be included in the climate boundary conditions in the calculations in order to obtain reliable results. Both the Clearness index method and WUFI 5.2 method inventing long wave radiation works in the context.

For both the reconstruction of diffuse and long wave radiation to hourly values the “true” values can never be achieved since the measured data do not have enough information. One can only create reasonable climate files with these methods. The choice of method should also be dependent of how these climate files would be used, i.e. if the aim is calculating a good average value or if the aim is to simulate worst case scenarios.
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REFERENSER
FRAGILITY CURVES FOR THIN-WALLED COLD-FORMED STEEL WALL FRAMES WITH DIFFERENT SHEATHING SYSTEMS AFFECTED BY GROUND SETTLEMENTS DUE TO LAND SUBSIDENCE

Jose A. Ortiz (1), Luis A. Hernandez (1), Martin Hernandez (1), Jesus Pacheco (1), Juan J. Soto (2), Jorge Ramos (1), Roberto Fernandez (1), Miguel A. Soto (1)

(1) Department of Construction and Structures, Autonomous University of Aguascalientes, Aguascalientes, Mexico – jose.ortiz.lozano@gmail.com; laherman@correo.uaa.mx; mhernandez@correo.uaa.mx; geochuy@gmail.com; ing.jorgerrf@gmail.com; robfer77@hotmail.com; miguelso_to_ic@yahoo.com.mx

(2) Department of Electrical and Electronic Engineering, Technical Institute of Aguascalientes, Aguascalientes, Mexico – j2sb@yahoo.com

Abstract

Land subsidence phenomenon due to ground water withdrawal is a current problem in many places around the world. This causes ground differential settlements that affect structures, mainly dwellings and buildings.

This work presents the results about a study regarding the performance of thin-walled cold-formed steel wall frames with different sheathing systems affected by angular distortions simulating ground differential settlements due to land subsidence.

The wall frames are composed by vertical (studs) and horizontal elements (tracks), with different sheathing systems: polystyrene, OSB, gypsum and calcium silicate. By means of experimental testing of wall frames subjected to monotonic lateral loads, the rotational stiffness was obtained for the wall frames with polystyrene.

Likewise the rotational stiffness of the other wall frame systems was calculated based on the data provided by other author’s publications. By means of numerical simulation, all the wall frame systems were modeled in a structural analysis software, calibrating them based on the rotational stiffness.

The moment-rotation curves were calculated for the studs and tracks based on the direct strength method. A non-linear static pull down analysis was performed producing several degrees of angular distortion simulating ground settlements.

With the data acquired fragility curves were calculated according three levels of damage.
1 INTRODUCTION

1.1 Background

In building materials, steel is an excellent option for use due to their structural characteristics such as its high tensile strength, good strength to compression, high efficiency in weight-strength among other qualities. The use of cold formed steel elements in the construction industry has increased significantly in the last decades due to its efficiency, cost, ease of fabrication, high strength-weight ratio. But the elements of cold formed steel, suffer the problem of some buckling modes and its interactions because they are usually slender sections.

One of the principal uses of this type of material is in the construction of structural frames as standard modules, which is common to assemble with rigid edge channel and simple channel in different dimensions and sizes. One advantage of this type of modular system is its versatility to be produced industrially, which directly impacts in construction time and better quality control.

Even cold formed steel frames have been used for decades; their application as main structural load-bearing components of frames is fairly new. As a result, guidelines that address the seismic design of cold formed structures are not yet fully developed and the standards and specifications relating to the implementation of this system and their behavior under lateral loads continue to be refined (Moghimi, 2009).

The lateral load behavior is analyzed by Push Over Analysis described in provisions of the Applied Technology Center (ATC-40). However, the case of cold formed steel affected by ground settlement and especially the phenomenon of subsidence, have been less studied.

1.2 Land subsidence

The phenomenon of subsidence by groundwater withdrawal is widespread in Mexican territory in recent decades in the valleys where the aquifer is composed of unconsolidated materials such as alluvial deposits, or volcano- sedimentary geologically recent (Garduño et al., 2001), (CENAPRED, 2001), (Arroyo et al., 2004), (Rojas et al., 2002).

When the bedrock aquifer is irregular, subsidence generates faults and fractures on the earth surface damaging existing urban infrastructure (Rojas et al., 2002). Fractures associated to subsidence has been widely studied in different places in the world (UNESCO, 1984), (Borchers, 1998), (USGS, 1995). In Mexico, it is not unknown, mainly alluvial valleys of such as Celaya, Silao, Queretaro, San Luis Potosi, Morelia, Mexico City etc., problems by subsidence and fractures associated with the exploitation of granular aquifer have been reported (Garduño et al., 2001), (CENAPRED, 2001), (Arroyo et al., 2004).

The first surface fractures and faults were reported about 30 years ago in Aguascalientes (Aranda-Gómez, 1989). These authors reported that the cracks began to be noticed in 1981 for damages caused to buildings, also documented the existence of cracks that had vertical movement, which formed an escarp of several tens of centimeters. In their conclusions noted as the immediate cause of the generation of cracks to abatement of the water table, although they noted that the structural geology plays a passive role, suggesting that the orientation of the fractures studied could to coincide with bedrock irregularities of landfill alluvial.

On the other hand (Zermeño et al., 2005) estimated a total regional subsidence 1.31 m in a period of 18 months measured in the city of Aguascalientes, and reports a displacement speed of 11.18 cm per year in a point located on the INEGI facilities in Aguascalientes, which is
monitored with GPS system. Finally, they present two maps one of subsidence measured in the city of Aguascalientes and other of abatement of piezometric level, the maps show a clear correspondence between areas of subsidence and regional abatement cones so they prove that Aguascalientes valley subsidence and cracks are associated with abatement.

2 METHODOLOGY

2.1 Experimental and numerical methodology

In this experimental stage, thin walled cold formed steel frames were used with dimensions of 1600 mm in length and 1500 mm in height (Figure 1), structured with single channel-section elements on 350T125 -33 top and bottom of the frame and vertical elements of channel section with rigid edge 350S162 -33. The separation between Studs was 400 mm center to center. As sheathing material high-density expanded polystyrene 75 mm thick was used. Connections between Studs and Tracks, were made with 20 mm flat head self-drilling screws, applied at each joint. For fastening to the floor anchors type "hold down" were used. Were attached to the frame by means of 38 mm self-drilling screws and anchored to the floor with a 16 mm diameter steel bolt.

By means of a 160 kN hydraulic actuator, connected to a load beam, a monotonic horizontal force on the top were applied, which was gradually increased to reach a target displacement of 151 mm. Measurement instruments were placed at points of interest to evaluate the displacements, using a laser measuring equipment in a total of 20 points uniformly distributed in the area of the frame. The speed average of load application was 8 mm/minute.

The system stiffness values used for the calibration of numerical simulation models were determined using the test data to lateral load on cold formed steel frames. This frames were structured with five vertical studs spaced a distance of 400 mm and horizontally top and bottom tracks. Expanded polystyrene is used mainly as thermo-acoustic insulation, but in the present study, we evaluated the contribution of this material on the structural behaviour of the constructive system.

As a second step of the methodology, numerical analysis was performed to evaluate the behavior of cold formed steel frames to lateral load, in order to calibrate the models in reference to experimental results. Stiffness values for the models considering different types of material were determined. As a third step, numerical analysis was performed to evaluate the behavior of frames with vertical displacement, simulating the effect of differential settlement produced by subsidence in a structure. For the construction of finite element models (FEM), SAP2000 software version 12.0 was used.

Models with different thicknesses, sizes and sheathing materials were generated. Numerical simulation models were obtained based on data from experimental testing and the information provided from others researchers (Xuhong, 2006), (Pan, 2011), (Baran, 2012) and (Nithyadharan, 2012); Figure 2 shows graphically the results of all these lateral load tests, including the frame with expanded polystyrene.
In the testing performed by these authors frames with different sheathing materials such as gypsum, calcium silicate and OSB wood, were considered. The FEM used for these simulations were formed using type bar elements for studs and tracks and type area elements for sheathings. Area type elements were discretized in maximum sizes of 10 cm, the connectivity between bar type elements were considered semi rigid and a rotational stiffness spring was assigned whose value by experimental testing, was determined.
2.2 Pull-down analysis

The pull-down effect into a structure is considered when one of the supports suffers a vertical displacement. This type of analysis is similar to that which occurs with nonlinear static analysis known as Push Over. The difference lies in the direction in which the displacements are evaluated. While in Push Over analysis, horizontal displacements are evaluated, in the Pull Down analysis, vertical displacements are reviewed, which can be generated by different causes.

An important feature in this type of vertical settlement is the speed at which these movements will generate because they depend primarily on the type of landslide affecting the structure.

The greatest difficulty in achieving reliable results for landslides in comparison to other natural hazards such as earthquakes or floods is due to the complexity of modelling landslide hazard, in identifying relevant intensity parameters and assessing vulnerability in quantitative manner (Negulescu, 2010).

According to Negulescu, there are three general methods for predicting structural damage due to movements and settlements of the foundation:

- Empirical methods which aim at establishing criteria of serviceability by relating the deformation observed from field surveys to the damage.
- Methods using structural engineering principles.
- Methods based on numerical modelling.

For this study, numerical models of frames were affected by vertical movements applied at one end of the structure, simulating the effect of regional subsidence, which develops slowly through the years. Once constructed, the numerical models were calibrated using the information obtained from the experimental stage carried out on cold formed steel frames. After that models were subjected to the pull-down effect, which consists in applying a vertical displacement simulating the effects produced by subsidence.

From simulation campaign were obtained bending moment values in each of the studs for each level of displacement of different types of frames.

The results were grouped according to the type of sheathing used and filtered moment maximum values for each level of displacement. These values were expressed in relative way taking as reference the plastic moment $M_p$ and registered using a relative moment–distortion graph.

3 RESULTS

3.1 Relative moment

The results regarding experimental tests and the ones obtained by other researchers in frames to lateral load are showed in Figure 3 and can be summarized as follows: from the four materials tested, the polystyrene as sheathing material in frames generates a high ductility system and allows large deformations (angular distortion) with minimal damage in comparison with other systems. It is important to note that to none sheathing material the studs have failure by total plasticizing of the cross section (do not reach $M_p$), since failures first occur due to distortional buckling, which is reflected in the moment-rotation curves of the sections.
It can be noted that the OSB panel frame offers the highest stiffness values than any other, more than ten times the polystyrene which is a condition not favorable if the purpose of the study is to review the capacity of the structure to support differential settlement without fail. The calcium silicate and gypsum frames provide more stiffness than polystyrene. In all cases the studs work until at least 64% $M_p$.

3.2 Fragility curves

For the development of fragility curves on subsidence, the results obtained from the simulations were taken as reference, performed on finite element models for each type of sheathing material considered. The angular distortion associated to plastic moment on frames was considered. Displacement levels applied to the models were 10, 20 and 30 cm which applied instantly at each displacement stage. By varying certain characteristics of the frames without changes to the aspect ratio of the models a statistical database was generated.

For this investigation the Plastic Moment ($M_p$) directly associated to angular distortion was applied as a reference parameter; three damage states related to this parameter were defined, 0.5 $M_p$ for light damage, 0.65$M_p$ for moderate damage and $M_{max}$ for complete damage.

Through statistical analysis of the results, median values and dispersion values were obtained by applying the pull-down procedure to each of the frames in study. Fragility curves for each state of damage and type of sheathing were generated applying fragility functions. The fragility curves for each state of damage considered due to land subsidence are presented in Figure 4.
4 CONCLUSIONS

- In the case of gypsum panel is observed that has a high probability to present moderate damage with only 5% distortion.
- The behavior of calcium silicate panel is more favorable than OSB and gypsum panel, because it accepts greater distortion than the previous materials.
- Although on experimental tests to lateral load, expanded polystyrene frame had the lowest strength and higher ductility compared to the results of other researchers in contrast to the fragility curves the polystyrene offer best performance than the other materials.
- According to the fragility curves of the frame with expanded polystyrene panel this structural system has a good performance against differential settlement and that for a vertical distortion of 19% there is a 50% probability that complete damage occurs, a 80% that moderate damage occurs and 98% probability of occurrence only slight damage in the structure.
- According to experimental evidence, it shows that the polystyrene provides sufficient stiffness to allow the frame has adequate performance to the pull-down effect.
- Cold-formed steel frame walls with polystyrene sheathing would increase service life and durability of dwellings in case of differential settlements due to land subsidence.
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SELF-HEALING OF SELF-COMPACTING CONCRETES MADE WITH BRAZILIAN BLAST FURNACE SLAG CEMENTS ACTIVATED BY CRYSTALLINE CATALYST

Emilio M. Takagi (1), Maryangela G. Lima (2) and Paulo Helene (2)

(1) Department of Aeronautical Infrastructure, Instituto Tecnológico de Aeronáutica, São José dos Campos / MC-Bauchemie Brasil, Vargem Grande Paulista – etakagi@ita.br

(2) Department of Aeronautical Infrastructure, Instituto Tecnológico de Aeronáutica, São José dos Campos – magdlima@ita.br

(3) PhD Engenharia, São Paulo - paulo.helene@concretophd.com.br

Abstract

Different approaches have been developed to study new types of concrete that have the ability to repair cracks. Autogenic self-healing improves the natural healing mechanism of concrete through incorporation of a catalyst that activates material components already present in the concrete. The occurrence of the crack is the trigger mechanism because it allows the penetration of water to activate the crystalline catalyst (Cat-X) and newly fractured sub-hydrated cement and non-activated blast furnace slag (BFS) particles. Test samples were examined using three types of Brazilian commercial cements: blast furnace slag cement (CP III RS), slag modified Portland cement (CP II-E) and Portland cement (CP V ARI) with percentages in the range of 55%, 35% and 0%, respectively. Test specimens were loaded in compression to 90% of failure load, in order to generate a network of micro-cracks. Samples were immersed in water to trigger the self-healing mechanism, followed by various tests after 28, 56 and 84 days. Recovery of mechanical properties and watertightness indicated greater self-healing in the CP III, CP V and CP II cement samples, respectively. As the BFS content ratio was increased to 55%, there was a noticeable increase in the mechanical recovery and permeation reduction properties due to the addition of Cat-X. Therefore, it appears that the specimens with added BFS and Cat-X are less affected by mechanical loads.

Keywords: self-healing concrete; self-compacting concrete; blast furnace slag; crystalline catalyst.
1 INTRODUCTION

Concrete is a material with a long history of use as a building material; its importance will continually require more research to solve the almost unavoidable issue of cracking in reinforced concrete. Research has focused on a scientific approach to the casual observations of self-healing cracks in concrete—a phenomenon that has been well-known since ancient times. The goal is the development of a self-compacting concrete with self-healing capabilities that enables the design of more durable concrete structures subjected to water exposure.

Self-compacting concrete (SCC) is one of the latest achievements in concrete technology. However, in comparison to conventional concretes, SCCs show much higher levels of thermal and autogenous shrinkage; these high strength SCCs are far more likely to crack at an early age than normal strength concretes. Investigations into the self-healing properties of SCCs subjected to mechanical loading led to the development of further hydration processes for unhydrated cementitious particles when exposed to water [1]. In subsequent investigations of the self-healing capabilities of cementitious composites incorporating different supplementary cementitious materials (SCM), a more evident self-healing product was observed from mixtures incorporating blast furnace slag (BFS) [2, 3]. Moreover, the high surface area of BFS provides more nucleating sites, as well as OH⁻ ions and alkalis for the pore fluid when BFS is hydrated [4]. The hydration products of Portland-slag cements are the same as Portland cement, except that smaller quantities of calcium hydroxide (Ca(OH)₂) are found at higher slag contents. The lower Ca(OH)₂ content and the finer pore structure of BFS concrete could therefore contribute to its greater chemical resistance [5].

Crystalline catalyst (Cat-X) is a synthetic cementitious material classified as a hydrophilic waterproofing and as a permeability reducing admixture hydrostatic according to ACI 212.3R-10. Its reactive components react with Ca(OH)₂ to form crystalline products that disconnect pores and fill cracks in the concrete. The crystalline products can only occur when sufficient moisture is present [6]. By means of diffusion, the reactive chemicals in Cat-X use water as a migrating medium to enter and travel down the capillaries of the concrete. This process precipitates a chemical reaction between Cat-X, moisture and the by-products of cement hydration, forming a new non-soluble crystalline structure, including apatite and enstatite crystals. This integral structure fills the capillary tracts rendering the concrete waterproof. Cat-X has many physical and chemical properties that are surprisingly similar to those of clay minerals. The high cation exchange capacity and the intercalation of carboxylic acids (-COOH) into the Cat-X structure makes their interlayer sodium ion (Na⁺) exchange through calcium ions (Ca²⁺) versatile and easily achieved. Cat-X has been extensively studied for the suppression and control of alkali-aggregate reactions (AAR) in concrete through the catalysed alkali discharge resulting from its chelating function [7-9]. This water stopping technique was used to improve the impermeability property of the concrete and decrease the migration of radionuclides (¹³⁷Cesium) during the treatment, storage and disposal of low level radio-active waste in Japan and was also used for waterproofing the bridges and tunnels of the Japan Railways Shinkansen bullet train lines [10].

The effect of water exposure conditions plays an important role in self-healing efficiency and the types of healing products formed. The exposure conditions of wet/dry cycles show that, in an optimal mechanical recovery, a mixture of calcium carbonate (CaCO₃), calcium silicate hydrate (C–S–H) and ettringite are found as major internal crack healing products.
Continuous water exposure leads to the formation of CaCO$_3$ on the crack mouth, which is preferable in terms of water tightness; however, this formation decreases the recovery of mechanical properties [11].

Therefore, the following mechanisms must be considered for a robust autogenic self-healing concrete: (a) A mechanism for continuous hydration with the use of cement composed of large amounts of BFS; (b) A mechanism of chemical healing with a dosage of Cat-X to cause a cementitious recrystallization effect in the concrete cracks; and (c) A mechanism of crack restriction with the addition of ductile type alkali-resistant (AR) glass fibers to enhance the cementitious matrix [12]. Multiple cracking with relatively small crack widths (in comparison to ordinary cracked concrete) generally results in the increased resistance of this material to the ingress of fluids and gases [13]. The proposed self-healing mechanisms have the added advantage that a second healing action may occur and will be easily implemented into concrete structures that are not easily accessible for maintenance and repair, such as underground structures, bridges, and dams. Although the initial costs will be higher, the maintenance costs can be reduced, and the service life of the structures may be extended, as damage is immediately repaired [14].

2 EXPERIMENTAL PROGRAM

In this program, the experiments performed on hardened concrete can be grouped into tests to determine the mechanical properties and tests to determine the permeation properties. Concrete specimens for self-healing tests were loaded to 90% of their compressive strength at 28 days age. Mechanical properties, including compressive strength and ultrasonic pulse velocity (UPV) were determined at 28 days, and then after 28 and 56 days of lime water curing following pre-loading by using three specimens at each age. The permeation properties were also determined at the same intervals by using water sorptivity and rapid chloride permeability (RCPT), in accordance with the ASTM C1585 and ASTM C1202 standards, respectively.

2.1 Material properties

Three types of Brazilian commercial cements with distinct percentages of blast furnace slag (BFS) ranging from 0 to 55%, were used in all mixtures: The CP III 40 RS cement (slag content < 55%) is equivalent to ASTM Type IS (MS) blast-furnace slag cement. The CP II-E 40 cement (slag content < 34%) is equivalent to ASTM Type I (SM) slag-modified Portland cement. The CP V 42.5 ARI cement is equivalent to ASTM type III high early strength Portland cement. A specific crystalline catalyst (Cat-X) was used in this study at the rate of 2.5% by weight of cement content.

2.2 SCC mix design, preparation and casting

Six SCC concrete mixtures were prepared with the proportions summarized in Table 1 for all three types of cements, with and without the addition of the crystalline catalyst. The total cement content and the water/cement ratio (w/c) of 0.5 (by mass) were kept constant. The crystalline catalyst was not considered as a binder. SP was added to achieve an initial slump of 80mm; then PCE was added to get a slump flow of more than 700 mm for all mixtures. The SP content was not kept constant, though the PCE content remained constant.
Table 1 - SCC mix design (kg perm$^3$)

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Quantities</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>196 kg</td>
<td>w/c ratio = 0.5</td>
</tr>
<tr>
<td>Cement</td>
<td>392 kg</td>
<td>CPIII / CPII-E / CPV</td>
</tr>
<tr>
<td>Crystalline catalyst</td>
<td>10 kg</td>
<td>2.5% of cement content</td>
</tr>
<tr>
<td>Quartz sand</td>
<td>573 kg</td>
<td>70% of fine aggregate</td>
</tr>
<tr>
<td>Crushed stone sand</td>
<td>246 kg</td>
<td>30% of fine aggregate</td>
</tr>
<tr>
<td>Crushed stone 9.5 mm</td>
<td>651 kg</td>
<td>70% of coarse aggregate</td>
</tr>
<tr>
<td>Crushed stone 25 mm</td>
<td>279 kg</td>
<td>30% of coarse aggregate</td>
</tr>
<tr>
<td>SP superplasticizer</td>
<td>2.4 kg</td>
<td>0.6% of cement content</td>
</tr>
<tr>
<td>PCE superplasticizer</td>
<td>4.0 kg</td>
<td>1.0% of cement content</td>
</tr>
<tr>
<td>Viscosity modifier</td>
<td>2.0 kg</td>
<td>0.5% of cement content</td>
</tr>
<tr>
<td>AR glass fiber</td>
<td>0.9 kg</td>
<td>Alkali-resistant 12 mm</td>
</tr>
</tbody>
</table>

Visual inspection of fresh concrete did not detect any segregation or bleeding in any of the mixtures during the tests. From each concrete mixture, Ø100 x 200 mm cylinder specimens were prepared for the mechanical and permeation tests. All specimens were cast in a single layer without any compaction. At 24 hr, the specimens were removed from the molds and stored in lime-saturated water at 23 ± 2° C for 28 days. To test the permeation properties of the pre-cracked concrete specimens, the cylinders were sawed into 50 mm thick slices, and the two middle slices from each specimen were used for testing.

2.3 Compressive strength recovery

After 28 days of moist curing, the compressive strength of each mixture was determined, and the remaining specimens were pre-loaded to 90% of their corresponding compressive strength. The pre-loaded specimens were then further stored in lime-saturated water at 23 ± 2° C for an additional 28 days and 56 days, respectively. The mechanical properties of the compressive strength of the concrete specimens before and after preloading load them to 90%, stop, then reload them to failure at 28 days.

2.4 Sorptivity test

The sorptivity test method is used to determine the rate of absorption of water in the concrete by measuring the increase in the mass of a pre-dried specimen resulting as a function of time, when only one surface of the specimen is exposed to water. The exposed surface of the specimen is immersed in water and the water ingress of the unsaturated concrete is dominated by capillary suction during the initial contact with the water.

2.5 Rapid chloride permeability test

The rapid chloride permeability test (RCPT) is a measure of the concrete’s resistivity which is an indirect measure of chloride penetrability. In this test, a 50 mm thick, 100 mm diameter, water saturated concrete specimen was subjected to a 60 V applied DC voltage for 6 h. One end of the specimen is in contact with a 0.3 M NaOH solution, while the other end is in contact with a 3.0% NaCl solution. This amount, determined in coulombs, is related to the concrete’s ability to resist chloride ion penetration through the fissured (F) specimens at 28 days and the fissured and cicatrized (FC) specimens (28+28 days) for all mixtures.
3 RESULTS AND DISCUSSION

3.1 Unhealed fissured specimens

The undamaged control specimens had a higher compressive strength comparing the properties of the fissured specimens. For this reason, the extent of any self-healing is best detected by comparing the results of fissured and undamaged specimens’ properties at a particular age.

Because these fissured specimens were tested immediately after the release of the pre-loading, they had no time to undergo any crack healing. The results in Table 6 reveal that, for the unhealed specimens, the compressive strength, ultrasonic pulse velocity (UPV), water absorption rate and rapid chloride permeability test (RCPT) were affected by the crystalline catalyst (Cat-X) and blast furnace slag (BFS) content in the commercial cement.

Table 2 - Properties of unhealed fissured specimens

<table>
<thead>
<tr>
<th>Mix</th>
<th>Strength</th>
<th>UPV</th>
<th>Absorption</th>
<th>RCPT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>50.6 MPa</td>
<td>4514 m/s</td>
<td>0.0243 mm</td>
<td>1764 C</td>
</tr>
<tr>
<td>2</td>
<td>44.2 Mpa</td>
<td>4413 m/s</td>
<td>0.0309 mm</td>
<td>1860 C</td>
</tr>
<tr>
<td>3</td>
<td>43.3 Mpa</td>
<td>4566 m/s</td>
<td>0.0326 mm</td>
<td>3927 C</td>
</tr>
<tr>
<td>4</td>
<td>40.7 Mpa</td>
<td>4247 m/s</td>
<td>0.0407 mm</td>
<td>4023 C</td>
</tr>
<tr>
<td>5</td>
<td>53.7 Mpa</td>
<td>4565 m/s</td>
<td>0.0327 mm</td>
<td>5139 C</td>
</tr>
<tr>
<td>6</td>
<td>52.6 Mpa</td>
<td>4679 m/s</td>
<td>0.0406 mm</td>
<td>5262 C</td>
</tr>
</tbody>
</table>

The Cat-X enhanced the compressive strength of the unhealed fissured specimens by 14% on CPIII, 6% on CPII and 2% on CPV. Therefore, it appears that the specimens with the addition of BFS and Cat-X are less affected by mechanical loads. The water absorption rate was reduced by 21.4% on CPIII, 19.9% on CPII and 19.5% on CPV. In addition, the RCPT values reduced by 5.2% on CPIII, 2.4% on CPII and 2.3% on CPV. However, an increase in BFS content from 34% to 55% showed no significant effect on the permeation reduction effect. The measurable damage reduction was least clear in the ultrasonic pulse velocity (UPV).

The use of supplementary cementitious materials (SCM) such as BFS may have a significant effect on the permeability of concrete as measured by the RCPT. While the active component in cement is a hydraulic binder, blast furnace slag also has latent hydraulic properties, this means that the hydration reaction with water must be activated by substances such as Ca(OH)$_2$, e.g., from clinker hydration. However, it is known that currently used SCMs such as BFS react more slowly compared to cement [15].

3.2 Effects of self-healing

Repairing cracks in mechanically loaded reinforced concrete elements has been a long-time goal. The conventional repair methods either physically block off water by applying a waterproofing material with crack-bridging abilities or inject low viscosity resins into the cracks. By contrast, self-healing concretes (SHC) can waterproof the entire concrete structure—as well as the cracks—with the application of a crystalline waterproofing catalyst [16]. Table 3 presents the results of compressive strength, UPV, sorptivity and RCPT tests from specimens that were subjected to preloading and later stored for up to 56 days in water.
to evaluate the extent of self-healing. Autogenous self-healing can reduce the water permeability of cracked specimens.

Table 3 - Properties of fissured and cicatrized specimens

<table>
<thead>
<tr>
<th>Mix</th>
<th>Strength</th>
<th>UPV</th>
<th>Absorption</th>
<th>RCPT</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>58.8 MPa</td>
<td>4540 m/s</td>
<td>0.0182 mm</td>
<td>1086 C</td>
</tr>
<tr>
<td>2</td>
<td>55.5 Mpa</td>
<td>4646 m/s</td>
<td>0.0204 mm</td>
<td>1170 C</td>
</tr>
<tr>
<td>3</td>
<td>47.7 Mpa</td>
<td>4566 m/s</td>
<td>0.0267 mm</td>
<td>3108 C</td>
</tr>
<tr>
<td>4</td>
<td>45.1 Mpa</td>
<td>4665 m/s</td>
<td>0.0273 mm</td>
<td>3225 C</td>
</tr>
<tr>
<td>5</td>
<td>55.7 Mpa</td>
<td>4492 m/s</td>
<td>0.0274 mm</td>
<td>2451 C</td>
</tr>
<tr>
<td>6</td>
<td>53.7 Mpa</td>
<td>4494 m/s</td>
<td>0.0265 mm</td>
<td>2658 C</td>
</tr>
</tbody>
</table>

Cat-X enhanced the compressive strength of the fissured and cicatrized specimens by 5.9% on CPIII, 5.8% on CPII and 3.7% on CPV. The water absorption rate reduced by 10.8% on CPIII and 2.2% on CPII, but increased by 3.4% on CPV. In the RCPT, the values reduced by 7.2% on CPIII, 3.6% on CPII and 7.8% on CPV. These values show that the self-healing effect depends on the type of cementitious materials used, the BFS content and the addition of Cat-X.

By comparing the plot of the water absorption of the healed specimens stored in water as the BFS incorporation level increased up to 55% with Cat-X (as shown in Figure 2), it can be observed that a significant reduction of cumulative water absorption with the square root of time has occurred. However, the healed specimens without Cat-X show a higher recovery percentage. This suggests that the self-healing effect also depends on the exposure conditions to water and air. One can hypothesize that, during the drying phase, the excess water has evaporated; therefore, the ion concentrations in the water present in the cracks have increased. In this situation, the amounts of reactants available for further reactions are considerably concentrated, while the amount of water was also sufficient for through-solution reactions. This phenomenon should enhance chemical reactions, precipitation and further hydration. Moreover, the penetration of CO$_2$ into the crack during the drying period would lead to the additional formation of carbonates that are helpful for sealing the cracks, as observed by others [11].

As the BFS incorporation level increased up to 55%, the compressive strength reductions were more visible in the 56 day samples. At 90% pre-loading, the recovery amounts reduced from 29.6% to 18.4% in CPIII, 14.3% to 13.6% in CPII and 4.0% to 3.9% in CPV. Based on the previous discussions, it was observed that, in the SCC mixtures with BFS, the unhydrated BFS material available for further hydration was also greater. Therefore, it appears that the BFS significantly influences the self-healing of the mechanically pre-loaded specimens; even after 56 days, moist curing is critical to long term self-healing. The C–S–H gels formed through the activation of the BFS reactions developed a good bond within the micro-cracks. The measurable damage and healing was the least clear with the ultrasonic pulse velocity (UPV).

Similar conclusions can also be made for the RCPT. However, it should be noted that, among the effects of self-healing, the reductions were more visible in the 28-day healing window. The reduction amounts were 52% to 49% for the CPV, 38% to 37% for the CPIII and 21% to 20% for the CPII-E. The effects of self-healing are more visible on the permeation properties when compared to the mechanical properties. Therefore, in looking at the RCPT reductions presented in Table 11, it can be observed that the results of tests such as the
recovery of the mechanical properties and watertightness show greater self-healing in the samples with CP III, CP V and CP II cement, respectively.

Pre-loading the concrete caused an increase in its total porosity and a loss in its ultimate compressive strength. As micro-cracks developed inside the concrete structure, the pore structure was modified and the continuity of the cracks was increased. Internal cracking due to the mechanical loading initially reduced the compressive strength from 5.1 ~ 18.9% when pre-loaded up to 90% of ultimate compressive strength; after 56 days, recovery was 3.9 ~ 29.6%. This was because the high volume of BFS, a hydraulic latent material, had a significant amount of unhydrated particles available in its microstructures; this contributed to the self-healing effect of the pre-existing cracks activated by the crystalline catalysts. The recovery of the compressive strength and permeation properties can be related to the progressive filling of cracks by the newly formed C-S-H gels due to their self-healing effects [17]. Slag cement and slag-modified concretes with addition of crystalline catalyst benefitted from prolonged limewater curing and show more significant reductions in water absorption rates than the control mixes. Ongoing chemical reactions between the crystalline catalyst and the cement by-products could be promoted by these curing conditions resulting in reduced volumes of permeable voids.

4 CONCLUSIONS

- As the BFS content ratio was increased to 55%, there was a noticeable increase in the mechanical recovery and permeation reduction properties due to the addition of Cat-X. Therefore, it appears that the specimens with added BFS and Cat-X are less affected by mechanical loads.

- Slag cement and slag-modified concretes with the addition of crystalline catalyst benefitted from prolonged limewater curing and show more significant reductions in water absorption rates than the control mixes.

- The recovery of the mechanical properties and watertightness indicate greater self-healing in the samples with CP III, CP V and CP II cement, respectively.

- If there is a tendency for a slower self-healing in samples with BFS (CP III and CP II) at an early age than compared with the samples with 100% Portland cement (CP V), the latent hydraulic behavior of the BFS is critical to its long-term self-healing.

- It was clear that the crystalline catalyst was effective in improving the mechanical and permeation properties of the SCC stressed by mechanical load. It is necessary a minimum dosage of 6 kg to 10 kg/m$^3$ (or 3 kg to 5 kg/m$^3$ in concentrated form) for a self-healing effect.

Exposure conditions play an important role in the self-healing process; in general, wet/dry conditions is recommended for the optimal mechanical recovery. For developing techniques that can assure long-term durability for concrete structures when subjected to continuous water exposure, is recommended improving with alternative binders such as blast furnace slag (BFS), silica fume and crystalline catalyst (Cat-X$^*$).

$^*$ Xypex Admix C-500
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DURABILITY ASSESSMENT OF INNOVATIVE BUILDING PRODUCTS IN BRAZIL: EXAMPLE OF PVC COMPONENTS USED AS STAY-IN-PLACE FORMS AND FINISHING OF CONCRETE WALLS


(1) PhD, Research Engineer, Institute of Technological Research of State of São Paulo – IPT, Building Technological Center – CETAC, luciana@ipt.br *corresponding author
(2) Research Engineer, of Braskem S.A, antonio.rodolfo@braskem.com
(3) Research Engineer, Institute of Technological Research of State of São Paulo – IPT, Building Technological Center – CETAC, jhfonte@ipt.br
(4) PhD, Research Engineer, Institute of Technological Research of State of São Paulo – IPT, Building Technological Center – CETAC, claumit@ipt.br

Abstract
In Brazil, innovative building products are described as those for which prescriptive technical standards do not yet exist. These products need to be evaluated by a technical institute accredited to participate in the National System of Technical Assessment of Innovative Building Products (SiNAT). This is the case even if such products have been employed in other countries, since Brazilian conditions must also be considered. This paper aims to demonstrate an approach to performing a technical assessment of the durability of innovative building products, considering Brazilian exposure conditions. The example used consists of PVC components used as stay-in-place forms and finishing of concrete walls. The paper is based on researches conducted by CETAC/IPT supported by some companies. The following tests were carried out: hygrothermal shock (heat and rain) test to evaluate the durability of a wall with PVC finishing; mechanical performance of the PVC components both before and after accelerated ageing by UV exposure; and natural ageing and accelerated ageing trials to evaluate the color change of the PVC finishing. The approach presented for a durability assessment was shown to be adequate and the tests results were satisfactory. Further research considering longer natural exposure and also the application of PVC as finishing in a real building in normal use is still necessary.

Key-words: innovative building products, durability requirements, technical performance assessment, PVC components.

1 INTRODUCTION
In Brazil, innovative building products are described as those for which prescriptive technical standards do not yet exist. These products need to be evaluated by a Technical Institute accredited to participate in the National System of Technical Assessment of Innovative Building Products (SiNAT), under the Brazilian Program of Building Productivity and Quality (PBQP-H). This is the case even if such products have been employed in others countries, since Brazilian conditions must also be considered.
Because Brazil is a tropical country some degradation agents may be more significant than in the USA or Europe, specifically high temperature, humidity, ultraviolet radiation, biological agents (Arnoult et al., 1995[1]). For example, in São Paulo, Brazil, the annual average maximum and minimum temperatures are around 24.9°C and 15.5 °C, respectively, with a temperature variation around 15 °C. The average precipitation is 1200mm, with relative humidity of the air around 80% (<INMET[2]). By contrast, Paris, France, for example, has an annual average maximum temperature of around 15°C and minimum of 8°C and precipitation of 630 mm, with relative humidity around 70% (data from <http://www.meteo-paris.com/ile-de-france/climat.html>[3]).

In this context, this paper aims to demonstrate an approach of performing a technical assessment of the durability of innovative building products, considering Brazilian exposure conditions. The example used for this technical assessment was the use of PVC components as stay-in-place forms and finishing of concrete walls.

Lewry; Crewdson (1994)[4] give four approaches for testing the durability of a material, component or constructive system: (a) benchmark tests; (b) reference materials/comparative testing; (c) environmental/stress testing, with accelerate ageing; (d) site testing. When an innovative product is considered, a comprehensive testing approach is required, including site and environmental/stress testing to give assurance of performance in service. The approach chosen should be based on: (a) consideration of the factors which might affect durability; (b) accelerated ageing trials to determine the most significant factors causing degradation and the mechanisms of that degradation; (c) evaluation of the significant causes of degradation in the proposed use environment; (d) construction of models and damage functions for the response of the material to the environment, based on the accelerated ageing test results and the environmental measurements. For ageing studies, John; Sato (2006)[5] proposed the following strategies: accelerated ageing tests, natural ageing tests and site testing (full scale and real use conditions).

In this paper, the approaches discussed by Lewry; Crewdson (1994)[4] and John; Sato (2006)[5] were considered. These premises are contained in the SiNAT Technical Guidelines which have been developed to standardize technical assessment of innovative building products in Brazil. The SiNAT Guideline 004 (MINISTERIO DAS CIDADES, 2010)[6] deals with the use of PVC components as stay-in-place forms and finishing of concrete walls. There are now thirteen technical guidelines available on the SINAT/PBPQ-H site (http://www.cidades.gov.br/pbqph/projetos_sinan.php).

According to CIB Report: Publication 331(2010)[7] and to SiNAT Technical Guidelines, the durability assessment of constructive system must consider both the isolated components and the full scale integrated systems and both the component and the system must be submitted to natural and artificial ageing tests. Also, the European guidelines for technical approval, ETAG 004[8], establishes that all components shall retain their properties during the overall service life of the system. The service life for the PVC wall (structural wall) is 50 years – the minimum value set by the Brazilian standard, ABNT NBR 15.575-1 (2013).[9]

For PVC components, the main factors influencing degradation include oxygen, humidity, mechanical stress and ultraviolet radiation, all accelerated by higher temperatures (Rodolfo Jr.; Mei, 2007[10] and Jakubowicz, et al., 1999[11]).

The present paper is divided into four parts: 1) Brief description of the wall system formed by PVC components used as stay-in-place forms and finishing of concrete walls; 2) Measurement methods and performance criteria; 3) Results and discussion; 4) Conclusion.
2 BRIEF DESCRIPTION OF THE PVC WALL SYSTEM

The system discussed in this paper is for one floor isolated (detached) and twinned (semi-detached) houses (see Figure 1) and it was evaluated under SiNAT Guidelines as published in DATec 17 (MINISTÉRIO DAS CIDADES, 2013). The walls were formed by PVC panels filled with concrete and had a total thickness of 80mm. The PVC panels had the function of form during the concreting process and also of wall finishing during the lifetime of the houses. These panels were formed by binding two PVC profiles with a coupling brace (see Figure 2). A basic PVC profile has a width of 200 mm.

The concrete used to fill the form had a compression characteristic strength of 25MPa and 23±1 cm in a slump flow test. The anchoring between the wall and foundation was made with steel bars of 10mm diameter (CA 50) each 600mm fixed in place with epoxy resin. Horizontal reinforcement was also provided by steel bars of 10mm diameter, in the regions of window and door spans and end walls.

3 METHODS AND PERFORMANCE CRITERIA

Tests to evaluate the mechanical strength of PVC components, before and after accelerated ageing tests were made. Durability tests on the wall system, considering the degradation by thermal shock, and by natural weathering exposure were also made.

The color stability of PVC was analyzed by comparing the results of natural ageing test (completed on the wall system) with the results of artificial accelerated ageing tests in Weather-O-Meter apparatus (with PVC samples).

For assessing the reduction of PVC mechanical strength after accelerated ageing test, a QUV apparatus it was used. This test method is considered to be the best way to take into account the influence of solar radiation on mechanical characteristics (Hachich, 1999). However, as Hachich (1999) and others, the color stability of PVC finishing cannot be evaluated in a QUV apparatus, because the amount of UV radiation is higher than the radiation in the stratosphere. Therefore, a Weather-O-Meter apparatus was used, as, in this device the radiation is closer to real conditions since the xenon-arc lamp simulates three wavelength radiation (UV-A and UV-B, visible light and infrared). In addition one PVC wall of 1,20m x 2,60m was exposed to natural weathering conditions in order to assess the color stability of PVC finishing. Another test using a sample of PVC was exposed to artificial
weathering in a Weather-O-Meter apparatus. An attempt was made to obtain an estimate of the correlation between the two methods, but was not adequately successful.

The thermal shock test, in which the surface temperature of the wall is elevated and then rapidly cooled, is usually performed at 80°C; as set down in ABNT NBR 15.575-4 (2013) \(^{14}\). However, as the properties of PVC can begin to change with temperatures above 80º C (Real; Gardette, 2001) \(^{15}\), the test is done with maximum surface temperature of 60º C. The PVC panels used as wall finishing have to be light-colored (absorptance (\(\alpha\)) ≤ 0.6) in order to avoid elevated surface temperatures.

### 3.1 Performance Criteria and Test Methods - durability of the PVC component

Table 1 shows those performance requirements and criteria for durability of PVC components, as defined in SiNAT Guideline 004, that were considered, by the authors of this paper, to be essential for PVC component assessment.

<table>
<thead>
<tr>
<th>Table 1 – Requirements, criteria and test methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>Item</td>
</tr>
<tr>
<td>------</td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>5</td>
</tr>
</tbody>
</table>

The color variation was measured by the tone deviation between the surface of the PVC sample before and after ageing, for each pre-defined period of exposure. By International Convention and Brazilian standard ABNR NBR 15077 (2004) \(^{22}\), the colors are defined in terms of three color coordinates "L", "a" and "b", represented on orthogonal axes. The “L”

\(^{1}\) The tensile impact test is performed only when the thickness of PVC profile is less than 1.60 mm, because in such cases it is not technically possible to perform the Charpy test.
coordinate indicates the direction from light to dark, “a” indicate the chromaticity from green (negative value) to red (positive value) and "b" from blue (negative value) to yellow (positive value). The reading of the parameters "L", "a" and "b" is performed using a colorimetric spectrometer template "Color Guide Sphere d/8 Spin", with D65 standard observer and 10° open angle. The difference of color coordinates expressed by \( \Delta E \) is obtained mathematically by extracting the square root of the sum of the differences of color coordinates squares (\( \Delta L \), \( \Delta a \), and \( \Delta b \)) as formula 1.

\[
\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2}
\]

Formula 1 - \( \Delta E \) calculation

The criteria adopted for evaluation the color variation are the correlation with the perception by the human eye, using the scale applied by color printing industry (table 2).

<table>
<thead>
<tr>
<th>Difference</th>
<th>Perception of color difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;0,2</td>
<td>Not perceptible</td>
</tr>
<tr>
<td>0,2 a 0,5</td>
<td>Threshold of perceptibility</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Difference</th>
<th>Perception of color difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>0,5 a 1,5</td>
<td>Weak, but perceptible</td>
</tr>
<tr>
<td>1,5 a 3,0</td>
<td>Perceptible</td>
</tr>
<tr>
<td>3,0 a 6,0</td>
<td>Highly perceptible</td>
</tr>
<tr>
<td>6,0 a 12,0</td>
<td>Strongly perceptible</td>
</tr>
</tbody>
</table>

3.2 Performance Criteria and Test Method - durability of PVC finishing wall (system)

The performance of the wall system, which includes the PVC finishing, was assessed by testing the wall resistance to natural ageing and also to heat and thermal shock. In addition, the impact resistance of PVC finishing was evaluated by testing the hard-body impact after testing the wall to heat and thermal shock.

- PVC wall resistance to natural ageing exposure

A sample of wall with PVC finishing was exposed to natural weathering conditions. This sample was 1.20 m wide and 2.6 m high and was set up in an experimental campus located at IPT-São Paulo, at latitude 23°30’S. One face of the wall faced Northeast (45° from North). The solar radiation incident on the face of the specimen was estimated considering the orientation and location of its surface (FROTA, 2004). The radiation on the exposed face was 1643 W/m\(^2\)/day at the summer solstice, 3422 W/m\(^2\)/day at the winter solstice and 3731 W/m\(^2\)/day at the equinoxes.

On the Northeast face of the wall, two regions were marked, in order do all measurements in the same region. Every three months during twenty four months, the color tone was measured in two regions, using a colorimetric spectrometer. The measurements were made taking the "L", "a" and "b" values. The \( \Delta E \) results were obtained by the difference between the initial measurements and those made after eight quarters (twenty four months). In this test two performance parameters were evaluated: the color variation over time and the mechanical damage and failures (e.g. deformations, cracks etc.).
PVC wall resistance to thermal shock
The test to determine the thermal shock resistance of the wall was carried out in accordance with ABNT NBR 15.575-4, Annex E (2013)[14]. The specimen was 2,40m in length and 2,60m high, tighten on the sides, in order to simulate real construction.

The criteria adopted to assess the wall performance when submitted to ten cycles of heat and cold exposure are: a) horizontal displacement of less than h/300, where “h” is the height of the wall sample; b) absence of failures, such as cracks, detachments, blistering and other damage.

Hard-body impact strength of PVC finishing after thermal shock testing
After thermal shock testing, the wall was subjected to a hard-body impact test, in order to evaluate the reduction of the mechanical resistance of PVC finishing. The hard-body impact tests were conducted according to ABNT NBR 15.575-4: 2013 (2013)[14] and SiNAT Guideline 004(MINISTÉRIO DAS CIDADES, 2010)[6], with impact energies of 3,75J and 20J (impacts to the outside face of the walls).

4 RESULTS AND DISCUSSION
4.1 Results of the durability of PVC components
The PVC samples were tested in their original condition and then after ageing in a QUV apparatus for 2000 hours. Tables 3 and 4 summarize the results of mechanical characteristics of PVC for their original situation and after the ageing trial.

Table 3 – Average of the results of Charpy impact tests, before and after ageing

<table>
<thead>
<tr>
<th>Item</th>
<th>Condition</th>
<th>Average of the results of Charpy impact test (kJ/m²)</th>
<th>Standard deviation</th>
<th>Type of rupture</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVC sample used as wall finishing</td>
<td>Original</td>
<td>14,5</td>
<td>2,9</td>
<td>Full break</td>
</tr>
<tr>
<td></td>
<td>Aged</td>
<td>13,3</td>
<td>1,4</td>
<td>Full break</td>
</tr>
</tbody>
</table>

Table 4 – Average of the results of bending strength and elasticity modulus, before and after accelerated ageing trials

<table>
<thead>
<tr>
<th>Item</th>
<th>Condition</th>
<th>Bending Strength to 5% (MPa), as ASTM D 790-72</th>
<th>Standard deviation (Bending Strength)</th>
<th>Elasticity modulus (MPa)</th>
<th>Standard deviation (elasticity modulus)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PVC sample used as wall finishing</td>
<td>Original</td>
<td>71</td>
<td>2</td>
<td>3232</td>
<td>134</td>
</tr>
<tr>
<td></td>
<td>Aged</td>
<td>75</td>
<td>2</td>
<td>2786</td>
<td>328</td>
</tr>
</tbody>
</table>

According to the requirements, as listed in table 1, the results of the tests carried out on PVC samples before and after ageing should not show a difference greater than 30%; table 3
shows that the results meet this criterion. Also, the results of Charpy impact test before the ageing should be ≤ 12 kJ/m².

Concerning the color variation PVC samples were exposed in a Weather-O-Meter apparatus for 2000 hours and measurements were made of the coordinates "L", "a" and "b", every 500 h, 1000 h and 2000 h. The results show that the maximum ΔE measured (ΔE = 0,92 at 1000h) is considered “Weak, but perceptible”, as shown in table 2.

Table 5 – Coordinate color, measured after artificial ageing test in a Weather-O-Meter

<table>
<thead>
<tr>
<th>Coordinate</th>
<th>Average Measured value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Original</td>
</tr>
<tr>
<td>L</td>
<td>95,58</td>
</tr>
<tr>
<td>A</td>
<td>-0,81</td>
</tr>
<tr>
<td>B</td>
<td>2,01</td>
</tr>
<tr>
<td>ΔE</td>
<td>0,5</td>
</tr>
</tbody>
</table>

4.2 Results of the durability of the wall system with PVC finishing

- **Natural ageing resistance - wall system**
  The walls exposed to natural weathering during twenty four months do not present failures, cracks, deformation or other defects.
  Concerning the color variation, the average ΔE of the measurements in the two regions can be seen in figure 3 which also shows the ΔE tendency. The average ΔE in Northeast face is 0.55, after twenty four months.

- **Thermal shock resistance – wall system**
  The results showed that the wall resistance to heat and thermal shock are in accordance with the criteria of NBR 15.575-4:2013, as during and after test there were no failures, cracks, and dislocations greater than h/300 (h = wall height).

![Figure 3 - ΔE tendency in the Northeast face, measured each three months](image)

- **Hard-body impact strength of PVC finishing after thermal shock test**
The PVC finishing also showed hard-body impact resistance, as the test on the faces of the wall did not present any sinking/dipping, neither before nor after the thermal shock test.

This result also demonstrates that Charpy resistance test of the PVC component has influence on the wall resistance of hard-body impact.

5 CONCLUSIONS

The approach to the durability assessment of innovative building products established in the SiNAT Guidelines, presented in this paper, is in accordance with researches already published and can be considered adequate, since the assessment considers the durability of both the individual components and the integrated system. Moreover, natural and artificial ageing trials were performed.

With reference to PVC wall finishing, the test results show adequate durability performance. Mechanical resistance of the PVC components, before and after ageing, was shown to be satisfactory. The results also show that the PVC wall is resistant to thermal shock, considering maximum surface temperature of 60 °C. Concerning the color variation, the results of both natural ageing for twenty four months and artificial aging in a Weather-O-Meter apparatus show acceptable variation (ΔE maximum around 0,7 and 0,92). However, correlation between ageing tests performed in the Weather-O-Meter apparatus and the natural ageing test was not investigated. Likewise, the correlation between the thermal shock test and service life has not yet been studied. The performance of the PVC system discussed, even though the durability tests results are considered adequate, further monitoring must be carried out over the lifetime of the houses. Such a program of monitoring is proposed as the next step of this work, specifically to established the correlation between natural ageing trials, artificial ageing trials and service life.

REFERENCES

Abstract

External sulfate attack is mostly studied on saturated matured samples. These laboratory investigations often focus on the expansive mechanisms altering the mechanical macroscopic properties of the cementitious matrix. In practice, however, most concrete structures are exposed to external sulfate attack at early age and initiation or incubation of sulfate attack more than its later consequences seem to be a recurrent topic of debate. In this work, paste samples prepared with either CEM I (Portland cement) or CEM III/A cement (blended cement with 62% slag) at a water to binder mass ratio of 0.6 are immersed in sodium sulfate solution (15 g/l Na₂SO₄) at controlled pH condition (pH = 8±1) after 24 hours. Sulfate ingress is measured using ICP-AES.

1 INTRODUCTION

In industrial practice, concrete structural elements are exposed to various physical and chemical aggressions as soon as their formwork is removed. This early age exposure has an obvious influence on the element future transfer properties. These properties are at the origin of the durability and more specifically of the ability of the material to sustain deterioration mechanisms due to the penetration of deleterious ions from external environment into the inner porous microstructure of the cementitious matrix. Sulfate ion in seawater or underground is one of these deleterious ions and can cause severe damage to concrete structures. Ingress of sulfate ions present in surrounding sulfate-rich environment and its consequences are known as External Sulfate Attack (ESA). The associated deterioration process involves some expansions, mechanical strength losses (tensile and compressive strengths, Young modulus...) along with surface spalling. This multi-scale process is complex and the circumstances of its initiation are not well defined. They seem to depend on various factors such as cement type, presence of mineral admixtures, water-binder ratio, type of cations associated with sulfate, pH of the solution...[1, 2, 3].
It is now accepted that the above deterioration finds its origin in the dissolution/formation process of ettringite and gypsum, the associated molar volume changes and the ability of the paste porosity to withstand such expansions [4-7]. Sulfate ions diffuse through concrete capillary pore structure [8, 9] in a similar manner as chloride ions [10]. Along with standard diffusion process in a tortuous porous medium, this involves both physical and chemical fixation of ions at the surface of the mineral phases.

In the present paper, we study the mechanisms of sulfate diffusion at very early age for samples made of either CEM I - Portland cement or CEM III/A - blended cement with 62% slag at a 0.6 water to cement ratio by monitoring sulfate ingress profiles every 2 weeks of exposure to a sodium sulfate solution (15 g/l Na$_2$SO$_4$) at a controlled pH (pH = 8±1).

2 MATERIALS AND EXPERIMENTAL PROTOCOLS

2.1 Materials and mix proportions:

Chemical compositions of the tested cements were measured using both Thermo-Gravimetric Analysis (TGA) and Inductively-Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). The results of these two tests are given in Table 1. It can be noted that the clinker phase in the blended cement with 62% slag cement is identical to the clinker in the so-called pure Portland cement. This clinker phase contents are shown in Table 2.

Cylindrical samples of 10 cm diameter were cast with cement pastes with water-to-binder ratio W/B equal to 0.6. This high W/B ratio was chosen in order to artificially “accelerate” sulfate ingress. A sample slow rotation devise was used during setting to prevent bleeding of cement paste (Cf. Fig 1). After 24 hours, all specimens were removed from their molds and were cut into 5 cm thick cylinders. They were then cured in water (for the reference samples) or in the test solution (diffusion bins) after applying an epoxy resin on the peripheral surface in order to insure unidirectional diffusion of sulfate ions (Cf. Fig 1). Only one side of the specimens was in contact with the test solution, which was prepared with 15 g/L of sodium sulfate in deionized water. The solution volume to paste volume ratio was 25. These test conditions are comparable to the study of C. Yu et al. [2].

The pH of the sulfate solution was adjusted to (8±1) by a daily renewal of the solution and addition of sulfuric acid at 0.05% concentration. This procedure allowed for constant experimental conditions (pH and concentrations).
Table 1: Chemical composition of CEM III/A and CEM I cement by using ICP-AES and TGA

<table>
<thead>
<tr>
<th></th>
<th>CEM I</th>
<th>CEM III/A</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>20.22%</td>
<td>29.58%</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>4.85%</td>
<td>8.93%</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>2.92%</td>
<td>1.51%</td>
</tr>
<tr>
<td>CaO</td>
<td>62.81%</td>
<td>49.46%</td>
</tr>
<tr>
<td>CaO (free)</td>
<td>1.58%</td>
<td>-</td>
</tr>
<tr>
<td>MgO</td>
<td>0.84%</td>
<td>4.57%</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>2.88%</td>
<td>1.46%</td>
</tr>
<tr>
<td>S</td>
<td>0</td>
<td>0.58%</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>0.77%</td>
<td>0.62%</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>0.34%</td>
<td>0.48%</td>
</tr>
<tr>
<td>Ignition Loss</td>
<td>2.59%</td>
<td>1.12%</td>
</tr>
</tbody>
</table>

Table 2: The main cement clinker phases according to Bogue analysis

<table>
<thead>
<tr>
<th>Cement</th>
<th>$C_3S$</th>
<th>$C_2S$</th>
<th>$C_3A$</th>
<th>$C_4AF$</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I</td>
<td>62.6%</td>
<td>3.8%</td>
<td>7.9%</td>
<td>8.9%</td>
</tr>
</tbody>
</table>
2.2 Analytical Experimental methods

ICP-AES was used to quantify sulfate content and/or sulfate ingress into the samples. The output from this experimental method is the total sulfur concentration, from which sulfate concentration is computed. In this study, specimens were grinded starting from the exposed surface. The obtained amount of powder corresponding to a 1 mm thick sample displayed an average particle size of 315 µm. All sulfates present in this powder were ionized by an acid attack. The resulting solution was then tested using ICP-AES.

3 RESULTS AND DISCUSSIONS

3.1 Sulfate profiles

The measured sulfate content profiles for CEM I cement paste during the first ten weeks of exposure are shown in Fig. 2 (a). As first observations, it can be noted that the depth of the ingress is about 3 mm after two weeks and reaches 5 mm after 10 weeks. The sulfate content in the first millimeters keeps on increasing with exposure time during the ten weeks with the exception of the first millimeter where a decrease can be noted between 8 and 10 weeks. The average sulfate content (of order 10 g/g) stays far above what could be expected from a simple equilibrium between internal pore solution and external sulfate solution (of order 10^{-3} g/g) and reached the lower bound in the bulk of the sulfate content of the cement powder. We can also observe that the front velocity obviously drops after 3 weeks as illustrated in Fig. 3.

The above observations suggest that:

1/ although a diffusion process due to concentration gradient is initially at the origin of sulfate ingress in the porous medium, sulfate ions get physically and chemically trapped in the paste microstructure

2/ the extremely high ratio (i.e. higher than 10^4) between sulfate content and free sulfate in solution (considering that free sulfate in solution shall be of the order of the external sulfate solution concentration at most) suggest that a chemical binding shall occur, as this ratio is too high for a simple sulfate physical adsorption alone.

3/ Most of the measured sulfate ingress occurs during the first three weeks along with the formation of the paste microstructure due to hydration. This phase is of course very delicate to analyze as many reactions are occurring simultaneously. This phase however exists in practice and the above results show that sulfate ingress measured on 28 days old or older laboratory paste or concrete could lead to strong underestimation of sulfate ingress in the real structure, in which early age ingress could dominate. Moreover, this early age sulfate ingress could be at the origin of a sacrificial coating zone, the thickness of which shall be of critical interest for engineers.

4/ Although the front velocity drops down to zero suggesting that local porosity and permeability after 3 weeks in the bulk have strongly decreased, the amount of sulfate keeps on increasing in the external zone suggesting that either the sulfate presence has prevented the hydration process from reducing the porosity or that a chemical reaction between sulfates and hydration, products is able to increase the local porosity and permeability.
5/ Finally, it can be noted that the decrease in sulfate content measured after 10 weeks in the first millimeter could find its origin in the interaction between sulfate ions, gypsum, hydrated phases and Ca. Release of sulfates in these zones could lead to a decrease through a leaching process. This phenomenon was also observed in A. Chabrelie studies [11] and Ch. Yu et al. [12]. In the specific case of this study, in this interfacial zone, it can be expected that ettringite and gypsum are not stable due to the very low pH of the external sulfate solution.
Figure 3: Front ingress velocity in mm/days as a function of exposure time for the CEM I paste (a) and CEM III/A paste (b).

The sulfate content profile in the CEM III/A paste is shown in Fig. 2 (b). The profiles are very similar in shape to the CEM I case. However, maximum values of sulfate content stay much lower than those of CEM I paste. The sulfate ingress depth is also similar to the CEM I paste for 2 and 4 weeks of exposure to the sulfate solution. At longer exposure times, this depth is inferior to the ones observed for the ordinary cement paste. In addition, the kinetic of the sulfate ions diffusion is lower in the case of CEM III/ cement paste (Cf. Fig. 3 (b)).

Those results suggest, at this stage of the study, that use of slag cement shall be beneficial to reduce the penetration of sulfate element inside the porous media as accepted in literature and industrial practice.

4 CONCLUSION

In the present paper, we have studied the mechanisms of sulfate diffusion at very early age for samples made of either CEM I - Portland cement or CEM III/A - blended cement with 62% slag at a 0.6 water to cement ratio by monitoring sulfate ingress profiles every 2 weeks of exposure to a sodium sulfate solution (15 g/l Na₂SO₄) at a controlled pH (pH = 8±1).

Our results show at first order:
1/ a strong physical and chemical binding of the sulfate ions to the paste microstructure
2/ a fast ingress of sulfate ions during the first three weeks of exposure which could lead in practice to the existence of sacrificial zone and in which chemical reactions seem to alter porosity
3/ a positive effect of slag of both sulfate content and sulfate ingress velocity
The results presented here will be completed by local mercury intrusion porosimetry along with XRD and SEM measurements in order to improve our understanding of the local physical and chemical reactions between sulfate ions and the cement matrix.

REFERENCES

DETERMINATION OF CRITERIA FOR THE EXPLORATION AND FOR OBTAINING INDICATORS IN EVOLUTIONAL ANALYSIS OF DEGRADATION IN URBAN FACADES

Vicenç Gibert (1), Carles Serrat (2) and Joan Ramon Casas (3)

(1) Dept of Architectural Technology II, Universitat Politècnica de Catalunya-BarcelonaTECH, Barcelona – vicenc.gibert@upc.edu
(2) Dept of Applied Mathematics I, Universitat Politècnica de Catalunya-BarcelonaTECH, Barcelona – carles.serrat@upc.edu
(3) Dept of Construction Engineering, Universitat Politècnica de Catalunya-BarcelonaTECH, Barcelona – joan.ramon.casas@upc.edu

Abstract

The interest of this research is motivated by the huge constructive development in the building sector, which has led to an enormous volume of buildings worldwide over the last decades. This fact, as well as poor management in conservation of buildings, mainly in urban cities, has led to total degradation situations on the facades of building stocks. To infer on the evolutional process of degradation that occurs in the facade, during its service life and based on the information obtained from inspections, the method for the data analysis (e.g. survival analysis techniques) is as important as the truthfulness and usefulness of the data collected on the subjects (facades) under study.

In this paper we propose a method based on the design of indicators for prospecting and global data collection on the injury state of the inspected facades. The method analyzes how to perform the identification, classification, methodology, resources and quality of the data in order to be reliable, testable and measurable. The proposed model enables the macro inspection and monitoring of large-scale urban zones and the data obtained allow the researcher to understand and manage the predictive degradation phenomenon and, in turn, eliminate potential risks in the urban front.

Keywords
Criteria validation, Data collection, Durability indicators, Inspection methodology, Risk analysis, Service life estimation.
1 INTRODUCTION AND STATE OF THE ART

In relation to the acceleration of the process of urbanisation and construction in recent decades in most big cities, the debate on the conservation and maintenance of the built environment is open. In addition, questions about how to articulate conservation measures in the construction sector are discussed. This is an important issue because the measures affect significant sustainable positioning for the future of the cities and their metropolitan areas. This research focused on the real evolution of the degradation of the facades of residential buildings, within their service life, regarded as a part of the fronts of the urban space of the cities, in which lies a great burden of media and social coverage.

Surely all decisions of intervention need, as a preliminary condition, of having as much good information as possible, therefore we need to determine what criteria and what indicators are needed to establish the evolutionary analysis of the degradation of urban facades. This paper shows a massive inspection methodology, with the aim of finding the optimal information to facilitate future statistical studies of durability and survival for each one of the construction processes of the urban front.

As a result, and despite the existence of some important experiences at different scales in the building sector [1], the methodology we are going to develop here is unique because it pursues the analysis of samples of study much larger and that force you to the application of multi-scale techniques. The methodology of inspection that pose to display a model designed for the control of the durability of urban facades, depending on the risk of an injury that these represent. It is scheduled for a data collection setting, wide, generalist, robust, and aimed at the whole population, and it makes easier the use of specific algorithms. This will give us a better understanding of what happens to the facades throughout its service life. Through this method it will be possible to propose preventive actions for the future within a territorial approach on a large scale.

The proposal raises field datasheets that collect all those parameters that contain relevant indicators for each of the subdivisions, in which each facade has been divided. However the interest focuses, on one hand, on the degree of accuracy of the resulting information coming from the data and, on the other hand, on the reliability of the methodology of inspection regarding the state of degradation of the facade elements.

Therefore one of the concerns of the proposal consisted in observing patterns of urban growth, as well as those that define the city considered as a part of a comprehensive system with certain hierarchies that mark the interconnections between them [2]. The description of the study scale in order to analyze different magnitudes of samples, has been defined by various researchers as the microscale, mesoscale and macroscale [1, 3 and 4]. On the basis of this classification, the concept of multiscale studies arises -in order to provide a better understanding by the up-and-down flow of the information available between different scales. An example of a multiscale study is conducted by Gluch et al. (2006) [3], in which a thermic analysis is created in order to better understanding the urban microclimate. Other authors apply their representations of the atmospheric environment in urban areas by reducing the regional scale to a city scale, using computational models [4]. The effects from mesoscale to microscale and vice versa were discussed by the authors and they concluded that, in a general sense, the scalar interaction can play an important role in all directions.

In our opinion, all method aimed at permitting broad inspections, in this case the building facades, must be able to show a clear traceability towards the aimed result, allowing the
adjustment of the parameters through the use of decision making techniques, such as the comparative techniques of analytic hierarchies (AHP) [5], ELECTRA [6] or the axiomatic method by Raynaud and Arrow [7].

One of the studies in urban scales, even though they are rare among the scientific community, is made by Mendes da Silva et al. (2010) [8] for the old town buildings of Coimbra (Portugal). The project focuses on the concerns of the conservation of the existing heritage. It also shows how to develop a platform for management of the park, aimed to the achievement of the social and economic improvement of the area. To carry out the study, it was necessary to understand that the process of improvement could not be sustainable without a prior vulnerability and risk assessment of the state of the buildings. The data could be implemented using a multi-purpose tool, connected to a relational database within a Geographic Information System. The methodology that they apply is practical and flexible, and it allows a multidisciplinary management, in addition to the potential applicability to other areas, cities and buildings just introducing some slight adjustments of the parameters. Another study that focused on the durability of buildings on a urban scale, is the methodology of evaluation introduced by Rodrigues et al. (2011) [9], which is based on the premises that the mechanisms of deterioration are the consequences of the interaction of two independent variables: the building, such as a physical object, and the environment, as a source of agents of acceleration of the deterioration process. The methodology has allowed the development of tools to support the systematic study of visual defects in the buildings. The matrix designed by the authors for the visual observation extends the application of Failure Mode and Effect Analysis (FMEA) techniques for the qualitative analysis of risks, and it represents an important guideline for the design teams.

2 MODEL PROPOSAL

This section develops the methodology in order to perform the analysis of the whole building park at a macroscale level. The model defines the criteria to be taken into account when creating an inspection tool, through the creation of strategic indicators and their respective weights of importance, aiming to develop a standard inspection sheet. The structure of the proposed tool optimizes the information collected in the field, considering the elements of observation and his condition.

2.1 A network of urban laboratories approach

To define the boundary conditions under which this methodology has been designed to inspect heterogeneous samples of urban facades, we start from the conceptual and operational idea that the territory under study is subscribed inside the are delimited by the perimeter of the limits of the urban area of the city. This territorial space is what we call "urban laboratory" with the clear aim of being a centre of operations and strategies that allows us to link the experiences acquired with other urban laboratories, thus creating a collaborative knowledge framework for sharing predictive formulas of conservation and sustainable maintenance strategies of the urban front.

2.2 Durability goals

In the building sector there is no proven information to determine what is the evolution of the degradation of the facades over time, in terms of use and their exposure to external agents. The proposed methodology allows the investigator to characterize different type of injuries and
categorised them according to their severity and magnitude levels, in relation to the risk that they represent for citizens in the public way. From this perspective, durability must be understood as the time to reach each one of these risky levels of deterioration.

Authors such as Serrat and Gibert (2011) [10] applied survival statistical models for estimating time to these levels of degradation during the service life of the facade. However, in order to achieve reliable results, it will be necessary to have a sample representative enough and repeated inspections throughout the life of the buildings.

Figure 1 shows the hypothesis that relates the causes of degradation of the elements of facade ($C_i$) with injuries arising in different levels ($L_{ik}$). It can be seen that different inspection times ($I_j$) get from the facade partial, but complementary, information on the evolution within a lesion and between lesions.

Figure 1: Illustration of the inspection methodology together with the progression among the different deterioration states along the service life of the facade

Although the causes are responsible for the presence of different types of lesions that appear on the facades, our methodology will focus on collecting data based on the evidence of detectable injuries. This approach agrees with the goal of describing the most recurrent lesions at macroscale level, in order to know how they evolve over time and how they influence the overall durability of the facades.

2.3 Criteria proposal for the indicators

Most of the authors in this research field and those that have reported experiences in the methodology of inspections at macroscale level, such as Jordana and Gibert (1999) [11], Peixoto de Freitas et al. (1999) [12], Mendes da Silva et al. (2010) [8] and Rodrigues et al. (2011) [10] support the idea of carrying out visual inspections as they offer greater representativity of the facts that investigators are interested in. However, for this it is necessary to fix the purpose pursued, the goals to be achieved, the types of data that are needed to compile and the methodology to be used. It is precisely in these strategic points, previous to the design, where maximum differences between authors are found. Although all agree the realization of visual inspections collected in a physical support of field datasheets, no consensual trend for data collection is given. In this regard, this circumstance may be due to the lack of common regulations among different countries.
After the analysis of the requirements and the goals of the research study, the criteria that we propose to take into account in the inspection methodology, as well as the specific characteristics of each one of them, are shown in Table 1.

<table>
<thead>
<tr>
<th>General</th>
<th>Specific</th>
<th>Relative / Overall Weight (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Identification 17.5%</td>
<td>Descriptive 36.25 / 6.34</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Methodical 28.75 / 5.03</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Universal 53.00 / 6.13</td>
<td></td>
</tr>
<tr>
<td>Classification 16.25%</td>
<td>Sequential 36.25 / 5.89</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Detailed 35.00 / 5.69</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ordered 28.75 / 4.67</td>
<td></td>
</tr>
<tr>
<td>Methodology 22.5%</td>
<td>Robust 35.00 / 7.87</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Standard 30.00 / 6.75</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Quick 35.00 / 7.87</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>General</th>
<th>Specific</th>
<th>Relative / Overall Weight (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resources 11.25%</td>
<td>Technological 25.00 / 2.87</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Human 35.00 / 4.03</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Time 40.00 / 4.60</td>
<td></td>
</tr>
<tr>
<td>Data 21.25%</td>
<td>Reliable 42.50 / 9.03</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Quantifiable 31.25 / 6.64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Verifiable 26.25 / 5.58</td>
<td></td>
</tr>
<tr>
<td>Analytical skills 11.25%</td>
<td>Multifunctional 26.25 / 2.95</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Processable 41.25 / 4.64</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Longitudinal 32.50 / 3.66</td>
<td></td>
</tr>
</tbody>
</table>

Table 1: Weighted criteria (in percentages) and indicators for the inspection methodology

2.4 Determination of the weights for the indicators

In order to determine the degrees of importance of each of the requirements, designed for the realisation of major inspections in urban facades, it was necessary to establish, by means of consulting experts, the weights based on a survey and a direct assignment strategy, in order to assess the degree of suitability of the inspection document based on the goals of the project.

First column in Table 1 shows the percentages of each of the requirements, among which we can point out that Methodology (22.50%) and Data (21.25%) are those that require greater attention. Looking at the third column we can see the relative (within the general criterium) and the overall impact of each indicator. As a consequence, all the efforts in the design of the inspection methodology must be focused on collecting reliable (9.03%) and quantifiable (6.64%) data, using a robust (7.87%), quick (7.87%) and standard (6.75%) methodology, and allowing a descriptive (6.34%) but universal (6.13%) identification of the facades.

2.5 Design of the areas to be inspected

The inspection of each facade includes the differentiation of three virtual vertical planes, where all those elements that form part of it are located. The first plane is the vertical closure of the building with the alignment in the road, the second plane represents the cantilever elements formed by balconies and, finally, the third plane contains all solid bodies of facades galleries. Once the planes of interest were established, we proceeded to determine what kind of information was necessary to collect either to eliminate the risk, or to enter into a process of adaptation and improvement of the facade. The planes mentioned above are shown in Figure 2. Next step will be to define on these planes the data collection on the basis of the constructive characteristics of each facade.
2.6 Data collection sheet structure

To preserve the essence of the predetermined requirements, it was necessary to establish a method of data collection that had to be fast without creating doubts in the inspector. It had to be standard and capable of bringing together the best information in the minimum possible space. The appropriate answer was given for a field datasheet. In order to achieve the expected results in the inspection phase, we designed general documents for a generic sample of buildings. The final documentation has been divided into two main blocks, one focused on the collection of information related to planning, building and uses, that allows to identify the property, and another more specific for the information on the level of degradation of the elements on the facade under inspection.

Figures 3a and 3b contain the fields of those sections that are object of study in order to achieve the objectives and they allow, without loss of time, take the maximum information for future studies. This first tab collects data from different administrative databases and, after being compared with those on site, provides the homogenization of municipal information at the same time that allows to characterize the property for their age, type, usage, dependencies and the transformations that have undergone throughout of his life.

![Figure 3: Field datasheet a) for the building and b) for the facade](image)

The scheme in Figure 3b shows two large sections where, systematically, we can collect (left-hand part) the different parts into which we have subdivided the facade, in concordance with the planes previously described, and (right-hand part) the type, extent and severity of the injuries.
Once we have fixed the sections, subsections and elements to be observed in the inspections, as well as the different concepts of classification of the status of each facade, we can design the field datasheets that will be used by different groups of inspectors for the collection of data.

The functional importance of the tabs and the apparent rigidity of its contents are essential to obtain information robust enough, aimed to determine the real status of the building stock. Despite this, it is necessary to instruct the inspectors to explain how they should organised the datasheets, what types of information they contain, as well as the criteria to be taken into account in front of a particular injury.

2.7 Application

The methodology introduced in the previous subsections, in preliminary versions, has been succesfully applied in several cities and locations. As a summary of the project and as a first picture of the results, Table 2 shows the location of the inspections, the number of inspected facades in each location and the percentage of facades in each severity level, in terms of the estimation of the time in which a maintenance action would be needed. Figures illustrate clear differences among locations and make evidence for the interest of a network of urban labs.

<table>
<thead>
<tr>
<th>Country</th>
<th>City</th>
<th>Number of Inspected facades</th>
<th>Percentage of Actions</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Long-term</td>
</tr>
<tr>
<td>Spain</td>
<td>L'Hospitalet de Llobregat</td>
<td>13193</td>
<td>50</td>
</tr>
<tr>
<td></td>
<td>Esporles (Mallorca)</td>
<td>291</td>
<td>43</td>
</tr>
<tr>
<td></td>
<td>Barcelona (Ciutat Vella)</td>
<td>2631</td>
<td>24</td>
</tr>
<tr>
<td></td>
<td>Barcelona (Eixample)</td>
<td>2736</td>
<td>39</td>
</tr>
<tr>
<td>Chile</td>
<td>Valparaiso</td>
<td>396</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>Santiago de Chile</td>
<td>1403</td>
<td>28</td>
</tr>
<tr>
<td>Mexico</td>
<td>Mexico F.D.</td>
<td>525</td>
<td>16</td>
</tr>
<tr>
<td></td>
<td>Global state of the cities inspected</td>
<td>21175</td>
<td>42</td>
</tr>
</tbody>
</table>

Table 2: Scope of the project for a network of urban laboratories

3 CONCLUSIONS

In response to the problem of how to approach, at macroscale level, an analysis of urban facades, a standard inspection methodology for the collection of information on their conservation status has been developed.

The core of the inspection tool was created taking into consideration a number of key indicators that focus the interest in collecting data according to its importance. Its structure allows to differentiate the identification details of the building from the descriptive data of the facade itself. It is also possible to classify all the observed elements, and it allows to record its condition at time of inspection.

Definitely the proposed methodology will facilitate the collection of information on the status of the built park, in order to record a visual inspection with objective data. According to
this design, the data concerning the state of conservation of the facade elements can be easily analyzed in subsequent statistical studies, and they can also be displayed and managed by Geographic Information System platforms.

ACKNOWLEDGEMENTS
This work has been partially supported by the School of Building Construction of Barcelona (EPSEB-UPC), by the Building Laboratory at EPSEB and by the Spanish Ministry of Economy and Competitivity under grant MTM2012-38067-C02-01. Special thanks are due to the members of the IEMAE and GRASS research groups for their valuable comments and suggestions.

REFERENCES
CORROSION RESISTANCE MEASUREMENTS AND THE INFLUENCE OF MATRIX ALKALINITY ON DURABILITY PERFORMANCE OF MEDIUM STRENGTH GEOPOLYMER CONCRETES

Kwesi Sagoe-Crentsil \(^{(1,2)}\) and Shiqin Yan \(^{(1)}\)

(1) CSIRO Materials Science and Engineering, Highett, Vic 3190, Australia – Kwesi.Sagoe-Crentsil@csiro.au; shiqin.yan@csiro.au

(2) Centre for Sustainable Infrastructure, Faculty of Engineering & Industrial Science, Swinburne University of Technology, Hawthorn, Victoria 3122, Australia –

Abstract

The alkaline environment of reinforced concrete (RC) remains a critical requirement for steel reinforcement protection not only in ordinary Portland cement (OPC) concrete but also for alternative binder systems such as geopolymer concrete. In this study, the corrosion current density \(I_{\text{corr}}\) and polarisation resistance \(R_p\) values of steel electrodes embedded in ambient cured 35MPa OPC and fly ash geopolymer concretes have been evaluated using the standard linear polarisation technique. While the polarisation curves display stable passivation of the steel by both concrete types, very high corrosion rates appear to characterise geopolymer concretes at very early stages with measured mean \(I_{\text{corr}}\) values of \(3.822 \times 10^{-3}\) mA/cm\(^2\). This trend correlates well with corresponding lower \(R_p\) values for geopolymer concrete samples which is partly attributable to its higher matrix alkalinity relative to conventional OPC, hence actual corrosion rates may be lower than indicated. The inter-relationship between the highly alkaline matrix of geopolymer concretes and durability parameters such as corrosion rate and potential alkali silica reactivity (ASR) performance are discussed.

Keywords: geopolymer concrete, corrosion resistance, compressive strength, water permeability

1 INTRODUCTION

The durability and serviceability requirements for reinforced concretes structures, in particular, concretes derived from alkali activated binders remain largely empirical and continue to gain considerable attention [1-3]. Generally, the durability and long-term engineering performance of conventional OPC concrete can be assessed by indirect measurement of fluid transport parameters such as ion permeability, porosity and gas diffusion, etc. [4,5]. Moreover the mechanisms of deterioration of OPC concrete arising from ingress of various ions, liquids and gases from the environment are well documented [6-8].
For instance, structural elements under aggressive chloride ion exposure either from marine environments or de-icing salt are known to induce corrosion cracking and spalling of concrete, causing serious loss of serviceability and structural integrity of bridge decks, parking garages and marine structures.

The resistance of concrete to chloride ion under aggressive salt exposure and fluid ingress is largely governed by transport phenomena of the concrete matrix and is known to be largely influenced by concrete mix design parameters including binder selection which, in turn, governs durability performance of the structure [9-11]. Corresponding studies investigating various aspects of mix composition, microstructure and durability issues relating to the family of geopolymer (GP) systems are now only beginning to emerge [2, 12-13]. In general, alkaline activated cements are believed to display better durability behaviour than conventional OPC binder due to their high resistance to attack by acids, sulphates and reduced susceptibility to expansion due to alkali–silica reactions (aggregate-alkali reaction, formation of thaumasite, etc.) [14]. Ismail et al. [15] demonstrated that the inclusion of fly ash in alkali activated slag mortars and concretes modified the permeability of the material and, hence, the diffusion of aggressive agents such as chlorides. Similarly, Kupwade-Patil et al. [2] indicated that Geopolymer concrete specimens made from Class F fly ash exhibited lower diffusion coefficients, free chloride contents, and porosity relative to OPC concretes.

The chemistry and transport mechanisms of chloride in cementitious binders may be influenced by the prevailing reaction products, mainly CSH/NASH gels and/or silicate network structures, which control ionic transport mechanisms. However, in the case of fly ash based calcium-free geopolymer systems, the parameters influencing long-term durability performance, in particular fluid permeability and their implications on rebar corrosion are yet to be fully addressed. Thus, this paper examines the influence of matrix alkalinity on rebar corrosion susceptibility and durability performance of medium strength and the mechanisms that control overall transport processes in these systems.

2 EXPERIMENTAL

Table 1 shows chemical composition of ASTM Type I OPC used for reference concrete mixtures and Class F fly ash used for the Geopolymer (GP) mixtures. Table 2 provides concrete mix proportions of the concrete formulations. River sand and local 9mm Hornfels coarse aggregate were used. Both the reference OPC and GP concrete mixes were designed to achieve target 28-day strength of 35 MPa.

The GP concrete samples were prepared in a similar fashion as reference OPC concrete and moulded according to standard requirements for compressive strength measurements. Ambient cured specimens were stored at 23°C and 100%RH until required for testing. Standard concretes cylinders of 100×200mm diameter were used for compressive strength measurements.
Table 1: Chemical composition of binder components

<table>
<thead>
<tr>
<th>Oxide</th>
<th>OPC (wt %)</th>
<th>Fly ash (wt %)</th>
<th>Na-silicate (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>20.1</td>
<td>48.0</td>
<td>28.7</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>4.61</td>
<td>29.0</td>
<td>—</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.92</td>
<td>11.8</td>
<td>—</td>
</tr>
<tr>
<td>CaO</td>
<td>61.9</td>
<td>3.72</td>
<td>—</td>
</tr>
<tr>
<td>MgO</td>
<td>1.22</td>
<td>1.59</td>
<td>—</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.16</td>
<td>0.50</td>
<td>8.9</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.39</td>
<td>0.55</td>
<td>—</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.24</td>
<td>1.54</td>
<td>—</td>
</tr>
<tr>
<td>SO₃</td>
<td>2.66</td>
<td>0.27</td>
<td>—</td>
</tr>
<tr>
<td>H₂O</td>
<td>—</td>
<td>—</td>
<td>62.4</td>
</tr>
<tr>
<td>LOI</td>
<td>5.1</td>
<td>1.6</td>
<td>—</td>
</tr>
</tbody>
</table>

Table 2: Concrete mix proportions

<table>
<thead>
<tr>
<th>Material</th>
<th>OPC Concrete (kg/m³)</th>
<th>GP concrete (kg/m³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fly ash</td>
<td>-</td>
<td>269</td>
</tr>
<tr>
<td>Ordinary Portland Cement</td>
<td>346.0</td>
<td>-</td>
</tr>
<tr>
<td>Coarse aggregates (9mm)</td>
<td>1112.4</td>
<td>1163</td>
</tr>
<tr>
<td>Silicate Solution/NaOH</td>
<td>-</td>
<td>148</td>
</tr>
<tr>
<td>Dry Sand</td>
<td>753</td>
<td>784</td>
</tr>
<tr>
<td>Water</td>
<td>186.2</td>
<td>27</td>
</tr>
<tr>
<td>Wet concrete property</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mix Temp. (°C)</td>
<td>15.7</td>
<td>25.5</td>
</tr>
<tr>
<td>Flow (mm)</td>
<td>-</td>
<td>260.0</td>
</tr>
<tr>
<td>Slump (mm)</td>
<td>120</td>
<td>160.0</td>
</tr>
<tr>
<td>Wet density (kg/m³)</td>
<td>2381</td>
<td>2430.0</td>
</tr>
</tbody>
</table>

Concrete cylinders for compression testing were tested in accordance with AS 1012.9 (1999)-Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens [16]. Concrete compressive strength was tested at 1, 7 and 28 days in a 2500 kN ELE Universal testing machine.

The coefficient of water permeability ($K_w$) was determined by measuring the amount of water passing through the specimen and calculated using Darcy’s law. The test determines rate of water absorption through the concrete surface. The experimental setup for high water pressure i.e. 525kPa was previously reported [11]. Prior to conducting permeability testing, 50mm sliced disc samples were cut from the concrete cylinders, and embedded in 25 mm thick epoxy resin to enable water-tight fit within the permeability cell. Test specimens were then loaded into the water pressure cell and a fixed pressure applied throughout the duration of the test. For each permeability test, three specimens were prepared and tested to ensure statistical significance.
The linear polarization technique (potentiostatic method) using mild steel rods (6mm diameter and 170mm length) embedded in concrete blocks was adopted. The set-up is shown in Fig 1. The set-up consisted of a 3.5% NaCl sodium chloride solution reservoir on the top of the concrete slab for testing duration at room temperature. The electrochemical measurements were performed using a three-electrode system using an EG&G Princeton Applied Research Model 273 potentiostat. A saturated calomel electrode (SCE) was used as reference electrode (RE). The counter electrode consisted of a graphite rod.

The corrosion current density ($I_{corr}$) was calculated through the Stern and Geary equation ($I_{corr}=B/R_p$), where $B$ is the proportionality constant ($B$ has value of 0.028). The internal resistance (IR) drop was corrected by using a feedback compensation technique.

The corrosion current ($I_{corr}$) and the polarization resistance ($R_p$) were recorded periodically and each $I_{corr}$ value was calculated as the average of the value of the two identical bars embedded in each specimen. Polarization resistance ($R_p$) measurements were scanned from -20 mV cathodic to +20 mV anodic corrosion potential, at a scanning rate of 0.166 mV/s. The polarization resistance, $R_p$ was determined as the slope at zero current on the potential versus current graph obtained from the experiments. The polarization resistance ($R_p$) is the ratio of change in measured potential to the change in applied current per unit area ($\Delta i$):

$$R_p = \Delta E/\Delta i$$

3 RESULTS AND DISCUSSION

3.1 Compressive strength

Table 3 shows the mean compressive strength development of ambient-cured OPC and GP concretes. It is observable, that the strength development trends of the two mixtures differ in that a relatively rapid early age strength development trend was observed for OPC specimens compared to GP concrete. The characteristic slower strength development rate for the GP concrete is as expected and is partly dependent on curing temperature, with higher curing temperatures resulting in marginally higher early compressive strength gains. The latter age strength increase in GP systems is a result of progressive polymerization reactions that occurs.
beyond the initial curing stage compared to relatively rapid hydration reactions characterising OPC systems at 1 day and beyond.

Table 3: Compressive strength development of ambient cured OPC and GP concretes

<table>
<thead>
<tr>
<th>Compressive strength (MPa)</th>
<th>GP Ambient-cure</th>
<th>OPC Ambient-cure</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-day</td>
<td>-</td>
<td>9.1</td>
</tr>
<tr>
<td>3-day</td>
<td>2.0</td>
<td>-</td>
</tr>
<tr>
<td>7-day</td>
<td>7.2</td>
<td>35.4</td>
</tr>
<tr>
<td>28-day</td>
<td>35.5</td>
<td>43.7</td>
</tr>
</tbody>
</table>

3.2 Water permeability

The results of water permeability ($K_w$) tests at 28 days show significant differences between GP and OPC concretes. The OPC concrete showed reduced permeability value of $8.67 \times 10^{-12}$ m/s which is two orders of magnitude lower compared to $7.21 \times 10^{-10}$ m/s for the equivalent GP concrete.

The measured permeability values are likely influenced by two primary factors; (i) the network of continuous capillary and interconnected micro-pores porosity binder matrix fraction and, (ii) micro-cracks in concrete, especially at the paste/aggregate interface [7,17]. For OPC concrete, the porosity and interconnectivity are controlled for most part by the w/c ratio, degree of hydration, and the degree of compaction. However the actual mechanism of fluid transport through the matrix is dictated by combination of diffusion, permeation, capillary action and absorption processes [18]. However, corresponding permeability of geopolymer concrete appears to be dictated by an inherent mesoporous capillary pore network structure for which transport properties appear to be partly dependent on mode of concrete curing. Whereas the underpinning mechanistic parameters that control the movement of gases, liquids and ions through GP concrete may be expected to be similar to OPC systems, the interactions between transported fluids and chemical constituents of the binder matrix as well as the pore water often govern the impact on concrete durability and performance [19].

3.3 Polarization resistance measurements

Fig 2 shows cathodic polarisation resistance curves at 7 days and 60 days for both GP and OPC concrete samples. The results are summarised in Table 4. The steel corrosion rate, expressed as millimetre per year (mmpy), for GP specimens were observed to be higher by nearly two orders of magnitude at 60 days with correspondingly low polarisation resistance values. The higher rate for the GP concretes is largely an artefact of the highly conductive matrix owing to excess alkalinity which characterises alkali activated binder systems. While the excess hydroxyl ions in the pore solution tends to distort measured polarisation values, such conditions are also likely to sustain sufficient high pH regimes required for maintaining passive film formation at the rebar interface. Moreover, properties of the interfacial layer depend on binder phase composition (combined C-S-H/N-A-S-H and aluminate phases in OPC systems), matrix porosity and pore fluid chemistry [20]. For OPC blends containing supplementary cementitious materials (SCM’s) such as, fly ash, blast furnace slag and/or
silica fume, generally observed reduction in chloride migration rates have been attributed to (1) enhanced binding capacity in systems with higher aluminate levels and (2) a refinement of matrix pore structure [21].

![Graph](image.png)

Fig 2: Tafel response using SCE reference electrode for cathodic polarization segment (a) GP-7 days; (b) OPC-7 days (c) GP-60 days and (d) OPC-60 days

<table>
<thead>
<tr>
<th></th>
<th>Polarisation resistance $R_p$ ($\Omega$)</th>
<th>$I_{corr}$ ($\text{mA/cm}^2$)</th>
<th>Corrosion rate (mmpy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC 7-day</td>
<td>$1.095 \times 10^3$</td>
<td>$6.406 \times 10^{-6}$</td>
<td>$2.959 \times 10^{-3}$</td>
</tr>
<tr>
<td>OPC 60-day</td>
<td>$1.436 \times 10^5$</td>
<td>$4.884 \times 10^{-6}$</td>
<td>$2.256 \times 10^{-3}$</td>
</tr>
<tr>
<td>GP 7-day</td>
<td>$2.251 \times 10^3$</td>
<td>$3.115 \times 10^{-6}$</td>
<td>$1.439 \times 10^{-3}$</td>
</tr>
<tr>
<td>GP 60-day</td>
<td>$1.835 \times 10^2$</td>
<td>$3.822 \times 10^{-3}$</td>
<td>$1.766$</td>
</tr>
</tbody>
</table>

It is conceivable that the single most important corrosion inhibition property of Geopolymer systems may be attributed to the potentially high buffering capacity which can
restrict local pH levels above threshold for corrosion owing to its alkaline reserves. Rapid repassivation of incipient anodes on steel reinforcement due to the reserve of hydroxyl ions at the steel concrete interface has the potential to lower chloride ion activity in the local pore. Accordingly, it may be inferred that higher chloride threshold levels compared to traditional OPC systems arising from the mitigating role of excess hydroxyl ions in GP binder matrices against potential corrosion may exist although in-service occurrence of such protection remains unclear and yet to be demonstrated.

Ismail et al. [15] showed that the chloride binding capacity of alkali activated mortars were indeed high with fly ash-based geopolymer. These results were consistent with the chloride penetration depth measurements and the observed trend was attributed to the notion that the surface area of the alkali-activated binding gel increases with fly ash inclusion [22]. However, excess alkalinity can also have adverse implications on other in-service durability parameters such as Alkali silica reactivity (ASR). Although GP binders contain a high level of alkali elements, the occurrence of ASR could be restricted, which may be due to the fact that the majority of alkali elements may be associated with other reaction products. Furthermore, given the critical role of calcium in ASR development, possible suppression of ASR in calcium-free alkali-activated binders is feasible and may require further investigation [23]. Thus, the mechanisms of deterioration can often be very complex, since in addition to the transported fluid, variables such as the nature and type of reaction products, degree of polymerization, system chemistry, humidity differentials, temperature and other secondary factors all contribute to effective concrete durability.

4 CONCLUSIONS

– It is noted that the primary inhibitive property of geopolymer binder systems appears to be its effective alkali reserve buffering capacity which regulates pH values above 12.6 similar to OPC binders.
– The steel corrosion rate, expressed as millimetre per year (mmpy), for GP specimens were observed to be higher by nearly two orders of magnitude at 60 days, with correspondingly low polarisation resistance values.
– Rapid repassivation of incipient anodes on steel reinforcement due to the reservoir of hydroxyl ions at the steel concrete interface has the potential to lower chloride ion activity in the local pore.
– The observed mean water permeability coefficient value of structural grade GP concrete was 7.21E-10 m/s at 525 kPa water pressure.
– The nature and integrity of the interfacial layer of solid reaction products formed in close proximity to the embedded steel while not investigated in the current study may provide critical insights to overall durability performance of GP binders.

REFERENCES


MAINTENANCE AND REFURBISHMENT; MARKET PRICE AND FIXED ASSETS VALUE

Björn Marteinsson\(^{(1)(2)}\)

(1) School of Engineering and natural sciences University of Iceland, Reykjavik – bjomar@hi.is

(2) Innovation Center of Iceland, Reykjavik – bjorn.m@nmi.is

Abstract

A building is designed and built for a defined service life period and with necessary economic maintenance the requirement in the Construction Products Regulation shall be fulfilled the whole time. The goal of maintenance is to ensure that the basic requirements (in building regulations) are fulfilled and so also keep up the economic value of the work. In calculating the fixed assets of properties owned by municipalities, and all properties when estimating the total figure for the nation, economists depreciate the value of buildings by 2-2.5 \% yearly. They are thus claiming that the maintenance cost does not ensure the value of the buildings; the maintenance is not effective. This controversy is discussed in the paper; based on actual market price for family housing in the capital area in the period 1995-2010. It is shown that market price is not necessarily affected by age of buildings.

1 DESIGN LIFE OF BUILDINGS AND DECAY

Buildings are designed and built for a foreseeable purpose with more or less known conditions. Thus the basis of the design is performance requirements and specifications, though the building also has to fulfil minimum requirements of regulations.

The Construction Products Regulation from the EU [1] contains the following text and list of basic requirements:

*Construction works as a whole and in their separate parts must be fit for their intended use, taking into account in particular the health and safety of persons involved throughout the life cycle of the works. Subject to normal maintenance, construction works must satisfy these basic requirements for construction works for an economically reasonable working life.*

1. Mechanical resistance and stability
2. Safety in case of fire
3. Hygiene, health and the environment
4. Safety and accessibility in use
5. Protection against noise  
6. Energy economy and heat retention  
7. Sustainable use of natural resources

Further aspects of each requirement are described, while it is also stated that each Member State shall describe in more detail which standards shall be used.

The requirements state that it is assumed that normal maintenance is sufficient to maintain properties for an “economically reasonable working life”. Pointers regarding assumed service life can be found in various EU documents, and it is expected that the building will fulfil the above items for at least the time shown in Table 1 – and longer if it seems this is possible. In general, residential buildings can be assumed to fit into the “normal” category while well-made public buildings would be part of the “long” category”.

<table>
<thead>
<tr>
<th>Category</th>
<th>Years of works</th>
<th>Repairable or easily repairable</th>
<th>Repairable or replaceable with some more efforts</th>
<th>Lifelong</th>
</tr>
</thead>
<tbody>
<tr>
<td>Short</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>Medium</td>
<td>25</td>
<td>10</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>Normal</td>
<td>50</td>
<td>10</td>
<td>25</td>
<td>50</td>
</tr>
<tr>
<td>Long</td>
<td>100</td>
<td>10</td>
<td>25</td>
<td>100</td>
</tr>
</tbody>
</table>

Notes
1. In exceptional cases, a working life of 3-6 years may be envisaged for certain repair products
2. When not “easily replaceable” or “replaceable with more effort”.

As soon as the building has been brought into operation, these premises could begin to change and the building itself to age. The function and condition of the building changes and maintenance must be carried out in order for the building to maintain its desirable qualities, while potentially changes might also be necessary to adapt it to new needs. The circumstances that might arise can be divided into three categories:

Technical
- Material qualities change, materials decay
- Equipment and safety requirements change

Economic
- Assessed value changes, e.g. because of community development or planning clauses
- Maintenance has become unduly extensive or expensive in relation to other options

Social
- Desire for renovations due to appearance or fashion trends
- Desire for transfer due to changes in the nature of the surroundings (safety, make-up of society, noise, pollution)
Maintenance and replacement are frequently split into foreseeable and unforeseeable factors; in particular, technical circumstances can be called foreseeable, while economic and social factors are less foreseeable, or perhaps not at all if looking to the long term. Various unexpected incidents, such as broken windows or natural disasters, are also unforeseeable in this sense, though the likelihood of those arising can be assessed from prior experience.

2 MAINTENANCE AND LOSS OF PROPERTY

For the first years of a building's usable life, the need for maintenance is minor, and consists mainly of painting the exterior and interior and also adjustments due to damage. When the age of the building has entered its third decade, the need for maintenance increases, as then windows frequently need to be fixed, glass needs replacing; some of the cladding has started to give way and requests for replacement of fittings increase. It has been shown that general maintenance of residential buildings in Iceland accounts annually for 1-2% of the building cost of a new building [3]; this is supported by the experience of large property owners. In terms of expenses, building maintenance can thus be considerable when calculated over the normal lifetime of a building, as the general idea is such that maintenance can ensure quality and therefore the property value of the building.

Loss of a property could occur for various reasons, such as fire, natural disasters or the above-mentioned economic and social reasons. Obsolescence because the building does not fulfil the demands of the house owner or environment, or because it is not economical to make the necessary changes, is perhaps the biggest threat to sustainability in the built environment, because this sort of thing calls on an increased need for new buildings that would otherwise not be necessary. A survey on the opinion of homeowners on the quality of their property and surroundings showed that owners of buildings built after 1950 in two urban areas in Iceland (Akureyri and the capital city Reykjavík) were generally very pleased or pleased with their property [4]. Generally the loss of buildings in Iceland is very low at present (figures for last 15 years) or yearly in the range of 0.05-0.25% of the building stock.

3 ASSESSED VALUE OF RESIDENTIAL PROPERTY

Effects of monetary value change over time (e.g. inflation) are measured by price indexes; figure 1 shows a comparison between three indexes: home price-, building cost- and wage indexes from 1994 to 2010. As can be seen, there is a certain degree of correlation, though the hump in the home price index is striking during the boom period 2004-2008. Moreover, it is noteworthy that the building cost index did not take a corresponding jump at that time. Thus it seems that the home price index follows other indexes, as confirmed in [5] with the words: “But for the last 40 years, the increase in the long-term secular trend of real estate prices above general price levels has generally been a little over 1% on average.” In [6] a foreign example in traced where the yearly increase is just above inflation.

Housing, at least residential housing, thus maintains its value, and in fact even more so. As the number of new buildings has been substantial until recently, some people might try to explain this by saying that the new buildings of the time lifted up the whole. There might well be some truth in this but it is by no means the main reason, as can be seen if the selling price of real estate is compared for various periods. Figure 2 shows the selling price per square
metre for each year of building based on all selling contracts in Reykjavík between 1994 and 2009, adjusted to prices in June 2010 by use of the home price index. The data is categorized according to year of building, and each period spans 10 years. In urban areas in Iceland it is customary that the land (building plot) is owned by the municipality and the price of a real estate thus does not include the price of land. In comparisons of real estate prices one must be aware that the price is dependent on many factors – such as location, size, condition, degree of finishing, appearance and planning – and prices can differ considerably between properties for these reasons.

Figure 1 Residential property-, building cost- and wage indexes from January 1994 to September 2010 (source: [7])

Figure 2 Reykjavik: Selling price of residential buildings (ISK/m² floor area) in the period 1994-2009, adjusted to price levels in June 2010; the mean value for each group is shown by a line marker. (processed from data in ref. [8])
The oldest residencies are often smaller than the newer ones, but are located nearer to the centre of the capital area where also the three biggest institutions; the countries two biggest universities and the National University Hospital are nearby. The location of the oldest districts is thus preferred by many and this can easily affect the prices of these residencies. As there are so many variables affecting the market price, at the moment no attempt will be made to analyse the data according to these variables but will instead only show the overall effect of the data as a whole. The figure shows that the newest buildings are slightly more expensive than the older ones, and the oldest buildings are not the cheapest. The spreading in prices for groups from different construction times is similar, and for the oldest group this should mean that location is not overly important factor affecting the market price. Thus the data definitely indicates that older buildings can maintain their value to the same extent as new buildings: depreciation (write-off) does not need to occur in a building if maintenance and replacement are carried out diligently.

4 WRITE-OFFS AND ESTIMATED ASSET HOLDINGS

The registered rateable value of a property is currently determined from the estimated new building cost and then this figure is depreciated based on the age of the building, in just the same way as is customary for machinery of different kinds. The depreciation for facilities is usually about 2% each year- and this results in that the market price can be considerably higher than the rated value in official statistics and the taxable value. This can easily be seen by comparison between registered value of a property and the quoted mean value according to sales information for the neighbourhood in which the property stands (see for instance the homepage of Íslandsbanki in collaboration with Datamarket, reference [9]). In the assessment of total asset holdings of the nation, which to a large extent are in the built environment, value is also quoted in terms of annual depreciation – the total value at today’s prices is quoted as ISK 6,000 billion, see Figure 3. Even though the annual investment in building construction has for the last 5 years been in the range of 150-200 billion ISK (price level in June 2010) the total asset holding decreases. Thus in official figures, no account is taken of the effect of property maintenance – maintenance which is, however, supposed to maintain performance and thus the value of a property.

In this respect, property is underestimated and the rated value gives the wrong impression of the real value. This is an unlucky situation in the current discussion on the debt status of individuals, companies and society as a whole. Moreover, it is unfortunate in real estate management if the purpose of maintenance is under-estimated financially as this could result in it being difficult to justify maintenance costs.
5 CONCLUSIONS

Market value of a property is affected by many different factors, of which location, areal, condition, appearance and spatial planning are probably the most important. Maintenance is needed to ensure performance over time, but at the same time it is sometimes questioned how well maintenance pays off. The stated design life of buildings is (in Iceland at least) often taken as the write-off period for a facility, and annual write-off therefore taken as 2% of building cost. This does not take into account the effect of average maintenance that is approximately of the same value. It is shown in the paper that average annual maintenance effectively ensures that market value of facilities is similar between age groups of buildings—it is therefore generally not correct to write off a facility value if appropriate maintenance is carried out.

ACKNOWLEDGEMENTS

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REFERENCES

CHEMICAL TREATMENT WITH OXALIC ACID TO IMPROVE THE DURABILITY OF CEMENT-BASED MATERIALS IN ACID ENVIRONMENTS

Célestine Voegel(1,2)*, Alexandra Bertron(1)**, Benjamin Erable(2)*** and Gilles Escadeillas(1)

(1) Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), 135, avenue de Rangueil, F-31 077 Toulouse Cedex 04, France
(2) Université de Toulouse, INPT, UPS, CNRS, Laboratoire de Génie Chimique, 4 Allée Emile Monso, 31432 Toulouse, France

* celestine.voegel@ensiacet.fr, **bertron@insa-toulouse.fr, ***benjamin.erable@ensiacet.fr

Abstract

This aim of the work was to improve the durability of cement-based materials exposed to acidic environments, using superficial chemical treatments of the hardened cement matrix. Our previous studies, focusing on the action of organic acids on the cement matrix, highlighted the protective effect of calcium oxalate salt formed during attack of the cement matrix by oxalic acid. It was shown that calcium oxalate was stable even at very low pH and seemed to act as a sealant for the cement matrix porosity. On the basis of these results, the chemical treatments implemented in the present paper consisted of impregnating cement matrices with solutions made of oxalic acid or mixes of oxalic and acetic acids. These treatments were carried out on CEM I cement paste specimens made with two water/cement ratios (0.4 and 0.27) in order to investigate the influence of the initial porosity of the matrix. After the treatments, the cement paste specimens were immersed in aggressive solutions of acetic acid with a pH of 4. The efficiency of the treatments was assessed by measuring mass losses and degraded depths. The chemical, physical and mineralogical changes in the cement pastes were investigated using XRD, EPMA and water porosimetry. In this work, the protective effect of oxalic acid, previously demonstrated for CEM I W/C=0.27, was confirmed for CEM I with W/C=0.40. The mix of acetic and oxalic acids in the treatment solution improved the protective effect of the treatment against further acid attack but the protection was not total.

Keywords: oxalic acid, calcium salt, calcium oxalate, porosity, protection, durability, acid attack

1 INTRODUCTION

The versatility of concrete is demonstrated by the many concrete infrastructures built around the world in every possible environment. However, aqueous environments, such as fresh water, sea water and ground waters, or industrial water, treated water and wastewater, are aggressive for cement-based materials. Such environments, enriched with chemical and/or
biological aqueous components, expose concrete to microstructural degradation and material breakdown. One solution for preventing degradation is to limit the diffusion of aggressive components through the cement-based material by reducing its porosity.

Larreur-Cayol’s study (2012) [1] on the impact of agricultural effluents on hardened cement-based material brought to light interesting chemical interactions between oxalic acid and the cement matrix. It showed that, when cement paste made of ordinary Portland cement with W/C 0.27 was attacked by oxalic acid at pH 1, no loss of mass and no degraded layer could be observed during the experiment. Investigations on microstructure showed that portlandite Ca(OH)$_2$ was dissolved sacrificially and led to the formation of calcium oxalate salt in the outer layer of the specimens. It was assumed that the protective effect of oxalic acid was linked with the physical properties the calcium oxalate, which enabled the porosity of the matrix to be sealed and led to its high chemical stability.

Singh et al. [2] incorporated oxalic acid in freshly mixed mortar or concrete in order to investigate the possible precipitation of calcium oxalate during setting and hardening and its effect on the durability of the cementitious material. They pointed out the efficiency of adding a binary mixture (1:3) of hydroxyethyl cellulose and oxalic acid to improve concrete properties (strength, hardness and fracture toughness). Oxalic acid improved resistance to acid attack (H$_2$SO$_4$ and HCl) because of the precipitation of calcium oxalate [2].

The aim of the present study is to improve the durability of cementitious materials in aggressive aqueous environments by exploiting the properties of oxalic acid and its calcium salts. The first aim was to progress in the understanding of the phenomenology of the reactions between oxalic acid and cement materials. CEM I pastes with water/cement mass ratios of 0.27 and 0.40 were immersed in oxalic acid solutions in order to evaluate the effect of the porosity on the protective effect of the acid. Next, cement pastes were treated by impregnation with chemical solutions made of oxalic acid - alone or mixed with acetic acid. After the treatments, the cement paste specimens were immersed in solutions of acetic acid with a pH of 4, acetic acid being known to attack the cementitious matrix ([3]–[7]).

The degradation kinetics were assessed by mass loss and degraded layer depth measurements. The chemical and mineralogical changes of the cement pastes were explored by Electron Probe Microanalysis (EPMA) and X-Ray Diffraction (XRD).

2 MATERIALS AND METHODS

2.1 Materials

CEM I 52.5 N [8] pastes were made with two water/cement ratios: 0.27 and 0.4. These two ratios were chosen in order to confer significant differences of porosity on the cement pastes tested. The porosity of the paste specimens, measured by water porosity intrusion, was 29% for W/C 0.27 and 38% for W/C 0.40.

The specimens were cast in cylindrical moulds 75 mm high and 25 mm in diameter. They were removed from their moulds 24 h after pouring and stored in water at 20 °C for 28 days. They were then subjected to the various treatments and/or immersed in the acid solutions for four weeks.
2.2 Aggressive solution: Organic acid data

2.2.1 Organic acids and their calcium salts

This study was conducted with oxalic and acetic acids. Acetic acid is aggressive to the cement paste matrix ([1], [3], [4], [6], [9]) whereas oxalic acid has been shown to be non-aggressive [1]. The chemical formulas, the polyacidity and the dissociation constants of the acids, together with the solubility, density and molar volume of the calcium salts are given in Table 1. Calcium acetate is soluble in water, whereas calcium oxalate monohydrate is insoluble.

Table 1: Properties of the organic acids and their calcium salts [10], [11]

<table>
<thead>
<tr>
<th>Acid</th>
<th>Acetic</th>
<th>Oxalic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical formula</td>
<td>C₂H₄O₂</td>
<td>C₂H₂O₄/C₂HO₄</td>
</tr>
<tr>
<td>Polyacidity</td>
<td>Mono-acid</td>
<td>Bi-acid</td>
</tr>
<tr>
<td>pKa (25°C)</td>
<td>4.76</td>
<td>1.23/4.19</td>
</tr>
<tr>
<td>Ca-salt</td>
<td>Formula</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ca(C₂H₃O₂)₂·H₂O</td>
<td>CaC₂O₄·H₂O</td>
</tr>
<tr>
<td></td>
<td>Ca(C₂H₃O₂)₂·2H₂O</td>
<td>CaC₂O₄·2H₂O</td>
</tr>
<tr>
<td>Solubility (g/100 mL)</td>
<td>43.6 (0 °C)/34.7 (20 °C)</td>
<td>Insoluble/insoluble</td>
</tr>
<tr>
<td>Molar volume (cm³/mol)</td>
<td>No data/No data</td>
<td>66.41/No data</td>
</tr>
</tbody>
</table>

It should be noted that calcium oxalate salt can theoretically be found in four forms: anhydrous, and mono-, bi or tri-hydrated. The anhydrous form has not yet been identified in cement paste exposed to oxalate. The most stable form is monohydrated calcium oxalate, called whewellite. This form is found mostly at low pH [12]. Arvaniti et al. precipitated monohydrated calcium oxalate on concrete seeds in supersaturated solution [13]. The bi-hydrated form, called weddelite, is less stable than the monohydrate ([1], [9]). These two forms were identified during oxalic acid attacks on cement paste ([1], [14]). Lajili et al. identified both whewellite and weddelite on cement pastes in lixiviating solution containing mainly glucose and the fungus Aspergillus niger [15]. The tri-hydrated form was found in degraded zone 4 of a fly ash and blast-furnace slag cement paste attacked by a solution of acetic and oxalic acids [16].

2.2.2 Composition of the acid solutions

2.2.2.1 Experiments aimed at a better understanding of the action of oxalic acid on cement paste

The paste specimens were immersed in two types of solutions:
- A solution of oxalic acid 0.280 M with a pH around 1, noted O.
- A mix of acetic acid and oxalic acid (2/3 – 1/3) with a total concentration of acid of 0.28 M and a pH around 1, noted AO.

The concentration of acid was chosen in accordance with previous studies ([1], [7], [17], [18]).

2.2.2.2 Experiments to develop protective chemical treatments of the cement paste

The treatment test included two phases: (1) the treatment impregnation phase, and (2) the attack phase, during which the treated specimens were exposed to an aggressive acid solution.

Two types of treating solutions were tested:
- an oxalic acid solution 0.28 M,
a mix of acetic (2/3) and oxalic (1/3) acids with a total acid concentration of 0.28 M as suggested by preliminary works showing the formation of large amounts of oxalate salt in such conditions (§3.1.2).

The impregnation phase consisted of the immersion of the cement paste specimens in the selected treating solution for 4 weeks.

The aggressive solution used in the second stage of the study was an acetic acid solution (0.280 mol/L) with a pH of 4 (addition of NaOH 0.056 mol/L). The pH condition was maintained at 4 by regular renewals of the solution [17]. The paste specimens were immersed in the acetic solution for 4 weeks.

### 2.3 Test method

#### 2.3.1 Monitoring of degradation parameters over time

The degradation kinetics of the specimens were evaluated by measuring the relative mass losses and depths of the altered layer over time. The mass loss measurements were carried out on two specimens. A phenolphthalein test was used to identify the degraded layer on the cross section of the specimens (change in colour of phenolphthalein for pH > 9.5). The cross sections were scanned and the images were exploited using the Videomet® software. Data provided in the paper were obtained from the average of twelve measurements distributed around the circumference of the slices [1].

#### 2.3.2 Degradation mechanisms

The chemical changes in the specimens after the immersion tests were analysed with an electron microprobe (Cameca SX 50, operating conditions: 15 kV, 20 nA). Details on the preparation and the specimens are given elsewhere [14]. The flat surface of the cylindrical specimens was analysed by XRD (Siemens D5000, Co cathode, 15 keV) after the immersion phase. Details are given in [7].

### 3 RESULTS AND DISCUSSION

#### 3.1 Action of oxalic acid on the cement paste

##### 3.1.1 Macroscopic observation

The outer layer of the specimens immersed in the oxalic acid and oxalic/acetic acid solutions for four weeks did not show any cracks or degradation visible to the naked eye. A thin white layer or some white stains were visible on the surface of the specimens immersed in the acetic/oxalic acid solution.

##### 3.1.2 Degradation measurements over time

After four weeks, no degraded depth layer was revealed by the phenolphthalein tests on the cement pastes immersed in the oxalic acid and the acetic/oxalic acid solutions. Figure 3 gives the relative mass variation over time. Mass gains are shown as positive and mass losses as negative. W/C 0.40 pastes exhibited immediate and significant mass gain in the test, whereas W/C 0.27 pastes showed no mass change over time or a slight mass loss (-0.6%) in the first 2 days of immersion and then a practically constant mass until the end (for the specimen immersed in the acetic/oxalic acid solution).
The porosity of the matrix seemed to have a strong effect on the action of oxalic acid. It may be supposed that higher porosity leads to the dissolution of portlandite and, later, the precipitation of larger amounts of calcium oxalate, the precipitation of which was identified by XRD, in the cement paste porous network. Moreover, the mass gain of W/C 0.4 paste in the acetic/oxalic acid mix was higher than in the oxalate acid solution, which also suggests a greater precipitation of calcium oxalate salt in the mixed solution. However, this phenomenon was not observed for W/C 0.27.

3.1.3 Degradation mechanisms

3.1.3.1 Chemical changes

Figure 2 shows the oxide composition in CEM I 0.40 specimens immersed in acetic/oxalic acid solution according to the distance to the surface in contact with the solution. Two zones can be observed in the specimen: zone 1, or the core, with the same composition as the control specimen, and zone 2, a 500 µm-thick outer layer, enriched in SO₃ and showing a slight decrease in CaO content.

At the surface of the specimen, a 300 µm-thick zone containing CaO only is observed. It can be assimilated to the white layer observed macroscopically, this layer being identified as calcium oxalate by XRD analyses. The chemical profiles of the other specimens (CEM I 0.27 and/or 0.4 in oxalic or oxalic/acetic solution) were similar to those presented in Figure 2, except that CEM I 0.4 specimens had a thicker salt layer at their surface than CEM I 0.27 ones.

3.1.3.2 Mineralogical changes

Monohydrated calcium oxalate, or whewellite, was identified at the surface of the W/C 0.4 and 0.27 cement paste specimens immersed in oxalic and acetic/oxalic solutions. No calcium...
oxalate di-hydrate was detected on the specimens immersed in the acetic/oxalic mix, contrary to Larreur-Cayol’s observations in the same conditions but with W/C 0.27 cement pastes [1].

3.1.4 Discussion

Bertron et al. [9] and Larreur-Cayol et al. [1] had highlighted the protective effect of oxalic acid toward CEM I pastes made with W/C of 0.27. This effect was confirmed in this study on cement pastes made with W/C 0.4. The amount of calcium oxalate salt seemed to be higher with W/C 0.4 pastes than with W/C 0.27 ones, which could be linked, at least partially, with the higher porosity of the matrix. Moreover, for W/C 0.4 pastes, the mix of acetic and oxalic acids seemed to enhance the precipitation of calcium salt. This could be explained by the presence of acetic acid which could act as a “calcium pump”, facilitating the release of calcium by the matrix - through the dissolution of hydrates - and thus the precipitation of calcium oxalate. The complementary behaviour of the two acids suggests the possibility of developing a treatment for concrete against aggressive aqueous environments.

The chemical profiles of the cement paste specimens immersed in the two types of solutions were similar to those obtained previously by Larreur-Cayol et al. ([9]). It was shown that calcium oxalate salt was formed from Ca(OH)_{2} dissolution in the outer layer (zone 2 on figure 3) and, as a first approach, at least a part of the C-S-H. The slight enrichment in sulphur in zone 2 has been observed many times in studies dealing with acid attack of cementitious materials and the various authors have attributed it to the precipitation of secondary, non-expansive ettringite [1], [14], [19]. Calcium oxalate is insoluble in water and is very stable even at low pH (pH < 1 in the present study). It forms an outer layer at the surface that protects the specimen even when acetic acid is present in the solution. We recall that attack by acetic acid alone leads to the complete decalcification of the cement matrix; calcium acetate precipitates neither in the matrix nor in solution [7]. Larreur-Cayol pointed out the importance of the calcium oxalate molar volume in the protection mechanism. An adequate molar volume of calcium oxalate seems to fill the porosity of the cement matrix of W/C 0.27 paste perfectly. In this work, the increase of porosity (W/C 0.40) did not prevent the protective behaviour of the calcium oxalate from being maintained.

3.2 Chemical treatment with oxalic acid

3.2.1 Macroscopic observation

After the treatment (immersion in oxalic acid or acetic/oxalic solutions), specimens kept their initial grey aspects, while presenting some white stains at their surface (Table 2). After immersion in the acetic acid solution (pH 4), they showed the typical aspect of material attacked by acetic acid, with an outer orange/brown layer, which could be easily removed [17].

3.2.2 Measurements of degradation over time

Figure 3 presents the degradation kinetics of the specimen during the treatment and the attack phases. The evolution of mass changes and degraded layer depths during the treatment phase were the same as those described in section 3.1.2. The evolution of mass losses and degraded depths of some non-treated CEM I 0.27 specimens has also been added on the graphs (dotted lines, caption: reference specimens). The corresponding data came from [18]. During the attack phase, the mass decreased and the degraded layer depths increased right from the beginning of the immersion in acetic acid. The kinetics was linear, suggesting that diffusion was not the predominant mode of reactive transfer during the attack. Mass losses
occurred with approximately the same slope for all the types of treatments. However, the degraded layer of the specimen treated with the acetic/oxalic solution was thinner than that of the specimen with oxalic treatment. It should also be noted that W/C ratio had no influence on the alteration kinetics for the specimens treated with oxalic acid alone, during the time of the experiment.

Table 2: Aspects of the cross sections of OPC specimens when tested with phenolphthalein after treatment (4 weeks) and attack phases (4 weeks)

<table>
<thead>
<tr>
<th>Acetic/oxalic treatment</th>
<th>Oxalic treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>W/C 0.40</td>
<td>W/C 0.27</td>
</tr>
<tr>
<td>After treatment</td>
<td>After attack</td>
</tr>
</tbody>
</table>

Figure 3: Alteration kinetics of specimens during the treatment and attack phases: a) relative mass variation, b) degraded layer depth. O: oxalic acid solution, AO: acetic-oxalic acid solution

The degraded layer thicknesses after 4 weeks of immersion in acetic acid of previously treated CEM 0.27 specimens were 1.6 mm for the oxalic acid treatment and of 0.8 mm for the acetic/oxalic acid treatment. These values are sensibly lower than those measured in previous studies carried out on CEM I 0.27 immersed in acetic acid without any preliminary treatment (> 2.4 mm, in 4 weeks of immersion) [18]. Nevertheless, the kinetics was different (proportional to the square root of time at the beginning of the test in the case of non-treated specimens). Consequently, long-term tests are now necessary to better evaluate the efficiency of the chemical treatments.

4 CONCLUSION

In this work, the action of oxalic acid on ordinary Portland cement materials was investigated. Oxalic acid was found to be non-aggressive in terms of degradation kinetics for
cement pastes made with W/C of 0.4 and 0.27, despite very low pH conditions (around 1). Formation of calcium oxalate was identified in the outer layer of the cement matrix. Moreover, calcium oxalate formation seemed to be enhanced in the acetic-oxalic (2/3-1/3) acid mix.

The protective effect of calcium oxalate salt was then explored. Two types of chemical treatment solutions were implemented: oxalic and acetic/oxalic acid solutions. The treated specimens were next immersed in acetic acid at pH 4. The treatments resulted in reduced degradation depths compared to non-treated cement paste specimens. Nevertheless, long-term experiments will be necessary to better evaluate the effects of the treatments implemented. Further studies will notably focus on mixes of acetic and oxalic acid and will explore the influence of the proportion of each acid on the treatment efficiency. Other treatment application methods, more suitable than immersion for construction applications (such as painting), will also be tested.

REFERENCES


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SYNTHESIS, CHARACTERIZATION AND DURABILITY OF SELF-CLEANING ANTIREFLECTION COATINGS FOR GLAZING APPLICATIONS

Tao Gao (1) and Bjørn Petter Jelle (2,3)

(1) Department of Architectural Design, History and Technology, Norwegian University of Science and Technology (NTNU), NO-7491 Trondheim, Norway – tao.gao@ntnu.no; arild.gustavsen@ntnu.no
(2) Department of Materials and Structures, SINTEF Building and Infrastructure, NO-7465 Trondheim, Norway – bjorn.petter.jelle@sintef.no
(3) Department of Civil and Transport Engineering, Norwegian University of Science and Technology (NTNU), NO-7491 Trondheim, Norway

Abstract
Monodisperse hollow silica nanospheres (HSNSs) were prepared via a template-assistant wet chemical method; their applications as self-cleaning antireflection (AR) coatings was discussed. The AR effect was originated from the hollow nature of HSNSs and the self-cleaning function was achieved by surface hydrophobization. Experimental results indicated good stability of the as-prepared coatings, which enables their potentials in window glazing applications.

Keywords: Antireflection, Self-cleaning, Hollow silica nanosphere, Durability, Surface property, Glazings

1 INTRODUCTION
The application of manufactured nanomaterials (MNMs) in building sector has attracted considerable attention in recent years due to the fact that various vital characteristics of construction materials, such as strength and durability, can be greatly enhanced, and, more importantly, new and useful properties can be imposed by using MNMs [1, 2]. For example, self-cleaning and antireflection (AR) are two important properties and have potential applications in solar cells, window glazings, curtain walls, and so on [3-5]. It has been reported that the self-cleaning and AR function can be readily achieved with hollow silica nanospheres (HSNSs) [6], which are also known for their predominant thermal insulation properties [7]. Hence, HSNSs may be used as a multifunctional material for window glazing applications, where an integrated, improved optical and thermal performance represents an interesting and desirable feature.

As one of the most important façade components, window glazings are intended to last for 20–50 years without significant degradation in performance (i.e. thermal, optical, and mechanical properties). Therefore, the long-term durability or stability of any coatings applied
to window glazings shall be evaluated, especially at the earlier stage of the development since different synthetic strategies with different process economics and environmental impacts can be employed. Apparently, the success of self-cleaning AR coatings in the building sector depends on not only the potential benefit that will be realized by the end user, but also a sophisticated design and selection of materials to make the coatings durable and affordable.

In this study, we report the synthesis, characterization and durability of self-cleaning AR coatings made of HSNSs. The porous and hollow nature of HSNSs enables a distinctive AR effect [8], which can be controlled by tuning the size of HSNSs. The self-cleaning function has also been achieved through surface hydrophobization. Preliminary tests indicate good durability of the as-prepared coatings, suggesting promising potentials in e.g. window glazings.

2 EXPERIMENTAL

2.1 Materials and samples

Monodisperse positively charged polystyrene (PS) nanospheres were prepared and thereafter coated with a thin layer of silica, as reported previously [8]. AR coatings on float glass substrates were prepared via spin-coating at 2000 rpm. An annealing treatment at 550°C for 3 h was performed in air to remove completely the PS nanosphere templates and to provide a good adherence between the HSNS coatings and the glass substrates. Figure 1 shows schematically the synthesis procedure used in this work.

![Figure 1. Experimental procedures for the synthesis of HSNS AR coatings.](image)

The self-cleaning function was introduced by surface hydrophobization, which was performed by a chemical vapor deposition of 1H,1H,2H,2H-perfluorodecyltrichlorosilane (FDTS) in an oven at 120°C for 10 h [9].

2.2 Characterization

Transmission electron microscopy (TEM, JEM-2010) and scanning electron microscopy (SEM, Zeiss Supra 55VP) were employed to characterize the morphology and microstructure of the as-synthesized products. Optical properties were measured on a Perkin Elmer Lambda 1050 UV/VIS/NIR spectrophotometer with a 150 mm Integrating Sphere Reflectance Accessory and measured from 290 nm to 2500 nm. Attenuated total reflectance (ATR) Fourier transform infrared (FTIR) spectra were recorded on a Nicolet 8700 FTIR Spectrometer (Thermo Scientific) with a spectral resolution of 2 cm⁻¹.
3 RESULTS AND DISCUSSION

3.1 AR effect of HSNS coatings

A template-assistant method has been used to prepare HSNSs in this work. The fact that monodisperse PS nanospheres can be readily prepared with controlled diameters and surface properties [7, 10, 11] makes them ideal template materials for the synthesis of HSNSs. In addition, the PS template nanospheres can be conveniently removed by calcination at high temperatures, during which a strong binding between the glass substrate and HSNSs may also be achieved (see Figure 1). This is of particular importance with respect to the long-term durability or stability of the HSNS coatings. It is worth noting that the methodology utilized in this work can also be extended to HSNSs with different sizes, from which different properties (optical, thermal, mechanical, etc.) may be obtained.

Figure 2 shows the general morphology of the as-prepared materials. The template, PS nanospheres, is monodisperse with mean diameter of about 200 nm (Figure 2a), which corresponds also to the inner diameter of HSNSs (Figure 2b). The shell thickness of HSNSs at the present experimental conditions is about 20 nm. Uniform coatings can be readily obtained via spin coating (Figure 2c). There are no obvious changes on the morphology of HSNSs after the calcination at 550°C, indicating a fairly good stability of HSNSs.

Figure 2. Microstructure of (a) PS templates, (b) HSNSs, and (c) HSNS AR coatings.

Thanks to their hollow nature, HSNSs usually have a reduced refractive index $n$ compared to that of the solid counterpart [8]:

$$n_{HSNS} = n_{silica} \cdot (1 - P) + n_{air} \cdot P$$

where $P$ is the porosity of HSNSs (i.e. the volume fraction of the inner cavity); $n = 1.46$ and 1.0 for silica and air, respectively. In this work, the as-prepared HSNSs have an intrinsic porosity of about 58%, which results in a reduced refractive index of about 1.2 for HSNSs. Moreover, the reflection $R$ at normal incidence and low absorbance is related to the refractive index of the material forming the air/material interface by the following [12]:

$$R = \left(\frac{1-n_m}{1+n_m}\right)^2$$

Although the reflection $R$ depends not solely on the refractive index, but also on other factors such as thickness of the coatings [12-14], a low refractive index material is in general desirable to optimize the AR effect. Obviously, HSNSs are of interest in this regard since their refractive index can be readily tuned, i.e. by controlling the porosity (see eq.(1)).
Figure 3 shows the reflectance and transmittance spectra of PS/glass, SiO$_2$ coated PS (i.e. PS@SiO$_2$)/glass, HSNSs/glass, and plain glass substrate for comparison. The as-prepared HSNSs show a reduced reflectance of 6.8% at 440 nm, compared to about 8.5% of plain glass. Comparing the reflectance and transmittance spectra it is seen that a part of the reduced reflectance of HSNSs on glass may be due to a slightly increased transmittance and an increased absorbance, as compared to plain glass. The ~20% AR effect is not significant if compared to the recent progress on AR coatings consisting of HSNSs [14]. This may be attributed to the relatively large size of the as-prepared HSNSs (i.e. outer diameter is about 250 nm), which is comparable to the wavelength of the visible light, resulting in strong light scattering that reduces the AR effect.

![Figure 3. Reflectance (a) and transmittance (b) spectra of plain glass, PS template, SiO$_2$ coated PS, and HSNSs, all on glass substrate, for comparison.](image)

One important feature that can be seen from Figure 3 is that, both the reflectance and the transmittance spectra of PS@SiO$_2$ (graph 3 in Figure 3) differ significantly from those of HSNSs (graph 4 in Figure 3), which is apparently due to the hollow nature of HSNSs. Moreover, the as-prepared PS template spheres in this work exhibit two main absorptions at 368 and 621 nm (graph 2 in Figure 3b), which shift to low energy side, 439 and 621 nm, respectively, after coated with silica (i.e. PS@SiO$_2$, graph 3 in Figure 3b). Such phenomena are probably analogous to those observed for silica coated metallic nanoparticles [15]. The fact that the silica coating affects also the corresponding reflectance of PS spheres (Figure 3a) indicates that PS@SiO$_2$ may be used as solar selective coating materials.
3.2 Self-cleaning effect of HSNS AR coatings

The self-cleaning effect of a surface can be achieved via different approaches; among those the most common method is surface hydrophobization [3]. In this work, FDTS with a long chain of hydrophobic C-F groups has been selected to modify the HSNS AR coatings [9, 10]. The involved reaction can readily be followed with FTIR spectroscopy, Figure 4. The HSNS AR coating shows typical absorption features related to SiO2; whereas after the hydrophobization several absorption bands related to C-F vibrations appear [16], indicating a successful implantation of hydrophobic groups in the HSNS AR coatings.

![Figure 4. FTIR spectra of HSNS AR coatings before (a) and after (b) the hydrophobization. The spectra are shifted vertically for clarity.](image)

The surface hydrophobization reaction changes also the AR effect of HSNS coatings. As shown in Figure 5, there is an enhancement of the AR effect after the hydrophobization process. It is probably related to the reduction of light scattering due to the presence of the organic coating on the top of HSNS AR coatings, of which, however, the details remain unclear at this stage.

![Figure 5. Reflectance spectra of HSNS AR coatings before (a) and after (b) the hydrophobization.](image)
It is important to point out that either a super-hydrophilic or a super-hydrophobic surface can enable the self-cleaning function [3, 5], though through different mechanisms. A super-hydrophobic surface can be used for self-cleaning window glazings and can remove surface contaminations with the help of rain water [3, 5]. In this work, it is found that the as-prepared HSNS AR coatings are super-hydrophilic when compared to the plain glass substrate, as shown in Figure 6a and 6b. Moreover, a super-hydrophobic surface can be readily obtained by reacting HSNS AR coatings with FDTS, as shown in Figure 6c. However, it is difficult at this stage to measure the contact angle of water droplets to demonstrate quantitatively the corresponding surface property of HSNS AR coatings.

3.3 Durability of self-cleaning HSNS AR coatings

The long-term durability or stability of self-cleaning HSNS AR coatings is crucial to their practical application. There are two main factors that are important to address, i.e. the HSNS/glass interface and the hydrophobic surface of HSNSs. For the former a good adherence between HSNS and the glass substrate is required. Annealing the PS@SiO$_2$ spheres at high temperature results in the formation of a “hard” HSNS coating on glass, where the stability of the HSNS/glass interface can be significantly enhanced, compared to the “soft” coatings made by, e.g. spin coating or layer-by-layer assembly. Figure 7 shows the results of a peel adhesion test by using 3M tapes, revealing clearly the substantially increase stability of the annealed coatings.
Figure 7. Stability test of HSNS AR coatings prepared by (a) spin coating and (b) spin coating after annealing at 550°C for 3 hours. Arrows indicate the edge between the original (untouched) and the test area. Scale bar: 1 mm.

It is also found that the hydrophobic surface of HSNSs is fairly stable. For example, no obvious degradations of the surface hydrophobicity have been observed after soaking the coated glass substrates in ethanol under ultrasonic irradiation for 30 min. The fairly good stability of the self-cleaning AR coatings made of HSNSs may suggest their potentials in window glazing applications. However, several relevant durability tests (e.g. accelerated climate aging and specific solar irradiation tests) should also be performed in future for detailed information [17, 18], which is of great importance for all newly developed MNMs with target applications in the building sector.

4 CONCLUSIONS

Self-cleaning antireflection (AR) coatings for window glazing applications have been prepared by using monodisperse hollow silica nanospheres (HSNSs) with a typical inner diameter of ~ 200 nm and a shell thickness of ~ 20 nm. The as-prepared HSNS coating shows a ~ 20% AR effect which is related to their hollow nature but relatively large particle sizes. The self-cleaning function is achieved via surface hydrophobization. The as-prepared self-cleaning AR coatings made of HSNSs have fairly good stabilities, suggesting potential applications in the building sector.

ACKNOWLEDGEMENTS

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REFERENCES


EVALUATION OF CHLORIDE ION PENETRATION METHODS IN CONCRETE WITH RECYCLED CONCRETE AGGREGATE AND RICE HUSK ASH

Marlova P. Kulakowski (1), Mariana B. Fedumenti (2), Claudio S. Kazmierczak (2) and Maurício Mancio (2)

(1) Grupo de Pesquisa em Materiais e Reciclagem - GMAT, Programa de Pós-graduação em Engenharia Civil – PPGEC, UNISINOS, São Leopoldo – marlovak@unisinos.br
(2) GMAT, PPGEC, UNISINOS, São Leopoldo – mariana.fedumenti@gmail.com; claudiok@unisinos.br; mancio@unisinos.br

Abstract

The use of recycled concrete aggregate as replacement of natural aggregate has been gaining increased attention due to the vast amounts of non-renewable raw materials used globally for concrete production. However, it has been shown that such recycled aggregate can increase concrete permeability and thus reduce its durability. Rice husk ash has been employed as a supplementary material in Portland cement concrete to enhance concrete sustainability and durability. The aim of this research was to study the influence of rice husk ash on the chloride penetration in concrete made with recycled concrete aggregate. Recycled concrete aggregate was used in amounts of 25% and 50% (as replacement of natural aggregate), while rice husk ash was employed as partial replacement of Portland cement in 10% and 20% rates. The materials’ resistance to chloride ingress was tested with the ASTM C1202 test method and with the NT Build 492 method, an alternative test has gained increased acceptance for being less aggressive than the still widely-used but often criticized ASTM procedure. The best performance was obtained in concrete with 50% of recycled concrete aggregate and 20% of rice husk ash. This concrete had its resistance against chloride ingress improved in average 91% when tested according to the ASTM C1202 and 64% when tested by the NT Build 492 method, as compared to concrete produced with 50% of recycled concrete aggregate without rice husk ash.

1 INTRODUCTION

Nowadays it is imperative that the construction industry look for more sustainable and eco-efficient initiatives in order to reduce its considerable environmental impact, mostly due to excessive waste of materials, energy consumption and related CO₂ emissions. It is generally
accepted that the use of waste materials and by-products to produce construction and building materials and components can reduce the CO$_2$ emissions and the extraction of non-renewable materials from natural sources.

In this context, recycled aggregates from concrete waste have been receiving special attention, and several recent studies have shown the viability of using recycled concrete aggregate up to the level of 50% in replacement of crushed basalt aggregates [1-3].

Other promising by-product is the rice husk ash (RHA) formed in the generation of energy using rice husk as biomass. It is characterized by its high pozzolanic activity when in presence of lime due its high amounts of amorphous silica and its high superficial specific area [4]. This ash is used as supplementary material in partial replacement of Portland cement or as active mineral admixtures, and it can significantly improve the compressive strength of concrete and its durability.

However, comprehensive studies focusing on the combined use of recycled concrete aggregate and rice husk ash are not available. Therefore, it is necessary to investigate its effect on the mechanical properties and durability of concrete. Considering that such characteristics are intrinsically related to the material’s properties, detailed studies on the penetration of aggressive agents that lead to the deterioration of concrete structures are required.

Among the most common aggressive agents, the effect of chloride ions is of particular concern due to its role on the corrosion of rebar in steel-reinforced concrete structures. It is important to understand how chloride penetration progresses and, most importantly, how to minimize chloride contamination in order to enhance durability and service life of our concrete structures. Usually chloride ion penetration studies rely on lab-scale accelerated test methods that can provide a result in a matter of just a few hours, making possible, for instance, a quick comparative evaluation of different concretes and an estimation of the materials’ durability and service life when exposed to aggressive environments. However, great care must be taken when choosing the test method to be used, as some procedures may be excessively aggressive and not as realistic as relatively slower but less aggressive alternatives [5-7].

Some of the most widely-used test methods have been criticized for being too aggressive and no longer bearing resemblance to real exposure conditions (such AASHTO T277 and ASTM C 1202) – particularly as a consequence of the high potential difference used to drive Cl$^-$ ions (60V) and the high temperatures reached during some experiments – while other methods are considered impractical due to the long time required to reach a result (such as AASHTO T259, which takes 90 days) [6],[8-9]. Thus, the evaluation and widespread adoption of alternative test methods that are less aggressive but still relatively quick and suitable for the evaluation of concretes with recycled aggregates is considered an important goal that still hasn’t been reached. Among less aggressive test methods, the procedures recommended by Andrade [5] and the Nordic test method NT Build 492 [11] are particularly sensible alternatives. NT Build 492 applies voltages as low as 10V, and results can be obtained in periods ranging from 6h to 96h (most commonly within 24h), depending on the characteristics of the concrete being evaluated.

In this way, the aim of the present research was to study the influence of rice husk ash on chloride penetration in concrete made with recycled concrete aggregate, using and comparing two different test methods (ASTM C 1202 and NT Build 492). Besides this, the relation between chloride ion penetration and compressive strength has also been investigated.
2 MATERIALS AND METHODS

The experiment design was planned with three controlled factors and each one was studied in three levels: water/binder ratio (0.42, 0.53 and 0.64); recycled concrete aggregate content (0%, 25% and 50%); and rice husk ash (10% and 20% cement replacement). The analyzed response variables were total charge passed, diffusion coefficient, and compressive strength.

2.1 Materials

A Portland cement with no pozzolanic or cementitious admixtures has been used for the experiments (type CP II-F, according to ABNT NBR 11578:1991). Cement characteristics are presented in Table 1.

Table 1: Cement characterization

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃ (%)</td>
<td>4.12</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>19.42</td>
</tr>
<tr>
<td>Fe₂O₃ (%)</td>
<td>2.60</td>
</tr>
<tr>
<td>CaO (%)</td>
<td>60.76</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>4.85</td>
</tr>
<tr>
<td>SO₃ (%)</td>
<td>3.03</td>
</tr>
<tr>
<td>Ignition loss (LOI) (%)</td>
<td>6.04</td>
</tr>
<tr>
<td>CaO Free (%)</td>
<td>2.89</td>
</tr>
<tr>
<td>Insoluble Residue (%)</td>
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<tr>
<td>Alkalies (%)</td>
<td>0.63</td>
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<td>Expansion - Le Chatelier test (mm)</td>
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<tr>
<td>Initial set (h:min)</td>
<td>03:20</td>
</tr>
<tr>
<td>Final set (h:min)</td>
<td>04:30</td>
</tr>
<tr>
<td>Water of normal consistency paste (%)</td>
<td>26.20</td>
</tr>
<tr>
<td>Blaine (cm²/g)</td>
<td>3,850</td>
</tr>
<tr>
<td>#200 (%)</td>
<td>3.4</td>
</tr>
<tr>
<td>#325 (%)</td>
<td>16.3</td>
</tr>
<tr>
<td>fc 1 day (MPa)</td>
<td>16.3</td>
</tr>
<tr>
<td>fc 3 day (MPa)</td>
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<tr>
<td>fc 7 day (MPa)</td>
<td>36.9</td>
</tr>
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<td>fc 28 day (MPa)</td>
<td>41.9</td>
</tr>
<tr>
<td>Specific gravity (g/cm³)</td>
<td>3.11</td>
</tr>
</tbody>
</table>

Rice Husk Ash (RHA), produced in a fluidized bed furnace used for energy generation by biomass combustion, was used as a mineral admixture. Its characterization is presented in Table 2, while Figure 1 presents RHA’s X-Ray diffraction results.

Natural quartz sand was employed as fine aggregate with a fineness modulus of 2.12, maximum size of 4.8 mm, and specific gravity of 2.55 g/cm³. As coarse aggregate it was used a crushed basalt, with nominal maximum size of 19 mm, fineness modulus of 6.78, and specific gravity of 2.67 g/cm³. The coarse recycled aggregate was obtained from crushing concrete waste from production of precast concrete slabs. The compressive strength of concrete was 35 MPa. The recycled coarse concrete aggregate presented a nominal maximum size of 19 mm, fineness modulus of 7.25, and specific gravity of 2.21 g/cm³. The water absorption curve of the recycled concrete aggregate showed absorption of 6.29% in 10 minutes and 10.34% after 24 hours.
### Table 2: Rice Husk Ash characterization

<table>
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<th>Parameter</th>
<th>Value</th>
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<td>Pozolanic activity index (%)</td>
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</table>

![Rice Husk Ash X Ray diffractogram.](image)

#### 2.2 Test methods
Two test methods have been used in order to evaluate chloride ion penetration, ASTM C 1202-12 and NT Build 492. The classification proposed by Nilsson et al. (1998) was adopted to evaluate the specimens tested according to the NT Build 492 procedures. Compressive strength was measured at 28 days, after moist curing.

#### 2.3 Concrete production
Both the replacement of cement by RHA and natural aggregate (NA) replacement by recycled concrete aggregate (RCA) were done in a mass% basis, while the relation between the volume of mortar (cement plus sand) and coarse aggregate was kept constant. Extra water was added in order to compensate for the high water absorption of the RCA. Based on previous studies [1-3], the water compensation rate adopted here was equivalent to half the absorption rate measured at 10 minutes, described earlier. The slump was fixed at 100±10mm, kept constant among all mixtures with the use of a polycarboxilate-based superplasticizer. The amounts of materials used (in kg/m$^3$) are presented in Table 2.
Table 2: Materials consumption (kg/m³).

<table>
<thead>
<tr>
<th>RHA (%)</th>
<th>RCA (%)</th>
<th>W/B</th>
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<th>RHA</th>
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<td>0.53</td>
<td>354</td>
<td>0</td>
<td>834</td>
<td>486</td>
<td>402</td>
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<td>50</td>
<td>0.64</td>
<td>298</td>
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<td>483</td>
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<td>20</td>
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<td>29</td>
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<td>49</td>
<td>882</td>
<td>483</td>
<td>400</td>
<td>262</td>
</tr>
</tbody>
</table>
3 RESULTS

Figures 3-5 present the results obtained for chloride penetration based on both test methods, for three levels of RHA and RCA replacements, and for a wide range of compressive strengths. In these figures, NT Build 492 results are expressed in terms of the diffusion coefficient, while ASTM C 1202 results are expressed as the total charge passing the samples. Figure 3 presents results corresponding to the control mixtures without recycled concrete aggregate (i.e. 0% RCA), while figures 4 and 5 present the results obtained for the concretes with recycled concrete aggregate (25% and 50%, respectively).

![Figure 1: Results of compressive strength and chloride ion penetration – 0% RCA.](image1)

![Figure 2: Results of compressive strength and chloride ion penetration – 25% RCA.](image2)

It can be observed in figure 3 that concretes with 10% and 20% RHA, and without RCA, present resistance to chloride ion penetration markedly improved as compared to the samples without RHA. Similarly, when natural aggregate is replaced by 25% and 50% RCA, chloride ion penetration is significantly reduced when the use of recycled aggregate is combined with either 10% or 20% RHA as a partial replacement to Portland cement (figures 4 and 5).

Regarding compressive strength, as expected, concretes with smaller w/cm ratios present higher compressive strengths and are typically more resistant to chloride ingress, a trend that can be easily observed for nearly all samples tested. However, it is worth noting that a simple specification of compressive strength does not guarantee a low diffusion coefficient or high durability. If we take concretes with strengths between 35 to 40 MPa, for example, resistance to chloride ion penetration varies widely depending on the percentage of RHA used in the mixture, or with the combined use of RCA and RHA.
The differences observed in the classification of concretes with 50% RCA (with 0% and 10% RHA) depending on the test method used, may be attributed to a combination of two main aspects. Firstly, a concrete with 50% RCA has a higher amount of paste, and therefore a higher content of hydration products, including aluminates that are capable of binding chloride ions therefore reducing the overall depth of chloride penetration. Secondly, test method ASTM C 1202 measures mostly the overall ionic movement as a function of the higher applied potential (that also increases temperature due to the Joule effect, which further affects ionic flow), and the use of RCA increases concrete porosity making it easier for ions (all ions, not just Cl\textsuperscript{−}) to move around. On the other hand, test method NT Build 492 uses smaller voltages that do not lead to excessive temperature increases. Its results are expressed in terms of the concrete’s diffusion coefficient (and not just overall charge passed), and it also includes physical measurements of chloride penetration (after the test, specimens are axially split and a silver nitrate solution is sprayed onto the freshly split sections). This way, it is possible to measure in a more direct manner the effect that a higher paste content may have on the binding of chloride ions, therefore reducing its penetration depth and therefore the calculated diffusion coefficient. This hypothesis may be confirmed, for instance, by analyzing the concentration of chloride ions extracted from different specimen depths, possibly aided by an investigation of the concrete microstructure after the tests.

The enhanced resistance to chloride ion penetration observed for the samples with RHA can be explained by the pozzolanic reaction between the amorphous silica from the ash and calcium hydroxide coming both from the cement hydration reactions and from the RCA, in addition to the filler effect and microstructure refinements provided by the RHA particles, which average about 6.2 μm. Both phenomena can enhance the transition zone between the aggregate and cement paste, which is consistent with the results obtained by Chindaprasirt et al. [12]. In addition, the partial replacement of cement by RHA may contribute to filling the voids within the RCA, thus reducing its porosity and improving its properties [13].

4 CONCLUSIONS

- Test method NT Build 492 seems to be less aggressive and a good alternative to test method ASTM C 1202, as the former does not present the high temperature increases commonly observed with the latter;
Results obtained with both methods indicate that the use of RHA enhances the resistance to chloride ion penetration in concretes produced with recycled concrete aggregate;

Concrete with markedly different levels of compressive strength can present similar resistance to chloride ion penetration, which further underscores the importance of the materials used and the composition of the concrete in the determination of such properties.

ACKNOWLEDGEMENTS

The authors thank FAPERGS, CNPq and CAPES for research funding and scholarship.

REFERENCES


PERFORMANCE OF THE COPPER TREATED, AND NATURALLY DURABLE WOOD IN LABORATORY AND OUTDOOR CONDITIONS

Miha Humar (1), Boštjan Lesar (2) and Nejc Thaler (3)

(1) Department of Wood Science and technology, Biotechnical faculty, University of Ljubljana, Ljubljana, Slovenia – miha.humar@bf.uni-lj.si
(2) Department of Wood Science and technology, Biotechnical faculty, University of Ljubljana, Ljubljana, Slovenia – bostjan.lesar@bf.uni-lj.si
(3) Department of Wood Science and technology, Biotechnical faculty, University of Ljubljana, Ljubljana, Slovenia – nejc.thaler@bf.uni-lj.si

Abstract
In present research, service life of wood exposed outdoor, above ground was investigated. These results of the field testing were compared with results of laboratory tests. In the first part of the investigation, Norway spruce wood specimens were impregnated with six different copper-ethanolamine based solutions, consisting of the following ingredients: Copper(II) sulphate, ethanolamine, quaternary ammonium compound, boric acid, and octanoic acid. Solutions of two different concentrations were prepared to achieve two different retentions. Impregnated and artificially aged specimens were exposed to two brown rot fungi (Gloeophyllum trabeum and Antrodia vaillantii) and one white rot fungus (Trametes versicolor) according to the EN 113 procedure. Additionally, leaching of the tested copper-ethanolamine based wood preservatives was performed according to the standard ENV 1250-2 protocol. Results of the laboratory tests showed that fixation of copper-ethanolamine based preservatives is significantly affected by its composition. Field test data and laboratory experiments clearly showed that addition of co-biocides to copper-ethanolamine solution is essential to ensure sufficient performance. Results of the field testing of copper based solutions showed that these treatments significantly prolong service life of wood, while untreated spruce wood is degraded in the period between 4 and 6 years.

1 INTRODUCTION
Copper based preservatives have been successfully applied for wood preservation for more than two centuries. In the past they had been combined with chromium compounds to enable fixation in wood and arsenic to improve performance against copper tolerant fungi and insects [1]. Due to arsenic toxicity, its use is no longer desired in the majority of European countries, thus it was generally replaced with boron. The situation changed further with the introduction of the Biocidal Products Directive [2]. BPD directive limits the use of chromium in wood preservatives and so the need for development of new solutions for fixation of copper arose

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Amines are one of the most appropriate replacements for chromium. Particularly ethanolamine is reported as the most promising amine source in several researches, and it is used already for some emerging preservative systems including alkaline copper quat (ACQ), copper dimethyl-dithio-carbamate (CDDC), copper hydroxyquinolinolate (Cu-HDO) and copper azole (CA) [4]. However, fixation of copper-ethanolamine based preservatives is still not comparable to fixation of copper-chromium ones. Emissions of copper from wood impregnated with copper-amine preservatives can be reduced with proper copper-amine molar ratio and addition of different hydrophobic agents. Octanoic acid (OA) is one of the chemicals that significantly decrease copper leaching from impregnated wood. This carboxylic acid has multiplicative effect, beside hydrophobic, OA forms new complexes with copper-amines which are less soluble in water and do not leach from wood as much. Additionally, octanoic acid has fungicidal effect itself, which results in improved quality of impregnated wood [5].

Although copper-amine preservatives are utilized for more than two decades [4], complete performance of this system is still not completely elucidated. One of the questions is, are the results of the laboratory testing comparable to the results of the field testing. In order to elucidate this question, our specimens were exposed to outdoor conditions and matched samples were assessed in the laboratory. The aim of this research was to obtain correlation between laboratory and field test for various copper treated materials, and to compare them with performance of naturally durable materials. Service life data will be the basis for carbon footprint calculation which will be one of the most important criteria for decision in green public procurements.

2 MATERIAL AND METHODS

2.1 Treatment solutions used

For impregnation, six different inorganic wood preservative solutions were used as resolved from tables 1, 3 and 4. They consist of the following ingredients: copper(II) sulphate, ethanolamine, alkyl dimethyl benzyl ammonium chloride, boric acid, and octanoic acid. For leaching test and fungicidal tests, samples were impregnated with copper concentration of 1%, while for double layer tests solutions of two different concentrations were prepared to achieve two different retentions as resolved from table 4. Impregnation was performed according to the full cell process (vacuum 0,2 bar, 20 min; pressure 9 bar, 120 min; vacuum 0,2 bar, 10 min). Uptake of treatment solutions was determined gravimetrically.

Table 1: Composition of various preservative solutions used in this respective experiment. Prior impregnation, aqueous solutions were diluted to achieve targeted retention.

<table>
<thead>
<tr>
<th>Preservative solution</th>
<th>Mass of the respective active ingredient in 1000 g of preservative solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CuSO₄·5H₂O (CuS)</td>
</tr>
<tr>
<td>CuS</td>
<td>39.3</td>
</tr>
<tr>
<td>CuEa</td>
<td>39.3</td>
</tr>
<tr>
<td>CuEaO</td>
<td>39.3</td>
</tr>
<tr>
<td>CuEaQO</td>
<td>39.3</td>
</tr>
<tr>
<td>EABQ</td>
<td>-</td>
</tr>
</tbody>
</table>

* alkyl dimethyl benzyl ammonium chloride
2.2 Wood decay test
After impregnation, specimens were conditioned for four weeks. Durability of steam sterilized wood impregnated with different biocidal solutions, and untreated wood samples, were determined according to the EN 113 [6] procedure with two brown rot fungi (Antrodia vaillantii and Gloeophyllum trabeum) and one white rot fungus (Trametes versicolor) on Norway spruce (Picea abies) wood samples of dimensions 15 mm × 25 mm × 50 mm. After 16 weeks of fungal exposure specimens were isolated and mass losses were gravimetrically determined and expressed in percentages of the initial mass.

2.3 Leaching tests
Leaching was performed according to the modified ENV 1250-2 [7] procedure. In order to speed up the experiment, two modifications were applied: three specimens instead of five were placed in one (single) vessel and water mixing was achieved with shaking on a non-rotatory shaker instead of a magnetic stirrer. Nine specimens per concentration/treatment were put in three vessels (three specimens per vessel) to have three replicate leaching procedures. Afterwards, specimens in the vessel were positioned with a ballasting device that prevents them from floating. 300 g of distilled water were added and the vessel with its content was shaken with the frequency of 60 min⁻¹. Water was replaced six times in four subsequent days as proposed by standard ENV 1250-2 [7]. Leachates from the same vessel were collected and aggregated. Afterwards, X-ray fluorescence spectroscopy (Twin-x; Oxford instruments) analysis of the leachates was performed. Percentages of leached Cu were calculated from the amount of retained Cu determined gravimetrically and amount of Cu in collected leachates.

2.4 Performance of wood in outdoor conditions
In parallel spruce wood specimens were exposed in use class 3 (outdoor use, above ground) conditions according to the double layer test method [8, 9]. 10 specimens were grouped together in double layer test setup 500 mm above ground. Decay was visually evaluated and rated as prescribed by EN 252 standard [10] (Table 2). For comparison, besides copper treated, durable (English oak – Quercus sp.) semi durable (Larch – Larix decidua) and non durable (common beech – Fagus sylvatica) wood species were exposed as well.

Table 2: Grading system for decay [8,10].

<table>
<thead>
<tr>
<th>Rating</th>
<th>Descr.</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Sound</td>
<td>No evidence of decay. Change of colour without softening has to be rated as 0.</td>
</tr>
<tr>
<td>1</td>
<td>Slight attack</td>
<td>Visible signs of decay, but of very limited intensity or distribution: changes which only reveal themselves externally by very superficial degradation, softening of the wood being the most common symptom, to an apparent depth in the order of one mm</td>
</tr>
<tr>
<td>2</td>
<td>Moderate attack</td>
<td>Clear changes to a moderate extent according to the apparent symptoms: changes which reveal themselves by softening of the wood to a depth of approximately 1 to 3 mm over more than 1 cm² per stake.</td>
</tr>
<tr>
<td>3</td>
<td>Severe attack</td>
<td>Severe attack - marked decay in the wood to a depth of more than 3 mm over a wide surface (more than 20 cm²) or by softening deeper than 10 mm over more than 1 cm² per stake</td>
</tr>
<tr>
<td>4</td>
<td>Failure</td>
<td>Impact failure of the stake</td>
</tr>
</tbody>
</table>
3 RESULTS AND DISCUSSION

One of the main criteria of wood preservatives is their efficacy against target organisms. In Central Europe, fungi are predominant reasons for failure of wooden constructions [11]. Therefore, this manuscript focuses predominantly in the fungicidal resistance of the copper-amine preservatives, although insecticidal properties are expressed as well [12]. The second criterion that indicates the quality of wood preservatives is leachability. Low leachability is important to ensure efficacy, as well for environmental reasons.

The data presented in the Table 3 shows that copper treatment, without fixatives, does not enable sufficient protection against wood decay fungi, as the majority of the copper is removed from wood in the process of leaching. The remaining copper is not sufficient to control brown rot fungi (*G. trabeum* and *A. trabeum*), but it can ensure protection against copper sensitive fungus *T. versicolor*. Addition of the ethanolamine considerably reduces copper leaching, what clearly reflects in the increased performance against *G. trabeum*. However, copper itself is not sufficient to stop the growth of the copper tolerant *A. vaillantii*. In order to stop this fungus, addition of secondary biocides is required. There were two secondary fungicides applied in this research. One was boron, which primarily role was to control the insects. Presumably, most of the boron leached from wood during artificial ageing, and the remaining concentration was not sufficient to stop growth of *A. vaillantii*. Therefore, quaternary ammonium compound (ADBAC) was introduced in the second step. From the data in table 3, it can be resolved, that only combination of these compounds ensured sufficient fixation of copper biocides and on the other hand guarantees sufficient performance against wood decay fungi. Besides already mentioned ingredients, octanoic acid was introduced into the preservative solution as well. The main reason for this is the fact, that octanoic acid improves fixation of copper ethanolamine preservative solutions in wood. Octanoic acid forms complexes with copper-amine that are much more leaching resistant compared to the normal copper-ethanolamine complexes [5]. Furthermore, octanoic acid makes surface more hydrophobic, what reduces leaching as well. It is rather interesting, that the aqueous solution without copper (EaOQ), protects wood against wood decay fungi to rather good extent. This indicates, that quaternary compound is fixed in wood and does not leach from wood even during severe leaching procedure like EN 84. Standard EN 84 is standard that is used for artificial ageing of the wood or treated wood prior biological testing. This standard is rather severe as it includes vacuum treatment as the first step, following 14 subsequent immersion days. Thus it is much more severe that 4 days lasting ENV 1250 standard leaching protocol.
Table 3: Leaching of copper ingredients (cCu = 1%) from impregnated specimens determined according to the standard procedure (SIST EN 1250-2) and mass losses of impregnated and leached (SIST EN 84) Norway spruce wood specimens exposed to wood decay fungi according to mini block procedure. Standard deviations are given in the parenthesis.

<table>
<thead>
<tr>
<th>Preserv. solution</th>
<th>Composition of preservative solution</th>
<th>Percentage of leached Cu (%)</th>
<th>Mass loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cu sulphate Ethanolamine Octanoic acid Boric acid ADBAC</td>
<td>Gloeophyllum trabeum Antrodia vaillantii Trametes versicolor</td>
<td></td>
</tr>
<tr>
<td>CuS</td>
<td>X</td>
<td>48.2 (1.5)</td>
<td>14.4 (4.3) 27.2 (3.1) 2.2 (0.2)</td>
</tr>
<tr>
<td>CuEa</td>
<td>X X</td>
<td>3.5 (0.2)</td>
<td>9.3 (3.3) 29.8 (4.3) 1.0 (0.5)</td>
</tr>
<tr>
<td>CuEaO</td>
<td>X X X</td>
<td>2.4 (0.3)</td>
<td>1.4 (0.4) 20.9 (4.2) 1.3 (0.2)</td>
</tr>
<tr>
<td>CuEaOB</td>
<td>X X X X</td>
<td>3.2 (0.1)</td>
<td>1.5 (0.3) 20.5 (2.3) 1.1 (0.4)</td>
</tr>
<tr>
<td>CuEaOQ</td>
<td>X X X X X</td>
<td>4.1 (0.4)</td>
<td>0.4 (0.4) 0.2 (0.1) 0.4 (0.1)</td>
</tr>
<tr>
<td>EaBQ</td>
<td>/</td>
<td>/</td>
<td>0.0 (0.1) 0.3 (0.2) 1.4 (0.9)</td>
</tr>
<tr>
<td>Spruce</td>
<td>/</td>
<td>/</td>
<td>32.1 (5.6) 20.1 (3.7) 18.4 (4.1)</td>
</tr>
<tr>
<td>Beech</td>
<td>/</td>
<td>/</td>
<td>48.2 (2.7) 17.6 (1.5) 42.2 (2.1)</td>
</tr>
<tr>
<td>Larch</td>
<td>/</td>
<td>/</td>
<td>16.1 (0.9) 3.7 (0.9) /</td>
</tr>
<tr>
<td>Oak</td>
<td>/</td>
<td>/</td>
<td>0.7 (0.2) 0.9 (0.6) 1.6 (0.4)</td>
</tr>
</tbody>
</table>

* alkyl dimethyl benzyl ammonium chloride

In the second step, we were interested, if performance of copper-ethanolamine based preservatives in laboratory is comparable to the performance in the outdoor conditions. Performance of the copper-ethanolamine preservatives was compared to the performance of classical copper-chromium based solution and with naturally durable wood species like English oak and semi durable European larch. When comparing laboratory and outdoor data, it should be considered, that in the laboratory impregnated specimens were exposed to the pure fungal cultures in sterile conditions, while in the outdoor tests synergistic effect of abiotic and biotic factors influences the performance of the impregnated wood. Wood is not exposed to fungi and leaching alone, but also to temperature differences, bacteria, UV-degradation… Therefore, only field testing reflects real performance of impregnated and non-impregnated-wood.
Table 4: Retention of copper-ethanolamine preservative solutions and performance in double layer application. Samples were exposed outdoor between 7. 4. 2006 do 2. 7. 2013.

<table>
<thead>
<tr>
<th>Preservative solution / wood species</th>
<th>Target retention (kg/m$^3$)</th>
<th>Retention (kg/m$^3$)</th>
<th>2007</th>
<th>2008</th>
<th>2009</th>
<th>2010</th>
<th>2011</th>
<th>2012</th>
<th>2013</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuEaQO</td>
<td>4.9</td>
<td>4.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
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<tr>
<td></td>
<td>19.6</td>
<td>18.1</td>
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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>CuEaO</td>
<td>1.2</td>
<td>1.3</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.1</td>
<td>1.3</td>
<td>3.4</td>
<td>3.6</td>
</tr>
<tr>
<td></td>
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<tr>
<td></td>
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<td>12.7</td>
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</tr>
<tr>
<td>CuS</td>
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<td>0.0</td>
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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.1</td>
<td>0.8</td>
</tr>
<tr>
<td>CuEa</td>
<td>3.4</td>
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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
<td>0.3</td>
<td>0.6</td>
<td>0.5</td>
<td>0.8</td>
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<td>0.0</td>
</tr>
<tr>
<td>EaBQ</td>
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<td>0.0</td>
<td>0.0</td>
<td>0.1</td>
<td>0.4</td>
<td>1.4</td>
<td>3.4</td>
</tr>
<tr>
<td></td>
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<td>0.5</td>
<td>0.5</td>
<td>0.7</td>
<td>0.8</td>
</tr>
<tr>
<td>CCB</td>
<td>4</td>
<td>4.1</td>
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<td>0.0</td>
<td>0.0</td>
<td>0.1</td>
<td>0.1</td>
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<tr>
<td>Spruce 06*</td>
<td>-</td>
<td>-</td>
<td>0.2</td>
<td>0.8</td>
<td>1.8</td>
<td>2.8</td>
<td>3.2</td>
<td>3.6</td>
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</tr>
<tr>
<td>Spruce 07**</td>
<td>-</td>
<td>-</td>
<td>1.4</td>
<td>2.5</td>
<td>3.7</td>
<td>4.0</td>
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<td>/</td>
</tr>
<tr>
<td>Spruce 08***</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>0.0</td>
<td>0.2</td>
<td>0.4</td>
<td>1.9</td>
<td>2.2</td>
<td></td>
</tr>
<tr>
<td>Larch</td>
<td>-</td>
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<td>-</td>
<td>0.0</td>
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<tr>
<td>Beech</td>
<td>-</td>
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<td>2.5</td>
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<td>3.3</td>
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</tr>
<tr>
<td>Oak</td>
<td>-</td>
<td>-</td>
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<td>0.1</td>
<td>0.0</td>
<td>0.4</td>
<td>1.1</td>
<td></td>
</tr>
</tbody>
</table>

Spruce 06*- Control, un-impregnated specimens exposed 7. 4. 2006
Spruce 07** - Control, un-impregnated specimens exposed 28. 5. 2007
Spruce 08*** - Control, un-impregnated specimens exposed 19. 6. 2008

Results presented in table 4 show, that impregnation of wood with tested wood preservative solutions considerably improve performance of impregnated spruce in outdoor conditions. Un-impregnated spruce was exposed in outdoor tests in different time frames in spring of 2006, 2007 and 2008. It is rather surprising, that one specimen from the 2006 is still exposed in the field test site, while all the specimens that were exposed in the field test site in the 2007 were completely decayed after 4 years of exposure. Similar performance was determined at beech wood specimens as well. Beech wood was completely degraded after 6 years of exposure (Table 4). Decay of beech wood is a bit slower than decay of spruce, as beech wood is denser and so fungi need more time to consume the material. In contrast; decay on larch and oak wood samples is significantly slower. First signs of decay are visible after 5 years, while more prominent decay appears on the oak wood after 6 years of exposure. This clearly confirms better decay resistance of those two materials compared to spruce, for above ground applications. This data is in line with reports in the EN 350 [13] standard as well.

All treatments significantly increased performance of impregnated wood in use class 3 applications. However, there are considerable differences between tested compositions. For example, spruce wood treated with preservative solution based on boric acid and quaternary ammonium compounds (EaBQ) had its lifecycle prolonged only for a few years, but after
leaching of the boric based ingredients from wood and potential bacterial degradation of quaternary ammonium compounds, decay started. After 7 years of exposure, samples impregnated with the lowest retention of the solution EaBQ were completely degraded. Similar influence of copper leaching was determined with copper sulphate (CuS) treated wood. As long as at least low copper contents were present in wood, wood was protected against wood decay fungi [14]. When copper is leached, decay proceeds, similarly as determined in the laboratory specimens after artificial ageing. Furthermore, at specimens impregnated with higher Cu concentrations (retention = 6.5 kg/m$^3$), decay was delayed in comparison to the samples with lower Cu retention (retention = 1.1 kg/m$^3$). This was expected and is in line with the laboratory results, where lower concentration of copper did not prevent but even promote fungal growth [12]. Even if copper is combined with ethanolamine, it does not ensure complete protection, if retention is to low (retention = 2.1 kg/m$^3$ of CuEa or 1.3 kg/m$^3$ of CuEaO). Field test data clearly shows that in order to ensure protection of copper ethanolamine preservatives, copper must be combined with additional co-biocides like quaternary ammonium compounds. Good combination of biocides resulted in sufficient protection of wood even in case the stakes were impregnated with solutions of the lowest concentration. However, it has to be considered that double layer exposure offers extremely good conditions for fungal development, as there is water trapped between the samples. Without water traps considerably better performance is expected. At vertically exposed stakes, there are no signs of decay after four years of exposure even at susceptible beech or spruce wood specimens. Double layer test, however, represents the worst case scenario and should be interpreted as such.

4 CONCLUSIONS

Service life of the wood is influenced by several parameters. In use class 3 conditions, service life of Norway spruce wood is between 4 and 6 years. Similar service life is determined for beech as well. On the other hand larch wood and oak heart wood expressed insignificant signs of microbiological degradation after four years of exposure. Impregnation of wood with copper based biocides increased performance of the impregnated wood significantly. However, if leachable biocides are used, or if biocides in to low retentions are applied, decay is not prevented but only delayed for 1 to 3 years. One of the most important findings of this research was that laboratory experiments offer good estimation of the outdoor performance if applied correctly.

Table 5: Comparison of durability classes according to CEN/TS 15083-1 (2005) and x values calculated as relative median service life of test specimens in field tests.

<table>
<thead>
<tr>
<th>Preserv. solution</th>
<th>Gloeophyllum trabeum</th>
<th>Antrodia vaillantii</th>
<th>Trametes versicolor</th>
<th>Field tests</th>
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<tr>
<td></td>
<td>Low retention</td>
<td>High retention</td>
<td>Low retention</td>
<td>High retention</td>
</tr>
<tr>
<td>CuS</td>
<td>3</td>
<td>4</td>
<td>1</td>
<td>4</td>
</tr>
<tr>
<td>CuEa</td>
<td>2</td>
<td>4</td>
<td>1</td>
<td>3</td>
</tr>
<tr>
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<td>4</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>CuEaOQ</td>
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<td>5</td>
<td>4</td>
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</tbody>
</table>
ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the Slovenian Research Agency within the framework of project L4-5517 and programme P4-0015.

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[12] Pohleven, F. and Humar, M., 'Addition of pineon compounds and octanoic acid for improvement of biocidal properties and copper fixation at copper-ethanolamine based wood preservatives' in 'International Resarch Group for Wood Protection', (Stockholm, 2006), IRG/WP 06-30408


FROM LAB SCALE TO IN SITU APPLICATIONS _ THE ASCENT OF A BIOGENIC CARBONATE BASED SURFACE TREATMENT

W. De Muynck (1,2), N. Boon (2) and N. De Belie (1)

(1) Laboratory Magnel for Concrete Research, Ghent University, Ghent – willem.demuynck@ugent.be, nele.debelie@ugent.be

(2) Laboratory of Microbial Ecology and Technology, Ghent University, Ghent – nico.boon@ugent.be

Abstract
Throughout the last decade, many authors have considered microbially induced carbonate precipitation as a promising technique to overcome shortcomings of conventional surface treatments. Despite high expectations and encouraging results from laboratory tests, biodeposition has hardly been applied in practice. So far, only one bacterial based treatment has been made commercially available. The rather low consolidation performance of this treatment together with its high product and labour costs may account for the limited spread of this technology. This paper presents our efforts to make the biodeposition treatment a highly performing and economically feasible technique and describes the parameters that affect the performance of the treatment. It summarizes the findings from several years of research indicating the advantages of a urea based pathway. By altering the product composition and application procedure we succeeded in optimizing the distribution of the biogenic crystals throughout the stone. The strengthening effect of the biodeposition treatment was evaluated by means of drilling resistance measurements and was compared to that of conventional surface treatments, both being applied under laboratory and in situ conditions. From this study, it is clear that our urea based biodeposition treatment performs well when applied in situ and can be offered at a reasonable price.
1 INTRODUCTION

Despite several decades of research on stone conservation, the quest for an effective CaCO$_3$ based surface treatment for calcareous stone remains ongoing. The application of such calcinogenic consolidants is considered to be a preferable conservation strategy, since they share physico-chemical affinity with the substrate to be treated.

Different strategies have been explored for the re-introduction of calcite into the pores of the stone, the lime-water treatment being one of the oldest and most well-known techniques. In the latter, formation of calcite is a result of the reaction of lime with atmospheric CO$_2$. This technique has been applied to wall painting mortars and deteriorated calcareous stones in order to impart a slight water repellent and consolidating effect [1].

With the advent of nanotechnology in the last decade, there has been a revival in the use of lime as surface treatment for stone. The use of lime nanoparticles has been claimed to overcome the problems related to the use of traditional lime, i.e. poor solubility and fast sedimentation rate resulting in a high number of applications required [2]. Larson et al. [3] combined the use of calcite nanoparticles with lime in order to decrease the number of applications and drying time required with the lime water treatment. The strength obtained with these nanoparticles treatments, however, was limited to the outer surface.

Another technology that gained considerable attention in the last decade is the use of biologically induced precipitation of carbonate in building materials [4]. Biogenic crystals may form a protective layer on the surface and act as a cementing layer between the grains of the stone increasing its cohesion [5]. Despite many efforts by several research groups, so far only one biological treatment is commercially available. This so called Calcite Bioconcept treatment was already patented more than two decades ago [6].

Similar to the lime based treatments, most of the biodeposition treatments fail to meet the basic performance requirements of a consolidant. Because of their limited penetration depth they exhibit little or no improvement of the mechanical properties of the stone.

This paper summarizes our attempts to develop an efficient biogenic carbonate based surface treatment for limestone. Initial research was focused on the microbiological and chemical factors of the precipitation process. Amongst different enzymes (plant or microbial based) and metabolic pathways screened, the hydrolysis of urea with B. sphaericus appeared to be the most adequate treatment in a wide temperature range [7]. Contrary to other metabolic pathways [8], the hydrolysis of urea can be easily controlled and allows for the production of high concentrations of carbonate within a short amount of time. In a second stage, we directed our research efforts towards the problems most reported for inorganic consolidants, i.e. poor penetration abilities resulting in shallow and hard crusts and the formation of soluble salts as by-products [9].

Several parameters have been reported to influence the penetration depth of surface treatments [10]. In addition to examining the effect of climatologic conditions [7] and stone porosity [11], we have tried to optimize the penetration depth by altering the application procedure and the rate of carbonate deposition. Since transport of bacteria occurs in pores of which the diameter is at least two times that of bacteria [12], precipitation of carbonate at higher depths could be mainly observed for macroporous stone [11].

Our research indicated that the performance of the ureolytic biodeposition treatment was very dependent on the dosage of carbonate precursors [13] and the porosity of the stone [11], since both parameters affected the production and distribution of calcite crystals. As a
consequence, the consolidation of degraded and very porous stone would require substantial amounts of carbonate precursors, which would in its turn lead to the production of large amounts of by-products. In order to decrease the formation of soluble salts we proposed to replace a part of the carbonate precursors with nanoparticles. Our primary choice was the use of water-based CaCO$_3$ nanoparticles. Such particles, however, were not commercially available which required their in-house production [14]. Silica nanoparticles on the other hand, being water-based, commercially available and of interest for calcareous sandstone, were additionally investigated for their use as replacement of the carbonate precursors of the biodeposition treatment.

While a variety of durability parameters have been investigated, indicating the good protective performance of the biodeposition treatment (i.e. decreasing the uptake of water, and thereby, improving the resistance of the stone towards water-based degradation processes), this paper only reports on its consolidative effect, as evaluated by means of drilling resistance measurements.

2 MATERIALS AND METHODS

2.1 Limestone

Maastricht stone is a soft limestone with a total porosity up to 47% and a low compressive strength (3.5 N.mm$^{-2}$). Its softness enables a clear evaluation of a strengthening effect. In this study, 2 types of stone specimens were used, i.e. cubes (10 cm) and prisms (30 x 30 x 5 cm$^3$) for the surface treatments applied by pouring and spraying, respectively. Prior to the experiments, stones were dried at 80°C until constant weight (a weight change less than 0.1% between two measurements at 24 h intervals) was obtained. Then, all sides were covered with aluminium foil, except the one to be treated, to ensure that evaporation of water could only occur through the treated side. In case the treatments were applied by pouring, the foil was applied in such a way that it reached 2 cm above the surface that had to be treated. As such, loss of liquid during pouring was prevented.

2.2 Nanoparticles

Three types of nanoparticles were used in this study, i.e. calcium carbonate, calcium hydroxide and amorphous silica. Citrate-stabilized, water-based CaCO$_3$ nanoparticles (100-200 nm, 30 g.L$^{-1}$) were produced in-house according to the procedure described in [14]. Ethanol-based calcium carbonate (200-300 nm, 10 g.L$^{-1}$), ethanol-based calcium hydroxide nanoparticles (CaLoSil: 150 nm, 50 g.L$^{-1}$ and CaLoSil Micro: 1-3 µm, 120 g.L$^{-1}$) and water-based silica nanoparticles (Dispercoll S3030: 9 nm, 400 g.L$^{-1}$ and Dispercoll S5005: 55nm, 695 g.L$^{-1}$) were obtained from IBZ Salzchemie (Germany) and Bayer (Germany), respectively. All nanoparticles dispersions were applied to the stone in a dosage of 250 mL per cube, unless otherwise stated.

2.3 Biodeposition treatment procedure

Initially, all biodeposition treatments were applied in at least two steps, i.e. separate application of urease and carbonate precursors (i.e. urea and calcium salts). In a first series of experiments, biodeposition treatments were applied by pouring. The effects of the following parameters on the performance of the surface treatment were investigated: type and concentration of urease (microbial or plant based), type of calcium salt, concentration of
carbonate precursors, order of dosing urease and carbonate precursor solutions, dosage ratio of urea and carbonate precursor solutions, total dosage (L/m²) and number of applications. Over 100 different treatments were evaluated. The most promising ones were selected for the second series of experiments, i.e. application by spraying.

In the second stage of our study, the urease and carbonate precursors were applied in a single step after modification of the composition of the urease medium. Because of commercial interest, the composition cannot be disclosed. The combined liquid was applied bottom-up by means of an Alta 2000 spray apparatus (Dimartino, Italy) from a distance of 30 cm from the surface. The dosage of liquid applied per spray application amounted to about 2.2 L/m². In a final step, experiments were performed with industrially produced bacteria. Experiments were performed at 20±2°C and 65±5% relative humidity.

Additionally, several stones have been treated with the Calcite Bioconcept (CB) treatment to allow a clear comparison of the two types of biodeposition treatments. The CB treatment was applied under laboratory conditions at the Royal Institute for Cultural Heritage (Belgium) by J.F. Loubière (representative of CB). The stones were treated by immersion, capillary absorption (20 sec) or pouring (100 ml). A more detailed description of the CB treatment (oxidative deamination with B. cereus) can be found in [15].

2.4 Biodeposition combined with nanoparticles

Nanoparticles were applied to the stone by means of pouring or spraying, as described above. In a first series of experiments, biodeposition treatments were applied after stones treated with nanoparticles had obtained a constant weight (weight difference less than 0.1% over a 24 h interval when dried at room temperature). In a second series of experiments, nanoparticles were applied to the stone together with the urease source, followed later by the application of the carbonate precursors. The parameters investigated in this part of the study were the type and dosage of nanoparticles.

2.5 Conventional surface treatments

To gain a better insight into the efficiency of the bacterial treatments, results were compared to those obtained from conventional surface treatments. An ethyl silicate-based consolidant (KSE 300 HV) and a siloxane-based water repellent (Funcosil FC) were obtained from FTB-Remmers (Belgium).

2.6 Drilling resistance measurements

The strengthening effect was measured by means of the drilling resistance measurement system (DRMS Cordless SINT Technology, Italy). The system is equipped with a software program allowing the continuous recording and monitoring of the drilling resistance in relation to the advancement of the drill bit (Ø 4.8 mm). For this study, a rotation speed of 600 rpm and a penetration speed of 40 mm.min⁻¹ were used. The maximum penetration depth is about 3.5 cm. The results of the DRMS measurements are expressed as differential hardness profiles, obtained by subtracting the drilling forces measured after treatment from the reference values obtained on the corresponding untreated stone. For each type of treatment, 4 drilling measurements were carried out on each sample from which the average hardness profile was calculated.
3 RESULTS AND DISCUSSION

3.1 Consolidating effect of different types of nanoparticles

The strengthening effect of the nanoparticle treatment was clearly dependent on the type (carbonate or silica), the size and the dosage of nanoparticles used (Figure 1).

The highest strengthening could be observed for the silica nanoparticles (Figure 1C). Differences in diameter (9 vs. 55 nm) and viscosity (7 vs. 30 mPa.s) may account for the larger penetration depth of the 100 mL treatment with S3030 particles (± 25 mm) compared to the one with S5050 particles (± 13 mm). The larger particle size and density (1.39 vs 1.21 g cm$^{-3}$), on the other hand, are responsible for the higher strengthening obtained with the S5050 nanoparticles dispersion. With both types of silica nanoparticles, a significant and homogeneous strengthening could be observed up to 30 mm for the 250 mL treatment. The 250 mL treatment with S5050 particles exerted a higher consolidating effect compared to the ethyl silicate-based treatment. For both treatments, the increase in hardness can be attributed to the deposition of amorphous silica in the pores, the higher solids content of the S5005 treatment (i.e. 695 g/L) accounting for the higher strengthening compared to the ethyl silicate-based treatment (i.e. 300 g/L). While the primary function of the Funcosil treatment is to inhibit or decrease the uptake of water, the protective treatment also exerted a consolidating effect, although being limited to about 8 mm depth. Due to the deposition of large amounts of silica, resulting in an alteration of the porosity as observed from thin sections, stones treated with high dosages of nanoparticles were more prone to damage due to freezing and thawing.

![Figure 1: Strengthening effect of consolidation treatments with different types and amounts of nanoparticles (a: CaCO$_3$, b: Ca(OH)$_2$ and c: SiO$_2$) and of conventional surface treatments (d). All treatments were applied by pouring.](image_url)
The lowest strengthening was observed for the CaCO$_3$ nanoparticles (Figure 1A). The in-house produced (IHP) nanoparticles showed a tendency to aggregate which resulted in a poor penetration ability and in the formation of a white layer on the surface. The latter prevented the uptake of large volumes of nanoparticles dispersion. While the commercially available CaCO$_3$ dispersion exhibited a better stability, it was also unable to exert a noticeable consolidating effect (Figure 1A). Moreover, it also resulted in the deposition of a white carbonate layer on the surface of the stone.

The use of Ca(OH)$_2$ nanoparticles resulted in a very limited strengthening (Figure 1B). The slightly better performance of the lime nanoparticles dispersions than that of the in-house produced carbonate nanoparticles could be attributed to the smaller particle size and higher density of the former.

### 3.2 Consolidating effect of different types of biodeposition treatments

While the strengthening effect of the Calcite Bioconcept treatment was very limited and restricted to the first 2 mm, a significant consolidation up to 30 mm could be observed with the Ghent University treatment (Figure 2). Contrary to earlier work, where the biodeposition treatment resulted in the formation of hard crusts or an inhomogeneous hardness profile ([14] and [15]), we succeeded in obtaining a homogeneous strengthening by modifying the application procedure and the composition of the bacteria and carbonate precursor medium.

The biodeposition treatment with nanoparticles exhibited a strengthening that was higher than that of the sum of the two separate treatments. The latter can probably be attributed to the fact that the silica gel affected the transport of the bacteria, and hence, the distribution of the carbonate cement throughout the stone, improving the connection between the stone grains. For the treatments applied by pouring, the biodeposition treatment (Figure 2) exhibited a much lower strengthening compared to that of the ethyl silicate-based consolidant (Figure 1).

![Figure 2: Comparison of the strengthening effect of the Ghent university biodeposition treatment (a), with that of Calcite Bioconcept treatment (b). Influence of the addition of silica nanoparticles on the strengthening effect of a biodeposition treatment (c).](image-url)
3.3 Consolidating effect of spray-applied surface treatments

When applied by spraying, the biodeposition treatment exhibited a strengthening similar to that of the conventional ethyl silicate-based consolidant (Figure 3). Even a more homogeneous hardness profile was observed for the former, when only two spray applications had been performed. The smaller consolidation effect compared to the treatments applied by pouring can be largely attributed to the smaller dosage of product applied, i.e. about 1-3 times 2.2 L.m\(^{-2}\) compared to 1-2 times 25 L.m\(^{-2}\).

Figure 3: Comparison of the strengthening effect of the Ghent university biodeposition treatment, with that of conventional surface treatments. All treatments were applied by means of spraying.

4 CONCLUSIONS

By modifying the parameters that affect the penetration ability, we have succeeded to obtain a biogenic carbonate based surface treatment for stone that is cost-effective, able to homogeneously strengthen limestone up to depths of 30 mm and that has a similar or better performance than that of traditional surface treatments.

The replacement of carbonate precursors with silica nanoparticles presents a promising strategy to decrease the amount of by-products, but needs optimization prior to being used in practice. Because of more stringent regulations on the use of solvents, our water-based and compatible biodeposition treatment presents a valuable alternative to traditional solvent-based surface treatments. Moreover, our biodeposition treatment presents some advantages over the commercially available Calcite Bioconcept method. Whereas, the protective effect of the latter treatment is obtained after multiple spray applications over several days, our treatment entails both a significant protective and consolidating effect after two spray applications within the same day. The latter accounts for the lower cost of our treatment compared to that of the Calcite Bioconcept method. Furthermore, by optimizing the concentrations of the urease and carbonate precursor solutions, we were able to lower the cost of our treatment within the range of that of traditional consolidants.

To our knowledge, we are the first to offer a highly effective biodeposition treatment that combines the application of microorganisms and carbonate precursors in a single step methodology.
ACKNOWLEDGEMENTS

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REFERENCES.

MICROSCOPY AND COMPUTED MICRO-TOMOGRAPHY FOR EVALUATION OF MICROBIAL SELF-HEALING IN CONCRETE

J. Wang (1)(2)(3), K. Van Tittelboom (1) and N. De Belie (1)

(1) Magnel Laboratory for Concrete Research, Ghent University, 9052 Ghent, Belgium – jianyun.wang@ugent.be; kim.vantittelboom@ugent.be; nele.debelie@ugent.be;

(2) Laboratory of Microbial Ecology and Technology, Ghent University, 9000 Ghent, Belgium

(3) SIM vzw, Technologiepark 935, BE-9052 Zwijnaarde, Belgium

Abstract

Autonomous self-healing of materials, implies that the material is adapted in such a way that damage is repaired automatically. To obtain self-healing in concrete, several smart mechanisms can be introduced, such as activators or fibres to stimulate autogenous healing, encapsulated (pre)polymers or bacteria. The technology of microbial self-healing makes use of bacteria that promote the precipitation of calcium carbonate when they make contact with the appropriate nutrients and water. In order to survive the high alkalinity in the concrete matrix and the reducing pore sizes due to the ongoing cement hydration, bacterial cells or spores are applied, protected by a carrier material or encapsulation system. Crack formation in the concrete acts hereby as a trigger to break the capsules and activate the bacteria. The precipitated bacterial calcium carbonate will fill the crack and restore the water tightness of the concrete element.

The most obvious technique to evaluate the self-healing efficiency is a visual examination of the crack filling. Optical microscopy allows to evaluate crack filling at the concrete surface, whereas computed micro-tomography also allows to visualize the complete crack internally in a non-destructive way. The current study gives an overview of crack healing ratios and maximum healed crack widths for various microbial based self-healing strategies. Incorporation of Bacillus sphaericus cells, protected by diatomaceous earth, allowed healing of 0.17 mm wide cracks in mortar specimens in 40 days time. B. sphaericus spores, protected by melamine based microcapsules, were able to heal cracks of almost 1 mm in three weeks time. Encapsulation of spores in hydrogels resulted in healing of 0.5 mm cracks within one week. Computed micro-tomography enabled quantification of the amount of deposited calcium carbonate. Also crack filling by polymeric carrier materials for bacteria, such as polyurethane, could be evaluated with this technique.

KEYWORDS: microbial CaCO₃, crack, quantification, healing ratio
1 INTRODUCTION

Microbial-based self-healing concrete is regarded as a promising material to enhance the durability and sustainability of concrete structures. The principle is that mineral precipitating bacteria, more specifically carbonate precipitating bacteria, together with nutrients, are added into the concrete mix. When cracks appear, bacteria in the crack zone are expected to be activated and precipitate CaCO$_3$ to in-situ heal the cracks. Due to the harsh environment inside concrete, encapsulation of bacteria is preferable. In this research, three carriers were selected, diatomaceous earth, microcapsules and hydrogel, to immobilize bacteria and investigate the self-healing efficiency in cementitious specimens with incorporated biological healing agents.

Diatomaceous earth (DE) is a natural soft siliceous sedimentation. It is highly porous, chemically stable, and inert. DE has been mainly used as filtration agent and functional filler for paints and plastics, and as filling material for concrete. It is also a widely used bacterial carrier. The microcapsules used in this study are impermeable, resistant to the high pH of concrete, and humidity sensitive. They are flexible in high humidity (like in water) and become fragile in dry state. This implies that the capsules can withstand the forces during concrete mixing and can be easily broken upon cracking. Hydrogels are hydrophilic gels which have high water absorption capacity and can retain a large amount of water or aqueous solution in their network without dissolving. The water absorbed will be released slowly, which will be of benefit for bacterial activities and stimulate CaCO$_3$ precipitation.

During self-healing, CaCO$_3$ precipitation can be induced by a bacterial and/or autogenic process. It is difficult to differentiate the biogenic-CaCO$_3$ from abiotic-CaCO$_3$. In order to examine the healing effect from bio-precipitation, the crack filling was investigated qualitatively and quantitatively by use of light microscopy analysis on the specimens with and without bacteria added. Furthermore, to obtain a view of the healing products deep inside the specimens, a high resolution X-ray computed microtomography (X-ray µCT) was used. X-ray µCT is a non-destructive technique, which generates three-dimensional (3D) images by combining a series of cross-sectional images. It provides information (visualization and quantification) about the internal structure of the matrix. X-ray µCT has become a frequently used technique in material research [1-3].

2 MATERIALS AND METHODS

2.1 Bacterial strain and cultivation

*Bacillus sphaericus* LMG 22557 (Belgian coordinated collections of microorganisms, Ghent) was used in this study. This strain has a high urease activity (40 mM urea hydrolyzed.$\text{OD}^{-1}.\text{h}^{-1}$), long survival time and can produce CaCO$_3$ in a controllable way [4].

*B. sphaericus* was cultivated in a sterile growth medium that consisted of yeast extract (20g/L) and urea (20 g/L). The cultures were incubated at 28°C on a shaker at 100rpm for 24h. The living cells were harvested by centrifugation (7000 r/min, 7min, Eppendorf MiniSpin, Hamburg, Germany) and were resuspended in sterile saline solution. The concentration of the cells in the suspension was about 10$^9$ cells/mL.

*B. sphaericus* spores were cultivated in the liquid minimal basal salts (MBS) medium [5]. Mature spores were transferred as inocula (1%) into MBS medium. The cultures were incubated (28 °C, 100rpm) for 14~28 days till more than 90% of the cells were spores. The spores were then harvested by centrifuging the culture for 7min. The centrifuged spores were resuspended in the sterile saline solution. Subsequently, the suspension of the spores was subjected to pasteurization to minimize the amount of
vegetative cells in the culture. The concentration of the spores in the suspension was about $10^9$ spores/mL.

2.2 Mortar specimens with diatomaceous earth (DE) immobilized bacteria

2.2.1 Immobilization of bacteria into/onto DE

DE particles had sizes ranging from 4µm to 20µm with large amount of tiny pores (0.1µm to 0.5µm) on the surface. The bacterial suspension (BS, $10^9$ cells/mL) was mixed with DE powders (20%, w/v); the mixture was then put on a shaker (100 rpm, 28°C) for 1h to make bacterial cells attach to the DE surface.

2.2.2 Preparation of mortar specimens

Two series of mortar specimens (40mm x 40mm x 160mm) were made with a water to cement ratio of 0.5 and a sand to cement ratio of 3 (Table 1).

<table>
<thead>
<tr>
<th>Series</th>
<th>Cement (g)</th>
<th>Sand (g)</th>
<th>Water (g)</th>
<th>DE (g)</th>
<th>BS (mL)</th>
<th>Nutrients (g)</th>
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</tbody>
</table>

Table 1 Components of mortar specimens in each series

Nutrients included 1.25g yeast extract, 11.25g urea and 22.5g Ca(NO$_3$)$_2$·4H$_2$O

Reinforcement was added to the mortar specimens to control the crack width. A 10 mm mortar layer was first added into the moulds. After this layer was compacted by vibration, two reinforcements (D = 2mm, L = 140 mm) were placed on top of it. Afterwards, the moulds were completely filled with mortar and vibrated. All moulds were put in a climate room (20°C, > 95%RH) for 24h. After demoulding, the mortar specimens were placed in the same climate room.

2.2.3 Creation of cracks and incubation of cracked specimens

After 14 days, specimens were taken out of the moist room and cracks were created by a crack width controlled three-point bending test. Crack width was measured by a linear variable differential transformer (LVDT) which was attached at the bottom of the specimens. The final crack width created ranged from 0.15-0.17mm. Afterwards, three cracked specimens were immersed in water and three were immersed in a deposition medium (DM, made of urea and Ca(NO$_3$)$_2$, 0.2M) for 40 days.

2.3 Mortar specimens with microencapsulated bacteria

2.3.1 Encapsulation of bacterial spores into microcapsules

The bacterial spores were encapsulated in melamine based microcapsules following a patented polycondensation reaction based microencapsulation process (Patent WO 2010/142401) [6]. The size of the microcapsules was about 5µm. After encapsulation, an emulsion consisting of microencapsulated spores and water was obtained. The concentration of the spores in the microcapsules was around $10^5$ cells/g microcapsules (dry weight).
2.3.2 Preparation of mortar specimens

Two series of specimens (30mm x 30mm x 360mm, including a rebar \( r_b = 6 \text{mm} \)) were made and the composition is shown in Table 2. Group NC and NCS are the specimens with nutrients and the microcapsules which were (NCS) or were not (NC) loaded with spores. The specimens were stored in the same moist room.

<table>
<thead>
<tr>
<th></th>
<th>Cement (g)</th>
<th>Sand (g)</th>
<th>Water (g)</th>
<th>Nutrients (g)</th>
<th>Microcapsule emulsion (g)</th>
<th>Dry weight of Microcapsules (g)</th>
<th>Bacterial spores</th>
</tr>
</thead>
<tbody>
<tr>
<td>NC</td>
<td>450</td>
<td>1350</td>
<td>201.4</td>
<td>57.84</td>
<td>26.1</td>
<td>13.5</td>
<td>N</td>
</tr>
<tr>
<td>NCS</td>
<td>450</td>
<td>1350</td>
<td>192.8</td>
<td>57.84</td>
<td>34.7</td>
<td>13.5</td>
<td>Y</td>
</tr>
</tbody>
</table>

Nutrients included yeast extract, urea and \( \text{Ca(NO}_3\text{)}_2\cdot4\text{H}_2\text{O} \). The addition ratio was 0.85%, 4% and 8% of cement by weight. \( \text{Ca(NO}_3\text{)}_2\cdot4\text{H}_2\text{O} \) contains 30.5wt% water. Therefore the amount of mixing water was reduced by the amount of the water in \( \text{Ca(NO}_3\text{)}_2\cdot4\text{H}_2\text{O} \) and the amount of water in the microcapsule emulsion. The last column shows whether bacteria were present (Y) or not (N).

2.3.3 Creation of cracks and incubation of cracked specimens

28 days after casting, the specimens were subjected to a tensile test to create multiple cracks (Amsler 100, SZDU 230, Switzerland). A uniaxial tensile load was applied to the specimen at a speed of 0.01mm/s under stroke control. The loading was stopped at the point where the average crack width in the specimen reached 150 \( \mu \text{m} \). The cracked specimens were subjected to five incubation conditions (for 8 weeks): 1) 20°C, >95%RH; 2) immersion in water; 3) immersion in the deposition medium (DM); 4) wet-dry cycles with water; 5) wet-dry cycles with DM. During one wet-dry cycle, the specimens were immersed in water/DM for 16 h and were then exposed to air for 8h. The incubation of 2), 3), 4) and 5) was performed in an air-conditioned room (20°C, 60%RH).

2.4 Mortar specimens with hydrogel encapsulated bacteria

2.4.1 Encapsulation of bacterial spores in hydrogel

Bacterial spores suspension \( \left(10^9\text{ cells/mL}\right) \) was first mixed with a 20% w/w polymer solution (modified Pluronic®F-127). Then the initiator (Irgacure 2959) was also added to the solution. The mixture was degassed, mixed for 5min, and then subjected to UV radiation for 1h. A hydrogel sheet was formed, which was then subjected to freeze grinding and freeze drying to obtain the dry powders.

2.4.2 Preparation of mortar specimens

Three series of mortar specimens were made (R, H and HS). The reference specimens (R) only contained nutrients (the dosage can be seen in Table 2). The specimens with nutrients and pure hydrogels or spores loaded hydrogels were represented as H or HS, respectively. The addition of hydrogels was 2% of cement by mass. The size of the specimens were the same as those in 2.3.2. After casting, the specimens were stored in the same moist room.
2.4.3 Creation of cracks and incubation of cracked specimens

28 days after casting, the prisms were subjected to multiple cracking (similar as in 2.3.3). The cracked specimens were subjected to two incubation conditions (for 4 weeks): 1) 20°C, >95%RH; 2) wet-dry cycles with water, 1h in water and 11h exposed to air (20°C, 60%RH).

2.5 Visualization and quantification of crack filling by light microscopy

The cracked specimens were investigated under the light microscope (Leica S8 APO, Switzerland) once a week during the period of incubation to monitor the crack healing. For multiple cracking, the crack healing efficiency was evaluated by the absolute amount of healed crack area (for the ones with microcapsules) or the healing ratio which is the ratio between the healed crack width to the initial crack width (for specimens with hydrogels). Both crack area and crack widths were analyzed by the Leica software (LAS).

2.6 Quantification of crack healing by 3D X-ray μ-CT

The X-ray tomography images of cracked small cylinders (Φ=8mm, h=10mm) were scanned before and after the incubation under wet-dry cycles. The scanning was done by the Center for X-ray Tomography of Ghent University (UGCT). The images were analyzed by use of the reconstruction software package Octopus and the 3D analysis software package Morpho+ and VGStudio. In order to obtain a 3D distribution of the healing products, digital image subtraction was performed on the scans taken before and after the healing, resulting in a differential volume.

In addition, X-ray μ-CT was also applied to visualize the crack healing in specimens which contained glass capillary encapsulated polyurethane, which was used as the bacterial carrier in previous research [7].

3 RESULTS AND DISCUSSION

3.1 Crack healing in the specimens with DE immobilized bacteria

The specimens with pure DE incorporated had very limited crack healing. Almost no precipitation was formed in the cracks for specimens immersed in water (Fig.1(a)), while the specimens immersed in DM showed a small amount of precipitation in the cracks and on the surface (Fig.1(b)). No cracks were completely healed. The specimens with DE immobilized bacteria incorporated showed improved crack healing. In this case, cracks in specimens immersed in water were partly healed (Fig.1(c)); and cracks in specimens immersed in DM were completely healed by the precipitation (Fig.1(d)). The improved healing in DM is due to the additional supply of urea and Ca$^{2+}$ from the DM which allows the bacteria to produce more calcium carbonate. More information can be found in [8].

Theoretically, the healing agents are distributed all over the matrix. Therefore, the healing should be similar for different locations along the crack. However, it was noticed that only for some positions the cracks in the specimens of the bacterial series (immersed in water) had closed. Hence, to obtain more representative and comprehensive values for crack healing efficiency, in the following self-healing systems, multiple cracks were created to evaluate the self-healing efficiency.
3.2 Crack healing in the specimens with microencapsulated spores

The initial crack area was different in all specimens (about 70-140 mm$^2$) and in none of the specimens all cracks were completely closed after healing (Fig. 2). Therefore, the absolute amount of healed crack area in the specimens was calculated (details of crack area processing can be seen in [9]) and was used to evaluate the healing efficiency, which is also indicated in Fig. 2. Crack healing was observed in all specimens except the ones stored at 95%RH, which indicated that the presence of water is an essential constituent for self-healing. When stored at other conditions, the specimen NC and NCS had a healed crack area ranging from 20mm$^2$ to 40mm$^2$ and 60mm$^2$ to 80mm$^2$, respectively. The maximum crack width healed in the specimen NCS was 970µm (Fig. 3). The increased healing efficiency (at least 50%) was due to bacterial precipitation. It was found that the specimens subjected to wet-dry cycles with water as the immersion solution had the highest healing efficiency. During the wet stage, the specimens absorbed enough water which can keep the matrix in wet state during the dry stage. When the specimens were exposed to the atmosphere, more oxygen/CO$_2$ becomes available for the bacteria or for carbonation than in the case of continuous immersion. For the specimens with bacteria, a reduced immersion time could also decrease the escape of the bacteria from the crack surface and the leakage of the nutrients into the incubation solution. An optimal wet-dry cycle can promote the diffusion of nutrients from the internal matrix to the surficial cracking zone without leaching too much to the bulk solution and can provide sufficient available water for bacterial activities during the dry state.

Multiple crack healing efficiency, using healed crack area as the evaluation index, can give an overall estimation of the healing in the whole specimens. The disadvantage is that such an absolute value is dependent on the initial crack widths; while a relative value, such as healing ratio, is more representative.

Figure 1. Light microscopy images of the cracks in different specimens (a and b: specimen with only DE, immersed in water and in DM, respectively; c and d: specimens with DE immobilized bacteria, immersed in water and in DM, respectively. The black bar indicates 1mm. The images were based on [8])

Figure 2. Crack healing in specimens NC and NCS in different conditions

Figure 3. Maximum healed crack width in NCS (max. 850µm–970µm; white bar indicates 1mm)
when comparing different specimens. Therefore, average crack healing ratio (in different crack widths ranges) was used to assess the multiple crack healing efficiency in the later self-healing system.

3.3 Crack healing in the specimens with hydrogel encapsulated spores

3.3.1 Light microscopy

All the specimens showed no obvious crack healing when they were stored at 95%RH. However, in wet-dry cycles, all specimens showed crack healing. The specimens with hydrogel encapsulated bacteria added, provided an obvious superiority in the healing ratio (Fig.4) and the maximum healed crack width. A maximum crack width of about 0.2mm and 0.5mm could be healed in the specimens H and HS, respectively (images not shown). The R specimen had no completely healed cracks. Hydrogels can absorb water during the wet stage and retain the water for continuous autogenous healing (secondary hydration, etc.) and bacterial activities (bio-precipitation), resulting in an enhanced healing efficiency in the specimens with hydrogels, especially in the ones with bio-hydrogels.

![Figure 4. Average crack healing ratio for different crack widths of the specimens in wet-dry cycles.](image)

3.3.2 3D X-ray CT

The distribution of the healing products through the whole matrix can be seen in Fig.5 (image of R is not shown). It can be seen that the precipitation was mostly distributed on the surface layer. Compared with the specimen H, the specimen HS had more precipitation distributed all over the matrix. The volume ratio of total precipitation was about 1.3% for the specimen H and 2.2% for HS, respectively. The increased amount of healing products was due to bacterial precipitation.

![Figure 5. 3D view of the spatial distribution of healing products (in yellow) in the sample H (a) and HS (b).](image)

X-ray CT has also been used in earlier research, where bacteria were protected through encapsulation by brittle tubes [7]. In addition to the bacteria, the tubes contained a polyurethane based liquid healing agent. Upon crack formation and capsule breakage, both, the bacteria (and nutrients) and the polyurethane, were released into the crack. While the polyurethane reacted fast and filled the crack, bacterial activity started somewhat later. However, it resulted in pore filling of the polyurethane foam and thus additional crack sealing. In Figure 6 an X-ray CT render of a mortar sample with embedded
tubes is shown. It can be clearly seen that due to crack formation the tubes were emptied and the crack was filled with the polyurethane foam.

Figure 6. The specimen with embedded glass tubes containing polyurethane after cracking

4 CONCLUSIONS

In this study, by combined use of light microscopy and 3D X-ray µ-CT, crack healing was visualized and quantified, and an enhanced healing effect was proven in specimens in which immobilized bacteria had been incorporated. It can be seen that light microscopy is a useful tool for quantification of surficial crack healing and X-ray µ-CT is more powerful for characterization of the healing inside the specimen.

Among the three bacterial carriers used, microcapsules and hydrogels seem to provide superior crack healing efficiency in comparison with DE. However, a disadvantage is that they both caused some strength decline of the virgin specimens (data not shown) while DE had a rather positive effect on the strength. Also the incubation condition has a big effect on the self-healing behaviour. Without free water in the surroundings, no self-healing was observed in all the specimens. Wet-dry cycles can stimulate more self-healing than full immersion in the specimens with encapsulated bacteria. And by use of hydrogels, shorter wet periods would be sufficient to induce self-healing.

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REFERENCES


MICROBIALLY INFLUENCED DEGRADATION OF CEMENT-BASED MATERIALS IN BIOGAS PRODUCTION ENVIRONMENTS

C. Voegel (1,2)*, A. Bertron (1) and B. Erable (2)

(1) Université de Toulouse, UPS, INSA, LMDC (Laboratoire Matériaux et Durabilité des Constructions), 135, avenue de Rangueil, F-31 077 Toulouse Cedex 04, France
(2) Université de Toulouse, INPT, UPS, CNRS, Laboratoire de Génie Chimique, 4 Allée Emile Monso, 31432 Toulouse, France

* celestine.voegel@ensiacet.fr

Abstract
In biogas production plants (digesters), concrete structures suffer severe chemical and biological attacks at every step of the anaerobic digestion process. The attack on concrete may be linked to the synergetic effect of (i) organic acids, (ii) CO₂ co-produced by the metabolic activity of microorganisms and (iii) the presence of the bacteria themselves and their ability to form biofilms on the concrete surface. Combined with mechanical action, the consequences on concrete are loss of alkalinity, drop in mechanical strength, progressive erosion and the loss of air/water-tightness, which rapidly decrease the durability of the concrete structures. In a context of significant expansion of the biogas industry, the mechanisms of concrete deterioration need to be better understood with the goal of proposing new, efficient solutions (protection, formulation, etc.).

This study aims, firstly, to characterise the evolution of the biodegradable wastes in terms of biochemical composition during the process of digestion in order to identify the conditions that produce the most aggressive conditions for concrete. Secondly, it aims to evaluate the mechanisms of concrete deterioration in different situations (storage, treatment, etc.) and to distinguish the role of microorganisms from that of the microbial metabolites.

The biowaste samples were collected from a farm-sized biogas plant at different stages of the digestion process. The solid and liquid fractions were analysed chemically and microbiologically. Cement pastes were then immersed either in the raw effluents, or in synthetic organic wastes, the input composition of which was well known. The mechanisms of alteration were investigated using XRD, EPMA and SEM analysis.

Keywords: Anaerobic digestion, cement biodegradation, durability, fermentative microorganisms, biodeterioration, organic acids.
1 INTRODUCTION

Anaerobic digestion is a natural biological degradation process of organic matter in the absence of oxygen [1]. Complex organic matter undergoes several successive steps of transformation: hydrolysis, acidogenesis, acetogenesis and finally methanogenesis. This process can be implemented intentionally (industrialised) in a digester starting from organic wastes [2]. It leads mainly to the production of biogas, a good fuel source similar to natural gas containing methane, and co-products such as solid or liquid digestates. The demand for biogas as a new form of energy is increasing due to the rising cost of oil and natural gas. Biogas appears to be a good alternative as it is relatively cheap and is available locally. Consequently, the industrial development of anaerobic digesters has become widespread throughout the world for many sources of wastes. European policies support anaerobic digestion projects. For example, France has the objective of increasing the number of installations by about 300% by 2020[3].

Concrete is the main material for the construction of structures (pre-tank, digester, post-digester, storage tank…) in the biogas industry because it is economical, waterproof, offers high thermal inertia and complies with the standards of hygiene required in waste treatment. However, in the biogas industry, concrete is degraded by several aggressive chemical and biological components in the waste effluents in the course of the anaerobic digestion process. The degradation is notably due to acid components (various organic acids, including volatile fatty acids or VFA) [4] and CO$_2$ [5] produced by microbial oxidation processes and probably accentuated by the colonisation of the concrete surface by microbial biofilms. These attacks, combined with mechanical stresses due to machine traffic and tank cleaning (high pressure, scraping) during draining, result in loss of alkalinity, a decrease in mechanical resistance, progressive surface erosion and increasing concrete permeability. The consequences of such degradation of concrete structures are (i) economic, related to lower production yield due to biogas leakage and to the cost of repairing the structures and interrupting production during the repair work and (ii) environmental because the loss of sealing generates leakage of polluting effluents into the environment.

In order to make the construction of digesters sustainable, the degradation mechanisms caused by the microbiologically produced chemicals and by the microbes themselves have to be highlighted at all stages of the waste fermentation process. While the action of synthetic volatile fatty acids or CO$_2$ on the cement matrix are quite well known ([6]–[12]), very few studies have so far concerned the combined action of organic acids, CO$_2$ and microorganisms on the cement matrix. The mechanisms of microbial adhesion to the concrete surface (biofilm) have also received little attention as yet. In this context, our preliminary work first focused on (i) analysing the composition of a synthetic biowaste in terms of organic acid natures and concentrations during the anaerobic digestion process and (ii) determining the kinetics and mechanisms of microbiologically influenced deterioration of concrete. The degradation kinetics was assessed by mass loss and degraded layer depth measurements. The chemical, mineralogical and microstructural changes were explored by Electron Probe Microanalysis and X-Ray Diffraction.
2 MATERIALS AND METHODS

2.1 Cementitious materials

CEM III/C 32.5 cement pastes were made with a water/cement ratio of 0.4. CEM III/C 32.5 cement [13] contains 18% of ordinary Portland cement and 82% of blast furnace slag. It is noted CEM III/C in this study. Cement paste specimens were cast in cylindrical moulds 75 mm high and 25 mm in diameter. They were removed from their moulds 24 h after pouring and stored in water at 20°C for 28 days. They were then exposed to the synthetic biowaste solutions.

2.2 Synthetic biowaste solution

A model of biowaste, representative of the organic domestic waste production in France, was provided by IRSTEA of Antony (France). The composition of the model synthetic biowaste was (by mass): potatoes (8.1%), tomatoes (3.4%), minced meat (8.1%), milk powder (0.7%), crackers (4.1%), water (75.6%). The whole mixture was blended for 10 min at 20°C to obtain a smooth, perfectly homogeneous mixture. Half the biowaste was sterilized by autoclaving (121°C, 15 min) to destroy the bacterial flora naturally present.

2.3 Cement paste exposure to synthetic biowaste

The cement paste specimens were immersed in 500-mL closed bioreactors containing raw or sterilized biowaste for 13 weeks at 35°C ([14][15]). The solid/liquid ratio (cement paste surface area/biowaste volume) was approximately 224 cm².L⁻¹ for each test. The pH of the raw and sterilized biowastes were in the pH 5-6 range (around 5.3 for the raw biowaste and around 5.7 for the sterilized one) during the whole experiment.

2.4 Analysis of organic acids by HPLC

Two-millilitre samples of biowaste were collected regularly, using sterile needles and syringes, filtered to 0.2 µm in Eppendorf tubes and then stored in a freezer at -18°C for further analyses of organic acids. The nature and concentrations of organic acids in the liquid part of the biowaste samples were analysed by High Performance Liquid Chromatography (Thermo Fisher U3000, Aminex HPX-87H BIORAD column)

2.5 Monitoring of the cement paste degradation

The kinetics of degradation of the specimens was evaluated by measuring the depth of the degraded layer over time. Phenolphthalein was used to identify the degraded layer on the cross section of the specimens (change in colour of phenolphthalein for pH > 9.5). The cross sections were scanned and the images were exploited using Videomet® software. The data provided in this paper are the average of twelve measurements distributed around the circumference of the slices [6].

2.6 Cement surface analysis

The chemical changes in the specimens after the immersion tests were analysed with an electron microprobe ( Cameca SX 50, operating conditions: 15 kV, 20 nA). Details on the specimens and their preparation are given elsewhere [7]. The flat surface of the cylindrical specimens was analysed by XRD (Siemens D5000, Co cathode, 40kV, 30 nA) after immersion. Details are given in [8].
3 RESULTS AND DISCUSSION

3.1 Characterisation of organic acids in fermented biowastes

In the process of anaerobic digestion, the first oxidation step in the absence of oxygen is the microbial fermentation of complex polysaccharide organic matter such as cellulose, pectin, and chitin. These fermentations release a variety of organic acids and alcohols, together with CO$_2$ and H$_2$. In our experiments, no exogenous microbial source of microorganisms (sludge from digester or from wastewater treatment plant, sediments or other) was co-inoculated in the biowaste. Only the microflora naturally present in the various elements constituting the biowaste was used as a source of fermentative microorganisms.

3.1.1 Sterilised biowaste vs. raw biowaste

Half of the biowaste was initially sterilized with the objective of obtaining a mix of biowaste, having a chemical composition that was not affected by microbial activity. However, after only a few days, the initially sterilized biowaste had taken on a yellowish coloration and fungal growth could be observed on its surface. Possible survival of bacterial spores and/or fungi that resisted autoclaving, or a contamination of the biowaste by microorganisms from the ambient air of the laboratory may explain the start of fermentation in the autoclaved biowaste.

Accordingly, the changes in the nature and concentrations of organic acids in sterilized and raw biowaste were quite similar. Only a shift (or latency) of a few days was visible. For example, at $t = 6$ weeks, the total concentration of organic acids was 40.3 gL$^{-1}$ in the sterilized biowaste, i.e. 84% of the concentration measured in the raw biowaste (Figure 1). In both biowastes, the predominant organic acid was lactic acid, with a concentration always higher than 30 g.L$^{-1}$. The second most concentrated acid was butyric acid, and propionic, acetic and malic acids were also detected but at lower concentrations.

![Graph showing the comparison of organic acid concentrations in sterilized and raw biowaste.](image)

**Figure 1:** Influence of the presence of solid cement pastes (solid/liquid ratio of 224 cm$^3$.L$^{-1}$) on the nature and relative concentrations of organic acids during anaerobic fermentation of synthetic biowastes.
3.1.2 **Influence of the presence of solid cement pastes**

The presence of cement paste in the bioreactors (solid/liquid ratio of 224 cm\(^2\).L\(^{-1}\)) had a significant influence on the concentrations of organic acids measured in the liquid portion of fermented biowastes. After 6 weeks of fermentation, the total load of organic acids was systematically 30% lower in bioreactors containing cement pastes (33.6 vs. 47.8 g.L\(^{-1}\) in the raw biowaste and 28.3 vs. 40.3 g.L\(^{-1}\) in the initially sterilized biowaste) and the hierarchy of the relative concentrations of organic acids was very different from that obtained without the cement pastes. In the presence of cement pastes, the proportion of lactic acid was significantly lower and butyric acid was the major acid (up 17.0 g.L\(^{-1}\)). There are several hypotheses that might explain this phenomenon:

(i) Lactic acid produced by the metabolic activity of microorganisms may combine with ions released by the cementitious matrix (mainly Ca\(^{2+}\)) to form lactate salts. However, considering the pH (5.2), the concentration of lactic acid (about 30 g.L\(^{-1}\)) and the high solubility of Ca-lactate ([16][9]), this hypothesis is not the most likely.

(ii) Release of alkaline ions by the cement matrix in the bioreactor (leaching mechanism) leads to an increase in pH (from 3.9 to 5.3). This change in pH has an impact on microbial metabolism and may result in the activation of the metabolic pathway leading to butyric acid and/or an inhibition of the production of lactic acid. In the literature, the pH has been shown to affect microbial cell growth and fermentation rate, and also to change final product yield and purity. Changing the pH of the medium may induce a metabolic shift. For example, Zhu and Yang [17] reported a shift from predominant lactate and acetate production at pH 5.0 to predominant butyrate production at pH 6.0 when studying the fermentation of xylose by *C. tyrobutyricum*. This metabolic shift was found to be associated with changes in the activities of key NAD-dependent bacterial enzymes involved in lactic acid metabolic pathways. NAD-dependent bacterial enzymes are reduced during oxidation reactions catalyzed by enzymes. These reactions are pH-dependent because of H\(^+\) involvement, and NAD\(^+\) regeneration occurs in the complex respiratory chain of micro-organisms (where pH is high).

3.2 **Degradation of cementitious matrices exposed to the biowastes**

3.2.1 **Macroscopic observations**

After 6 weeks of immersion, the surface of the cement paste specimens changed from a grey colour to orange-brown. The same colour change has already been described for cement paste exposed to acetic acid and to mixes of VFA (acetic, propionic, butyric, iso-butyric and valeric acids) [10][9]. The cement paste surface also showed some macroscopic cracks but the outer altered layer still adhered to the specimen and was not dissolved during the immersion period. In addition, there was no visible precipitation characteristic of calcium salt. Thus, cracking was probably due to the shrinkage of the degraded zone that resulted from the loss of calcium during the attack.

After 12 weeks, the outer layer (500-µm thick) of the cement paste specimens immersed in raw biowaste had dissolved. After drying of the specimens at 20°C in the laboratory atmosphere, their external surfaces showed a cracked, whitish outer layer, characteristic of the degraded external layer of CEM III/C [11].

The cross sections of the cementitious specimens are presented in Table 1. After 6 weeks of exposure, the degraded layer of specimens in the raw biowaste was about twice as thick as the degraded layer observed on specimens exposed to the initially sterilised waste. After 12
weeks, macroscopic observations revealed a clearly visible zonation: zone 1 (core of the specimens) had the same colour as the control specimen; zone 2 was a light-grey zone immediately adjacent to zone 1; and zone 3 was the whitish outer layer. Zones 1 and 2 caused phenolphthalein to change colour, so their pH was higher than 9.5.

**Table 1:** Observation of the cross sections (slices) of solid cement pastes after 6 and 12 weeks of immersion in fermented biowastes (raw and initially sterilised)

<table>
<thead>
<tr>
<th>Immersion time</th>
<th>Initially sterilised biowaste</th>
<th>Raw biowaste</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>6 weeks</strong></td>
<td><img src="image1" alt="Microscopic observation" /></td>
<td><img src="image2" alt="Macroscopic observation" /></td>
</tr>
<tr>
<td>Microscopic observation</td>
<td>of the cross section of a cement specimen</td>
<td></td>
</tr>
<tr>
<td>of a cement specimen after phenolphthalein test</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>12 weeks</strong></td>
<td><img src="image3" alt="Macroscopic observation" /></td>
<td><img src="image4" alt="Macroscopic observation" /></td>
</tr>
<tr>
<td>Macroscopic observation</td>
<td>of the cross section of a cement specimen</td>
<td></td>
</tr>
<tr>
<td>of a cement specimen (untreated)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Macroscopic observation</td>
<td>of the cross section of a cement specimen after phenolphthalein test</td>
<td></td>
</tr>
</tbody>
</table>

### 3.2.2 Kinetics of concrete degradation

The degraded depth of the cement matrices was monitored over time (Figure 2).

**Figure 2:** Degradation kinetics of the surface layer of cement pastes immersed in fermented biowaste
In the fermented raw biowaste, the degradation kinetics was linear (0.17 mm.week\(^{-1}\)) over the 12 weeks of the study. The kinetics was slower (0.04 mm.week\(^{-1}\)) during the first 6 weeks of exposure in the initially sterilised biowaste. The fermentation started after a delay due to the inhibition of microbial activity by autoclaving and the total load of organic acids may consequently have been lower in the first few weeks. After 6 weeks of fermentation, production of organic acids was almost the same in both types of biowaste (figure 1), meaning that their microbial fermentation rates were almost the same.

Accordingly, degradation kinetics in the initially sterilised biowaste was faster between week 6 and week 12, increasing from 0.04 to 0.13 mm.week\(^{-1}\). Everything suggests that the degradation rate would eventually move towards that determined in the raw biowaste (0.17 mm.week\(^{-1}\)). With these degradation kinetics, the cover concrete of reinforced structures exposed to such environments (typically 5 cm-thick) would be entirely degraded in 6 years by the chemical action of the medium only. It should be noted that the alteration would probably be much more rapid when combined with mechanical action such as machine traffic or high pressure cleaning of the digester tanks.

### 3.3 Degradation mechanisms

#### 3.3.1 Chemical changes

Figure 3 presents the chemical analysis, according to the distance to the surface, of the cement pastes immersed in the initially sterilised and the raw biowastes. The chemical data are given as relative percentages of the oxide amounts [7][12]. The 3 zones observed on Table 1 are indicated on the figure. From the core of the specimen to the outer layer, these are [7][12]:

- **Zone 1**, core of the cement paste, having the same composition as a control specimen for the two conditions of biowaste exposure.
- **Zone 2**, slightly decalcified and presenting a significant increase in SO\(_3\) content. The relative amounts of SiO\(_2\), Al\(_2\)O\(_3\) and Fe\(_2\)O\(_3\) increase moderately with both raw and initially sterilised biowastes.
- **Zone 3**, showing a significant decrease of the CaO of the total amount of oxides. Zone 3 is completely decalcified in the raw biowaste. However, decalcification is not complete in the case of exposure to initially sterilised biowaste. The thickness of this zone is significantly less in the sterilized biowaste than in the raw one. The concentration of SO\(_3\) is zero in the raw biowaste. Increases in SiO\(_2\), Al\(_2\)O\(_3\) and Fe\(_2\)O\(_3\) amounts are also observed in both conditions.
**Figure 3:** Chemical composition profiles of solid cement paste immersed for 6 weeks in initially sterilised biowaste (A) and in raw biowaste (B)

The cement paste degradation observed in the case of raw biowaste was greater in terms of altered depth and intensity of alteration (notably decalcification) than that caused by the autoclaved biowaste. The presence of microorganisms leads to faster fermentation and seems to accelerate the degradation. This is in accordance with observations by Magniont et al. on synthetic bacteria-bearing media [18].

### 3.3.2 Mineralogical changes

The mineralogical characterisation of the different zones (1, 2 and 3) of the cement pastes, as defined in 3.3.1, was performed by XRD analysis after 6 weeks of exposure to the fermented biowastes. Figure 4 presents the X-ray patterns obtained in the core (zone 1) and in the altered zones (zones 2 and 3).

**Figure 4:** X-ray traces of cement paste specimens immersed for 6 weeks in raw and initially sterilised (autoclaved) biowastes

In each case, zone 1 showed the same composition as the control specimen. In Zone 2, the disappearance of the Ca(OH)$_2$ peaks and an increase in the ettringite peaks were observed. These observations can be related to the slight decalcification and the enrichment in sulphate observed in this zone with electron probe microanalysis analyses (EPMA). The absence of sulphate in zone 3 suggests diffusion of sulphate in zone 2. Thus, sulphate attack occurs and leads to ettringite formation in zone 2 [19]. Zone 3 contains calcite. The precipitation of calcite is linked to the diffusion of dissolved CO$_2$ from the aggressive solution through the degraded zone of the specimen [18][19][20]. In this zone, HCO$_3^-$ combined with Ca$^{2+}$, released by the cement paste, precipitates in the form of calcite. Calcite peaks are smaller in the outer layer of the specimen exposed to raw biowaste than in the specimen exposed to the autoclaved biowaste. As in Magniont’s study [18], calcite precipitation on the surface occurred mostly with immersion in autoclaved biowaste (without bacteria). Magniont et al.
explain that the presence of microorganisms on the surface probably leads to particular conditions of acid concentrations and pH locally. These conditions could account for the difference in calcite precipitation between the specimens immersed in raw and autoclaved biowastes. The degradation mechanisms of cement paste in autoclaved and raw biowastes combine progressive decalcification and a carbonation front.

4 CONCLUSION

- During incomplete anaerobic fermentation due to external factors, important acidification could occur and lead to a total production of approximately 40 g.L⁻¹ of organic acids.
- When cement paste was added to the biowaste, the production rate of organic acid decreased by 30%. The weak alkalinisation of the biowaste (from pH 3.7 to pH 5.3), due to the release of alkaline species by the cement paste, induced a metabolic shift in the microbially driven fermentation process. The production of butyric acid became prevalent, rather than lactic acid production. Under these conditions of organic acid production, the kinetics of cement paste degradation was estimated at 0.17 mm.week⁻¹ (nearly 9 mm.year⁻¹).

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DURABILITY PROBLEMS WITH ZINC CLADDED ROOFS DUE TO IMPROPER UNDERLAYS

Tommy Bunch-Nielsen (1) and Erik Brandt (2)

(1) Director, M.Sc., Bunch Building Physics, Vedbaek, Denmark
(2) Senior researcher, M.Sc., Danish Building Research Institute/Aalborg University, Copenhagen, Denmark

Abstract

In Denmark the use of zinc as roof cladding has been popular among architects over the last 10 years, and a number of corrosion problems have occurred after less than five years in some of these roofs. It is corrosion from the back side of the zinc cladding and in most cases it is so called hot water corrosion due condensation on the downside of zinc. The underlays for the zinc cladding have changed over the years and the cladding is often layed directly on hard mineral wool or plywood with a thin underlay of diffusion open textile roof underlay, which is also used for clay tiles.

The corrosion is related to the moist environment between the zinc and the underlay and the lack of ventilation of the underside of the cladding and lack of protection from oxidisation of the down side of the zinc. The condensation is related to moisture in the underlays and the sub cooling from radiation to the sky on clear nights.

Bitumen corrosion from organic acids has also been used as explanation for some corrosion problems in zinc roofs when zinc is installed directly on an underlay of bitumen and the use of structured underlays are discussed.

The paper presents results from a number of inspections of zinc roofs in Danish environment and the laboratory test of the zinc plates and leaching of chemicals from the underlays.

New design of zinc roofs to prevent corrosion is suggested in the conclusion of the paper.

1 INTRODUCTION

The use of metal roofs has increased over the last 10 to 15 years mainly due to architectural trends. The design of roofs has also changed from the traditional design with
wooden underlays to highly insulated roofs with underlays of insulation or prefabricated roof components. Zinc has been widely used due to its nice ability to protect its surface by forming zinc carbonate on the weathered side.

The change in design has been made without long term experience of the durability and this has led to a number of problems with the service life of the zinc roofs.

2 DESIGN OF ZINC ROOFS

Zinc with standing seems is the most normal used application for zinc roofs and the thickness of the zinc material is typically 0.8 mm or less.

The standing seems can be made on site and this makes it possible to design roof with a length of up to 40 meters. In the long roofs the temperature movements must be taken into account.

![Figure 1: Zinc roof with standing seems](image)

The traditional underlays has for the century been wooden boards of 25 mm thickness spaced with 5 – 10 mm distance between the boards.

For the last 25 years plywood or OSB have been used instead of boards and some experience has been gained with this underlay.

For the last 10 – 15 years zinc roofs designed as warm roofs with the zinc installed directly on mineral wool or perhaps with an underlay of diffusion open roof underlays based...
on nonwoven textiles or nonwoven laminated with micro perforated foils as shown in figure 2.

Figure 2: Zinc with diffusion open underlay of nonwoven laminated with micro perforated foil on mineral wool

One of the problems with this design is built in moisture. The mineral wool will often be left open for a period and in most European countries there is risk of rain all year around. If the mineral wool becomes wet in the construction period the moisture will be trapped in the roof structure.

The drying out is very difficult in a warm roof structure with a diffusion tight vapour barrier on the warm side and a metal roofing on the cold side, an typically drying will take several years.

There have been some indications from the suppliers and consultants that there is a drying capacity in the standing seams. That means that the moist air in the roof should be able to be pressed out of the roof when the roof is heated by the sun and the air in the roof expands. This has not been proved by the experience from roofs in practice.
Probably this would be compensated by in going air in the nights when the roof is cooled. In low sloped roofs with slopes down to 2 or 3 degrees it is important to close the seams with jointing material to make the roof water tight. To have watertight and diffusion open joints at the same time is not possible in practical installation.

The use of moisture adaptive vapour barriers have been tried and can function for small amounts of built in moisture. The sun will press the moisture down in the roof where it can dry out to the interior of the building as the moisture adaptive vapour barrier opens when it gets moist. Unfortunately the night and day cycles in temperature will to some extent send the moisture back to top during the night time - in highly insulated roofs the moisture may stay in the middle of the insulation.

3 CORROSION

Zinc and titanium zinc in contact with the atmosphere will protect its exterior surface by forming zinc carbonate. On the back of the zinc carbonate is not formed when the zinc is in close contact with plywood or a roof underlay. Therefore the zinc surface is unprotected and subject to corrosion if there is moisture present, e.g. due to build in water or leaks, and the zinc surface is heated a phenomenon called hot water corrosion will occur.

This hot water corrosion will cause pitting corrosion from the backside as shown in figure 3.

![Figure 3: Hole in zinc roof due to corrosion originating from the back seen from top side (left) and from the back (right).](image)

The pitting corrosion cannot be observed until a hole occurs and the roof leaks. The corrosion cannot be seen from the topside and can develop in few weeks.
The roofs can be visually inspected without the very small holes or signs of corrosion are observed. To investigate corrosion destructive inspections is needed. Repair is difficult and will be visible afterwards and furthermore the problem with corrosion is not solved and will consequently come back. To avoid further problems the only solution is a total refurbishment with a suitable substrate.

It is possible to inspect with a cover meter to determine the thickness of the zinc plate but this is not an accurate measurement and it is difficult to perform over a whole roof surface.

5  SEPARATION LAYERS

The use of structured separation layer was introduced approximately 10 years ago. Structured separation layers can be used on board materials like plywood and OSB where there is no air penetration from the underside.

When zinc is installed on an underlay of mineral wool in a warm roof there has been no clear rules for the use of structured underlays, as the mineral wool by some suppliers have been considered open to diffusion as well as to air infiltration.
Practical experiences from a number of roofs from 2006 to 2009 have shown that installation on mineral wool will often lead to corrosion of the back side of zinc roofs. The mineral wool used has a relatively high density of 120 to 180 kg/m$^3$ and the air penetration is therefore very small and availability of air is not sufficient to provide a surface protection of zinc carbonate to protect the zinc from corrosion. The corrosion starts as soon as moisture is present and the formation of zinc hydroxide starts.

The use of structured separation layers has three purposes:
- A: To ensure that the moisture cannot come in direct contact with the zinc surface – drainage effect
- B: To ensure that moisture from the underlying substrate, e.g. mineral wool is not transported to the zinc surface
- C: To ensure that air is available and can protect the surface by forming zinc carbonate.

![Figure 4: The backside of zinc plates taken from a roof without (right) and with (left) structured separation layer. It is clearly seen that no zinc carbonate is formed (right) and consequently no protection against corrosion achieved without separation layer. Instead zinc hydroxide is formed as with powder on the surface.](image)

There are two different types of structured separation layer. One type is a product with stiff plastic fibres that gives a distance between zinc and substrate of 7-8 mm. This type is open to diffusion and does not solve purpose B. The other type is a plastic board with “knots” that provides a distance of 7- 9 mm between zinc and substrate. This type is tight to diffusion and besides it is airtight. This type makes a barrier that prevents water from the insulation in reaching the zinc surface.
Figure 5: Typical structured separation layer of the type open to diffusion.

6 CONCLUSION

The backside of a zinc roof cladding must be accessible for air in order to form a protective layer of zinc carbonate to avoid corrosion from the back. If air is not accessible the protective layer is not formed.

The traditional way of constructing zinc roofs by use of board with a spacing of 5 – 10 mm over a ventilated gap, see figure 6, is one way of achieving accessibility of air. This type of construction has by long times experience proved to have a long service life.
Figure 5: The traditional solution with an underlay of wooden boards with a spacing of 5 - 10 mm over a ventilated gap.

When zinc is used as roofing in warm roofs or directly on a substrate of plywood and similar the access of air must be secured by a structured separation layer. There is so far no precise knowledge of the maximum length for a roof design with structured separation layer. For a typical structured separation layer of 8 mm the length of the roof must probably be limited to 20 m to provide enough air infiltration.

It is important to avoid accumulation of moisture in the structure from built in moisture (from the construction period) or water from leaks in flashings around windows or ventilation equipment on the roof. The design and workmanship of details, e.g. ventilation ducts and chimneys are therefore important.

REFERENCES

THE HIGH SCHOOL "GIUSTINO FORTUNATO" IN RIONERO IN VULTURE (POTENZA, ITALY): HOW TO REFURBISH AN HIGHER EDUCATION INSTITUTE MADE IN BRUTALIST ARCHITECTURE OF '80, LEADING IT TO BE A NZEB AND PRESERVING ITS FORMAL FEATURES.

Filiberto Lembo (1) and Francesco P.R. Marino (2)

(1) School of Engineering, University of Basilicata, Viale dell’Ateneo Lucano n. 10, 85100 Potenza (Italy) - filiberto.lembo@unibas.it

(2) School of Engineering, University of Basilicata, Viale dell’Ateneo Lucano n. 10, 85100 Potenza (Italy) - francesco.marino@unibas.it

Abstract

This study is about a building which was built after the 1980's earthquake in Campania and Basilicata, as USAID gift, in Paul Rudolph’s style, with external walls made in facing concrete, without insulation and lacking of flashings and of downpipe so much, to look, after twenty-five years, as a ruin; with very thin cover for reinforcement, and so with widespread pathologies of spalling of carbonated concrete; with roof wrongly designed; windows partly too little for a good dayligting and too exposed to dazzling; internal walls per 80% not performing acoustic performances fixed by standards, and all greater spaces (atrium and corridors, library, auditorium, sports hall) with reverberation time many times the standard value. The building is awful from thermal point of view: it disperses 76,7 kWh/m3 year, and places itself in the worse class of actual Italian thermic rules.

His management become ever more expensive: so this study has the aim of demonstrating how to find a remedy to design faults and to existing pathologies, upgrading the building to an NZEB, which produces from renewable sources the energy he need, without disfigure his formal configuration. The solutions developed can serve as “rules of the art” for refurbishment of brutalist architecture.

Keywords: brutalist architecture, refurbishment, thermal upgrade, NZEB.

1. DURABILITY AND ENERGY EFFICIENCY OF THE BRUTALIST ARCHITECTURE

In the '70s and '80s in Italy Brutalist movement has spread, which had its birth in England in
1954 by Alison and Peter Smithson (but has a forerunner in the Mies van der Rohe of 860-880 Lake Shore Drive Apartments, Chicago, 1949-1951), was theorized by Reyner Banham [1], had interpreters as Le Corbusier, Atelier 5, Louis Kahn (Salk Institute for Biological Studies, La Jolla, CA, 1959-1965) and Paul Rudolph (Art and Architecture Building, New Haven, 1963). Even in later years, the poetics of exposed concrete has permeated the masterpieces of contemporary architecture. For example, in the Vitra Design Museum in Weil am Rhein near Basel, there are two: the Feuerwehrhaus designed by Zaha Hadid, and the Konferenzpavillion designed by Tadao Ando, both dated 1993.

The brutalist architecture has laid bare the walls of buildings, and has made them with basic materials: concrete, steel, brick, wood, glass, explicitly exposed and devoid of both the normal layers of sacrifice, delivered from the historic building tradition (plaster, paintings, sheets and planks of protection), and devices developed by an age-old practice (frames and covers, window sills and thresholds, as well as thermal insulation, waterproofing barriers and multi-layer defenses). The result was buildings that have created many problems for designers, builders and users, when they were not "museum pieces", the subject of a formal and poetic use only; and when not designed to last and with attention to detail, by Louis Kahn or Zaha Hadid and Tadao Ando.

The concrete, if it is not sealed with paint and plaster and if unprotected, is carbonated in depth, changes color depending on the point where it receives more or less meteoric water, and then becomes different from that of the outer walls of the covers, the one between the intrados of windows, that of window sills, or that of the base section. Even more so, if the water is allowed to flow freely on the facades, without realizing the downspouts, but only bouncers and small chains. The concrete is covered with mold, in different ways depending on exposure to sun, wind and rain; it is slit and is no longer able to protect the steel, which corrodes and causes the expulsion of the concrete cover. The walls, when not isolated either from the inside, nor in the thickness of the wall, nor from the outside, are unbearably cold in winter and become the ideal place for surface and/or interstitial condensations; survival in spaces of this kind is entrusted fully to the plants, with energy costs that are now unbearable. So, if the building is not listed as "monument of modern architecture," design the retrofit is required, as soon as possible, because the cost of its continued operation becomes higher every winter.

2. AIM OF THE RESEARCH

The research aims to define a methodology to refurbish the brutalist architecture of this buildings in order to solve their technological contradictions, and radically improve their energy efficiency, transforming them into Nearly Zero Energy Building, in accordance with the European Directive 2010/31/EU, without altering their formal characteristics. Several buildings have already been examined: the Technical Institute "Albert Einstein" in Picerno (PZ), built by USAID pulse after the earthquake of 1980 [2], the former headquarters building of the Bank Mediterranean in Potenza (1976-1985) [3], the Scientific High School "Enrico Fermi" in Muro Lucano (PZ), also built by USAID after the same earthquake. This report presents the study carried out on the third of the three high schools, built by USAID in the Basilicata region after the 1980 earthquake.
3. CONTENT OF RESEARCH

3.1 Current status of the building

The Institute of Higher Education "Giustino Fortunato" was designed, like the other two schools mentioned, by the Interplan Design Studio of Naples, of Alberto Izzo and Camillo Gubitosi professors architects, in collaboration with the study Blurock Partnership in Newport Beach, California. The school is laid out, with greater axisly from West to East, on a plot of 22,250 m² with well 10% slope towards the South, at an altitude ranging between 660 and 648 m above sea level, with the disposal of buildings, set at different heights along the slope, part two-storey and partly to one, around two courtyards, with a useful floor area of 5,250 m² (see fig.1).
The entrance is from the North (see photo 1) and immediately introduces to a large double height atrium, 266 m$^2$, distribution element to the Gym, the library, classrooms and upstairs (see photo 2), probably thought of as the real school's Great Hall. Regular classrooms and special classrooms for labs are mostly facing South, but there are three, plus the Great Hall (very undersized compared to the requirements of the standard), which are addressed to the North. The Gym, structurally independent, occupies the west side of the building (see photo 3). The scheme, "plate" type, is not compact at all and is very energy-dispersing (see photos 4 and 5).

Photo 1: Panoramic view of the North elevation
At a site where the design must consider especially winter conditions, the building is practically devoid of insulation: its concrete walls, the roof and the first floor have U-values of transmittance ranging from 3.08 to 2.49-2.10-1.95 W/(m²K). Only some of the walls are provided with an insulation from the inside, made with 12 cm of mineral wool and gypsum board, with $U = 0.33 \text{ W/(m}^2\text{K)}$, which is obviously cause of interstitial condensation eight times higher than that in the Standard, so as to impregnate the mineral wool and degrade its insulating qualities. Thermal bridges are everywhere. The windows of the school are for the most part sliding on a track, with double glass and with a thickness of 3 cm, "not classified" as to air seal, tightness to the water and resistance to the wind, with $U_w$ ranging between 4.55 and 3.45 W/(m²K). Therefore, the annual heat demand of the building is 76,713 kWh/m³ year (energy class G, the worst of all) [4].

Moreover, the covers have proved poorly designed ("hot roof", with sheath outside, without vapor barrier), resulting in many points in moisture stains for condensations not evacuated outwards; in more points covers have become a quagmire, because of insufficient slope (see photo 6). The free run-off of rainwater has degraded surfaces and produced widespread or localized fungal infestations (see photos 7 and 8). The small concrete cover has resulted in more points spalling of concrete béton brut (see photos 9 and 10).
In addition to this, the main defects present inside the building, are: a) the insufficient sound insulation to airborne noise between classrooms and between classrooms and corridors; b) reverberation times, too high, in the spaces of higher dimension, such as the corridor between the classrooms, the atrium, the auditorium and especially the gym, where the T60 varies from three to almost eight times that of the standard; c) the inadequate natural lighting of the gym and classrooms that overlook the North side; d) glare, which affects all classrooms facing south (photo 11); the insufficient natural ventilation, despite the bad sealing of windows and doors.

3.2 Design solutions to problems

The solutions have involved first of all the need to provide a casing strongly isolated and ventilated, in order to allow the building heating (and cooling) as possible in a "passive" and "natural" way, and exploiting the great thermal inertia of its heavy concrete walls (of the order of 750 kg/m²). A system of isolation from the outside was then sought on the market, durable and which had the finish surface like an exposed concrete.

The research has not been extended to the rainscreens, given the stringent cost limits imposed. To facilitate the impermeability of the system, given the presence of non-vertical sculptural conches (as for the building " Ex Post" in Bolzano), has been used as insulating the EPS - sintered expanded polystyrene with expanded graphite, which has a λ=0.31 W/mK, in a thickness of 20 cm. On it, the application of an organic plaster, on reinforcement mesh glass fibers, has been expected, choosing between its different finishes that "Betonoptik", which is obtained by arranging on the armature mortar StoArmat the finishing plaster fine-grained modeling Stolit K 1.5 MP color in reinforced concrete (RAL 7023), and then further finishing plaster Stolit Milan, treated with a special technique of smoothing with steel trowel and then
sanded. In this way, it is possible to obtain a finish that, both in the distant views from that in those closely, is very similar in color, grain and texture, to béton brut. Naturally, the isolation from the outside is mounted only after having restored the damaged surfaces of concrete with the usual methods for the rehabilitation of reinforced concrete surfaces. Depending on the wall of the existing support, U values between 0.208 and 0.187 W/m²K are obtained.

As for the window areas, given the poor quality of the existing windows, it was decided to replace them with fixtures from the type "passive house" with triple glazing (two low emission) with argon, with external aluminum blinds for the glare protection, also useful for correcting the deficit of lighting in the rooms where they were found. A complete system of Controlled Mechanical Ventilation has been designed, with presence and CO₂ detectors placed in every room, so as to allow the air changes are continuous, proportional to persons and their activities, and are carried out with almost total recovery of energy. On the covers, healed and isolated to obtain a transmittance U=0.16 W/m²K, the placement of solar panels has been provided until exhaustion of the free sunny surfaces, for a total of 75.2 kW (see fig. 2). The replacement of the heat generators, with two heat pumps powered by photovoltaic, was also provided.

![Figure 2: Arrangement of photovoltaic panels (marked in blue) on the roof](image)

The problems of illumination of the Gym are solved with the construction of five sheds, open to the north, and covered with photovoltaic panels to south (see fig. 3), while his acoustic problems find their solution in the lining of the lower part with sound absorbing panels of mineral wool covered with perforated aluminum sheet. A new lay-out solution allows to use the gym independently from the school and at different times. Similarly, a series of actions with slight counter-walls solves the problems of isolation between classrooms and between classrooms and corridors.
The problems of the reverberation time of the atrium, the auditorium and classroom corridors are adequately resolved through the use of sound-absorbing panels and plaster of various kinds, matched according to their properties and characteristics of form and color (see figg. 4, 5 and 6).

The building maintains the volumetric features and *brutalist architecture* texture: it assumes, however, voluntarily, a color palate, which is the sign of the times, very different, in which the intervention of a NZEB upgrade is done.

### 4. CONCLUSIONS

- The substantial increase in thermal and energy performance of the building, through the use of a heavy thermal insulation from the outside of the "coat" type, the upgrading of technological systems, the realization of a photovoltaic system for electricity, and the
replacement of fixtures, makes building in Class A + (best), with an Epi equal to 5,941 kWh/m³ year [4].

- Regarding the replacement of the obsolete heating systems, ventilation and cooling with last generation plants would have a savings in terms of emissions, but above all, allow the return of the economic investment cost to improve the performance of the building in a few years.
- Based on a similar case, analyzed in detail, it can be stated that the pay-back time of the investment is approximately 15 years. The useful life of the building changes from 90 to 120 years.
- The value of the building increases more than the intervention costs. It is, therefore, economically viable and beneficial. And above all, perfectly compatible with the conservation of the formal characteristics of the building, which is an important document of the cultural climate of the 900’s in Italy.

5. CONTRIBUTIONS

The contribution of the authors in the research and in editing and writing the text of the paper, was equal.

6. REFERENCES

DURABILITY AND MICROSTRUCTURE OF MORTAR WITH CALCINED MARL AS SUPPLEMENTARY CEMENTING MATERIAL

Harald Justnes (1) and Tone A. Østnor (1)

(1) SINTEF Building and Infrastructure, Trondheim, Norway – harald.justnes@sintef.no; ToneAnita.A.Ostnor@sintef.no

Abstract

Mortars (sand:cement = 3:1 and w/c = 0.50) were made where Portland cement was replaced with 0, 20, 35, 50 and 65 vol% calcined marl. The compressive and flexural strengths were determined after 1, 3, 7, 28, 90, 365 and 730 days curing at 90% RH and 23°C. The strength up to 28 days age was higher or equal to the reference for up to 50 vol% marl replacement of cement, while only 20 and 35 vol% replacements gave higher strength than reference at 90 days. The strength of mortar with 65 vol% calcined marl continued to increase until 2 years and approached the strength of mortars with 35 and 50 vol% cement replacements.

The microstructure of the mortars was characterized by SEM after 90 days and 2 years curing, as well as capillary suction and saturation giving total volume of capillary pores and amount of macro pores. Electrical resistivity was also measured.

Mortars of all compositions were tested for durability starting after 90 days curing with respect to chloride diffusion, expansion caused by sulphate attack (5% Na₂SO₄) at both +5°C and +20°C, as well as carbonation (accelerated by 1% CO₂ and 60% RH).

1 INTRODUCTION

Marl, or calcareous clay, is considered "bad" clay for production of burnt clay products (e.g. bricks and light weight aggregate) since it is clay contaminated with substantial amounts of calcium carbonate that will form CaO after burning. This can lead to “pop outs” when calcium oxide reacts with water to calcium hydroxide during service.

Calcined marl has been proven earlier by Justnes et al [1] to be an effective pozzolan in cementitious products. Thus, marl can be a large SCM resource that is not yet exploited to make blended cements or as mortar/concrete additive. Marl with 10-20% CaCO₃, or rather calcareous mudstone, was calcined at 800°C leaving 20% of the original CaCO₃ intact.

Calcined marl can be considered “industrial pozzolan” within the European cement standard (EN 197-1), and it may be feasible to make a pozzolanic cement with up to 55%
clinker replacement (CEM IV/B) considering the 28 day strength and sufficient early strength documented in this paper.

2 MATERIALS AND EXPERIMENTS

2.1 Materials
The marl was provided by Saint Gobain Weber who calcined it in a rotary kiln close to industrial conditions. The calcined marl were ground to $d_{50} = 7 \, \mu m$. Normal Portland cement (CEM I 42.5R according to NS-EN 197-1) produced by Norcem Brevik, Norway, was used for all the mortar mixes. The aggregate used was 0 – 8 mm crushed granite rock from Årdal, Norway. The superplasticizer used was Dynamon SP 130 supplied by Mapei AS, Norway.

2.2 Mortar mix designs
The mortars were made with 0, 20, 35, 50 and 65 vol% replacement of cement with marl to secure a constant volume of binder. The consistency of fresh mortar was determined using a flow table. The water-to-binder ratio (w/b) was 0.5 in all the mortars while the flow was maintained within ± 5 % of the reference by varying the amount of superplasticizer; 0.0, 0.2, 0.3, 0.5 and 0.9% (of binder weight) for the mortars with 0, 20, 35, 50 and 65 vol%, respectively. The mortar mixes were cast in 40x40x160 mm moulds and 100x200 mm cylinders. After 24 hours the prisms and cylinders were removed from the moulds and stored in a cabinet at 90 % RH and 23 ± 2 ºC.

2.3 Methods for assessment and analysis

2.3.1 Compressive and flexural strength
The compressive and flexural strength were measured on the 40x40x160 mm prisms at 1, 3, 7, 28 and 90 days of curing according to NS-EN 196-1. There were 3 parallel prisms for the flexural strength and five parallels for the compressive strength.

2.3.2 Capillary suction rate and saturation
The capillary suction technique was performed on four parallel 20 mm slices sawn from cast cylinders from each mortar mixture that were dried at 105°C before start. The discs are placed on a grating 1 mm below the water surface in a covered box and the increase in weight as specimen suck water is monitored for 4 days and plotted versus square root of time. The method consist of 6 subsequent weighing steps from which one can calculate initial moisture content, total porosity ($\varepsilon_{\text{tot}}$), capillary porosity ($\varepsilon_{\text{cap}}$), entrained air volume ($\varepsilon_{\text{air}}$), average density of mortar solids ($\rho_s$) and dry density of mortar ($\rho_d$) as described by Justnes et al [2].

2.3.3 Resistivity measurements
The resistivity measurements were performed on the 20 mm discs from the capillary suction measurements after they were pressure saturated. Conducting gel was applied on the end surfaces ensuring electrical contact before placing two steel plates towards them and the resistivity was measured using a multimeter in the AC mode and a frequency of 1 MHz.
2.3.4 Resistance to carbonation
Determination of resistance to carbonation was performed according to NS-EN 13295 on all the mixes after 90 days of curing. The prisms were stored in a cabinet with an atmosphere of 1 % CO₂ and 60 ±10 % RH secured by flowing gas of that composition and by replacing the desiccating agent "blue gel" (silica gel with indicator) whenever needed. Periodically the prisms were split in two and the carbonation depth were measured on the surface sprayed with phenolphthalein becoming pink if pH > 10 and colourless if pH < 10.

2.3.5 Expansion by sulphate attack
Each set of 3 prisms with embedded studs from each mortar mix were submersed in 5 % sodium sulphate solution at 20 and 5 ºC and the prisms were weighted above and below water for calculation of the volume, followed by uniaxial length measurements each month. The prisms were submerged 3 days in water before measuring their length as a starting point before sulphate exposure. The 5 ºC test was carried out in case thaumasite was formed.

2.3.6 Chloride ingress
Determination of chloride penetration resistance was performed according to NT Build 443. The specimens were immersed in chloride solution (165 g NaCl per litre) for 35 days. At the end of the exposure period thin layers (2 mm) were ground inwards from the exposed surface. The chloride content for these layers was determined by spectrophotometric method.

2.3.7 Scanning Electron Microscopy (SEM)
One sample from all the mortar mixes cured for 90 days were cast in epoxy resin, plane polished to achieve a cross-section of the material and sputtered with carbon. The instrument used in this study was JEOL JXA – 8500F Electron Probe Micro analyser. The samples were analysed in the BSE (back scattered electron) mode where dense compounds and/or compounds composed of heavy elements appear bright (e.g. unreacted C₄AF mineral in cement) and compounds of low density and/or composed of elements with low atomic number appears dark (e.g. CSH). Details of interest were first checked for elements by EDS (energy dispersive spectra) semi-quantitatively, and further quantitatively by WDS (wave length dispersive spectra) for compositional determination.

3 RESULTS

3.1 Compressive and flexural strength
The average compressive and flexural strength for all mortars as a function of time are given in Table 1 together with standard deviations based on 5 and 3 parallels, respectively.

3.2 Capillary suction
The average data with standard deviations extracted from capillary suction profiles of 4 parallel discs for each mortar mix are listed in Table 2 after 90 days and 2 years curing.

3.3 Resistivity measurements
The electrical resistivity of the different mortars as a function of cement replacement by calcined marl and curing time is given in Table 3.
Table 1: Compressive (upper value) and flexural (lower value) strengths as a function of time for mortars where various amounts have been replaced by calcined marl.

<table>
<thead>
<tr>
<th>marl (vol%)</th>
<th>Average strength ± standard deviation [MPa] at ages [days]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1 d</td>
</tr>
<tr>
<td>0</td>
<td>22.0±0.3</td>
</tr>
<tr>
<td></td>
<td>4.8±0.1</td>
</tr>
<tr>
<td>20</td>
<td>17.9±0.4</td>
</tr>
<tr>
<td></td>
<td>3.8±0.3</td>
</tr>
<tr>
<td>35</td>
<td>14.0±0.1</td>
</tr>
<tr>
<td></td>
<td>3.1±0.1</td>
</tr>
<tr>
<td>50</td>
<td>9.5±0.2</td>
</tr>
<tr>
<td></td>
<td>2.2±0.1</td>
</tr>
<tr>
<td>65</td>
<td>5.7±0.1</td>
</tr>
<tr>
<td></td>
<td>1.3±0.1</td>
</tr>
</tbody>
</table>

Table 2: Mortar properties extracted from capillary suction experiments

<table>
<thead>
<tr>
<th>Marl (vol%)</th>
<th>Capillary porosity ε_cap [vol%] at 90 days</th>
<th>Capillary porosity ε_cap [vol%] at 2 years</th>
<th>Air content ε_air [vol%] at 90 days</th>
<th>Air content ε_air [vol%] at 2 years</th>
<th>Average density of solids, ρ_s [kg/m³] at 2 years</th>
<th>Dry density ρ_d [kg/m³] at 2 years</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>17.3 ± 0.4</td>
<td>16.8 ± 0.3</td>
<td>3.4 ± 0.2</td>
<td>3.2 ± 0.2</td>
<td>2660 ± 3</td>
<td>2127 ± 10</td>
</tr>
<tr>
<td>20</td>
<td>19.4 ± 0.2</td>
<td>19.0 ± 0.1</td>
<td>2.4 ± 0.2</td>
<td>2.5 ± 0.1</td>
<td>2692 ± 3</td>
<td>2114 ± 7</td>
</tr>
<tr>
<td>35</td>
<td>20.6 ± 0.2</td>
<td>20.4 ± 0.1</td>
<td>2.1 ± 0.1</td>
<td>2.2 ± 0.0</td>
<td>2706 ± 2</td>
<td>2093 ± 4</td>
</tr>
<tr>
<td>50</td>
<td>21.1 ± 0.1</td>
<td>20.9 ± 0.2</td>
<td>2.1 ± 0.2</td>
<td>2.2 ± 0.1</td>
<td>2683 ± 13</td>
<td>2063 ± 15</td>
</tr>
<tr>
<td>65</td>
<td>22.6 ± 0.1</td>
<td>21.8 ± 0.1</td>
<td>2.3 ± 0.1</td>
<td>2.1 ± 0.1</td>
<td>2680 ± 2</td>
<td>2040 ± 2</td>
</tr>
</tbody>
</table>

Table 3: Electrical resistivity for mortars where cement is replaced by calcined marl vs. time

<table>
<thead>
<tr>
<th>Marl (%)</th>
<th>Resistivity (ohm-m)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>8 weeks</td>
</tr>
<tr>
<td>0</td>
<td>93±6</td>
</tr>
<tr>
<td>20</td>
<td>182±20</td>
</tr>
<tr>
<td>35</td>
<td>208±12</td>
</tr>
<tr>
<td>50</td>
<td>492±34</td>
</tr>
<tr>
<td>65</td>
<td>229±9</td>
</tr>
</tbody>
</table>
3.4 Resistance to carbonation
The carbonation depths for the different mortar mixes as tested with phenolphthalein indicator after different exposure times to 1% CO$_2$ are listed in Table 4.

Table 4: Measured carbonation depth of all mortar mixes exposed in 1 % CO$_2$ for 33 weeks

<table>
<thead>
<tr>
<th>Marl (vol%)</th>
<th>Carbonation (mm) depth after 8 weeks</th>
<th>16 weeks</th>
<th>20 weeks</th>
<th>25 weeks</th>
<th>29 weeks</th>
<th>33 weeks</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
<td>2.0</td>
<td>3.5</td>
<td>3.2</td>
<td>4.8</td>
</tr>
<tr>
<td>20</td>
<td>0.5</td>
<td>2.5</td>
<td>2.5</td>
<td>4.5</td>
<td>6.3</td>
<td>6.5</td>
</tr>
<tr>
<td>35</td>
<td>1.5</td>
<td>3.5</td>
<td>5.5</td>
<td>6.5</td>
<td>9.9</td>
<td>14.8</td>
</tr>
<tr>
<td>50</td>
<td>2.5</td>
<td>5.0</td>
<td>9.0</td>
<td>11.0</td>
<td>15.3</td>
<td>&gt;20</td>
</tr>
<tr>
<td>65</td>
<td>4.0</td>
<td>7.5</td>
<td>14.0</td>
<td>18.0</td>
<td>&gt;20</td>
<td>&gt;20</td>
</tr>
</tbody>
</table>

3.5 Resistance to chloride ingress
The chloride ingress profiles for mortars with different cement replacements by calcined marl are plotted in Figure 1, while the parameters obtained from the curve fitting are listed in Table 5.

Figure 1: Chloride ingress in mortars where cement is replaced with 0 (M0), 20 (M20), 35 (M35), 50 (M50) and 65 (M65) vol% calcined marl after 35 days to 165 g NaCl/liter.

3.6 Resistance to sulphate attack
The linear expansions of the mortar prisms at 5 and 20°C after 20 months exposure are listed in Table 6.
Table 5: Parameters obtained from curve fitting of the chloride ingress profiles according to mathematical solution of Fick’s 2nd law of diffusion; the chloride surface concentration, \( C_0 \) (% of mortar mass), the apparent diffusion coefficient, \( D_{\text{app}} \) \((10^{-12} \text{ m}^2/\text{s})\) and the amount of chloride intrusion, ingress (g Cl⁻/m² surface).

<table>
<thead>
<tr>
<th>Marl (vol%)</th>
<th>Parameters from Cl⁻ profile</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( C_0 ) (%)</td>
</tr>
<tr>
<td>0</td>
<td>1.29±0.11</td>
</tr>
<tr>
<td>20</td>
<td>1.68±0.10</td>
</tr>
<tr>
<td>35</td>
<td>1.54±0.10</td>
</tr>
<tr>
<td>50</td>
<td>1.32±0.04</td>
</tr>
<tr>
<td>65</td>
<td>1.18±0.03</td>
</tr>
</tbody>
</table>

Table 6: Linear expansion (%) of mortar prisms (160 mm length) where cement is replaced with various amounts of calcined marl after 20 months exposure to 5% Na₂SO₄ at 5 and 20°C.

<table>
<thead>
<tr>
<th>Marl (vol%)</th>
<th>Linear expansion (%) with standard deviations (3 parallels)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>At 5°C</td>
</tr>
<tr>
<td>0</td>
<td>0.0148±0.0038</td>
</tr>
<tr>
<td>20</td>
<td>0.0163±0.0023</td>
</tr>
<tr>
<td>35</td>
<td>0.0100±0.0017</td>
</tr>
<tr>
<td>50</td>
<td>-0.0152±0.0343</td>
</tr>
<tr>
<td>65</td>
<td>0.0113±0.0010</td>
</tr>
</tbody>
</table>

3.7 Microstructure by SEM

Overviews (100x, not given here) of mortars showed a number of “dark spots” that turned up to be decalcified inner CSH of cement grains reacted through. Examples of such dark spots after 90 days and 2 years curing for mortar where 65 % cement is replaced with calcined marl are reproduced in Figure 2. The overall composition of the material in the “dark spot” after 2 years was determined by WDS to be in atom%; 5.6 Mg, 6.6 Al, 17.0 Si, 0.5 K, 13.1 Ca, 2.1 Fe and 54.4 O. The BSE images of mortar with 35 vol% cement replacement of calcined marl after 2 years curing in Figure 3 show to the left in the left images a cement grain that has reacted through and that is NOT decalcified with an inner CSH product with Ca/Si = 1.56. The products in the pores in the centre of images in Figure 3 are calcium aluminate hydrates (CAH), and the one to the right has composition 16.5 Ca, 8.3 Al, 1.1 Si, 0.4 S and 0.4 Fe in atom% by WDS. A Ca/Al = 2 corresponds to CAH compounds starting with \( \text{C}_4\text{A} \) in the shorthand notation of cement chemists (i.e. general \( \text{AF}_m \) phases), including calcium monocarboaluminate hydrate.
4 DISCUSSION

The compressive strength at 28 days is about equal to or higher than reference for cement substitution of calcined marl up to 50%. For the higher substitution, the compressive strength is substantially lower probably because all calcium hydroxide has been used up. The faster strength gain of the reference from 28 to 90 days compared to mortar with ≥ 50% replacement is an indication of this, but it is peculiar that the strength gain from 90 to 365 days, as well as from 1 to 2 years, is greater for mortar with the higher cement replacements with no calcium hydroxide present than for the reference again. There are four hypotheses that might explain the latter effect:
1) There are two types of CSH in the system. One with high C/S from hydration of cement and one with low C/S from the direct pozzolanic reaction between calcined marl and calcium hydroxide. The one with higher C/S has higher solubility of Ca\(^{2+}\) and is considered weaker mechanically than the one with lower C/S. Slowly the two different CSHs will equilibrate to an overall stronger CSH with intermediate C/S.

2) The solubility of Ca\(^{2+}\) from CSH with high C/S and pH is so high that one can have a direct further pozzolanic reaction with unreacted calcined marl.

3) The silicate anions of in particular CSH with low C/S will polymerize over time creating longer chain lengths of the CSH which may lead to even higher strength.

4) When calcined marl reacts, both silicate and aluminate is released forming CSH and CAH, as well as possibly intermediate products like \(\text{C}_2\text{ASH}_8\). Crystalline CAH generally demands a higher atomic Ca/Al than amorphous CSH demand Ca/Si, and the CSH is more flexible in its Ca/Si. The craving for Ca by the aluminate could lead to the de-calcification of inner CSH as observed as well as lowering the Ca/Si of the overall CSH that will adapt to that by increased polymerization of silicates (i.e. point 3 above). Inclusion of aluminate in the CSH structure (i.e. bridging of dimers) will also contribute to higher degree of polymerization of CSH. In total this may lead to a higher strength binder.

In addition, some calcium carbonate in the marl is maintained after the calcination, which may lead to formation of calcium carboaluminate hydrates in a secondary reaction between calcium carbonate and calcium aluminate hydrates from the pozzolanic reaction. This would be in analogy to the synergy between limestone and fly ash found by De Weerdt and Justnes [3] and De Weerdt et al. [4] and could be an explanation of the unusual high strength achieved by calcined marl as a pozzolan.

The porosity measurements by capillary suction and water saturation under pressure in Table 2 show that the capillary porosity increases steadily with increasing calcined marl replacement of cement and that the air content is about equal. The exception being the reference without calcined marl that had 1 vol\% more air and the lowest capillary porosity. This increase in capillary porosity with increasing cement replacement by calcined marl is expected as more crystalline calcium hydroxide is transferred into amorphous “gels” in the pozzolanic reaction. This increase in capillary porosity could also influenced by the “cellular” nature of unreacted calcined marl grains. The results in Table 2 also shows that the dry density of the mortars are decreasing with increasing volume replacement of cement by calcined marl, simply because the calcined marl has lower density (about 2.6 g/cm\(^3\)) than cement (about 3.15 g/cm\(^3\)). Increased porosity should lead to lower strength, not higher, unless the product formed is stronger in nature.

Comparing the porosity measured at 90 days with that at 2 years, one can see that the relative reduction in capillary porosity for the mortar with 65 vol\% cement replacement by calcined marl is 3.5\%, while the pore refinement of mortars with 35 and 50 vol\% replacement is only 1\%. Note that the absolute porosity values in Table 2 are of mortar volume and should be multiplied by 2.37 to become per binder volume (e.g. cement + water volume). The pore refinement in the period can explain the faster increase in strength for the mortar with 65 vol\% cement replacement, but the question remains what causes the pore refinement when there is no calcium hydroxide left for pozzolanic reaction.

Electrical resistivity of water saturated samples is expected to decrease with decreasing total porosity, pore sizes and decreased connectivity of pores, as well as with decreased
concentration of ions in the water phase (most notably hydroxyl ions responsible for the highest charge transfer). The electrical resistivity results listed in Table 3 show that the electrical resistivity of all samples is increasing with time as the porosity decreases or pores are becoming increasingly segmented. The mortar with 50 vol% cement replacement by calcined marl has the highest electrical resistivity, while mortar with 35 and 65 vol% cement replacement has about equal electrical resistivity considering the standard deviations. Thermogravimetry (not shown here) proved that mortar with 35 vol% cement replacement was the highest replacement containing a detectable amount of calcium hydroxide after 2 years curing, and thereby higher ionic strength in the pore water than the mortar with 65 vol% cement replacement. Hence, it is an indication that the latter sample (65 vol%) has a denser structure, or less connected pores, than the former (35 vol%), but a more open structure than the mortar with 50 vol% cement replaced by calcined marl after 2 years curing.

The carbonation rate steadily increases with increasing cement replacement by calcined marl, and there seems to be an extra increase in rate somewhere between 20 and 35% replacement of cement. When the carbonation depth is plotted versus square root of time, the curve is usually expected to be linear as a sign of diffusion controlled reaction. However, in this case it seems to be exponential (not shown). The decreased carbonation resistance seems to be the only drawback for cement replacements by SCMs in general. However, one should be careful of drawing conclusions from accelerated carbonation experiments with increased CO2 concentrations (1% CO2 in this case) over relative short time, as carbonation rates of concrete where cement is replaced by supplementary cementing materials (SCMs) under natural conditions (about 380 ppm or 0.038% CO2) happens over several years while the porosity is continuously refined over time.

Calcined marl replacing cement in mortars reduced chloride ingress until 50% replacement as can be seen directly from the chloride profiles. The ingress increases again for 65% replacements due to the same reasons as discussed under electrical resistance. Mortar where 50% calcined marl replaces cement has an apparent diffusion coefficient for chlorides of 40% of reference with 100% cement, while the values for mortars with 20 and 35% replacement is an apparent diffusion coefficient for chlorides of about 24% of reference. The reasons for the reduced chloride ingress can both be the pore refinement by calcined marl replacing cement as well as increased chloride binding by the higher content of CAH produced by the pozzolanic reaction that can lead to formation of Friedel’s salt in reaction with chlorides.

The linear expansion of the mortars after 20 months storage in 5% sodium sulphate at 5 and 20°C given in Table 6 showed that none of the mortars has any particular large expansion and there is little difference between them. The measurements will be continued.

Overviews (100x magnification) in the BSE mode of SEM of samples cured at 90 days and 2 years not shown here indicates a general brighter binder after 2 years compared to 90 days corresponding to a densification of the binder responsible for the strength increase. The difference seems to be greater for mortar with 50 and 65 vol% replacement of cement by marl than for the mortar with 35 vol% replacement. Another observation was that there seems to be less unhydrated cement (appears as bright, nearly white spots) as cement is increasingly replaced with calcined marl. This is natural since there will be less cement per area to begin with when the calcined marl content is increased. The degree of hydration of cement may also increase in general as it is dispersed by increasing content of calcined marl or as the pH is dropping when calcium hydroxide is depleted. Comparing images at 90 days with those at 2 years curing, there appears not to be any significant difference in the amount of “black spots”,
but some "black spots" in low magnification (100x) can actually be holes or so called Hadley grains (i.e. former cement grains dissolved completely). The element analysis of the “black spot” to the right in Figure 2 reveals that this is a decalcified inner product with a current Ca/Si = 0.77 enriched in magnesium (≈6%) and aluminum (≈7%). This composition is typical for a number of other “black spots” analyzed.

5 CONCLUSIONS

Calcined marl – ordinary "blue" clay contaminated with calcium carbonate – can be an effective pozzolan in cementitious materials if calcined at the correct temperature. Marl is unsuitable for the clay product industry, and might thus be the high-volume available alternative pozzolan globally that the cement and concrete industry is looking for in order to reduce their carbon footprint.

Excellent strength at both 1 and 28 days relative to reference is obtained when up to 50 vol% is replaced by calcined marl. The capillary porosity volume is increasing, but the electrical resistivity is also increasing, indicating a pore refinement or segmentation, as well as perhaps reduced ionic strength in the pore water. These are properties that might reduce the propagation rate of rebar corrosion once initiated by carbonation. The chloride ingress is drastically reduced relative to reference for mortars with ≤ 50% marl replacing cement.

The accelerated carbonation rate is increasing with increased cement replacement by calcined marl, but the effect may be less in practice with much lower CO₂ concentration and carbonation happening over years while the pore structure continuously densifies.

ACKNOWLEDGEMENTS

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REFERENCES

EMBODIED ENERGY AS A TOOL TO SUSTAINABLE STRATEGIC DECISIONS ON REFURBISHMENT

Pedro Lima Gaspar (1), António Lobato Santos (2)

(1) Faculty of Architecture, University of Lisbon – pmgaspar@fa.ulisboa.pt
(2) Faculty of Architecture, University of Lisbon – ajsantos@fa.ulisboa.pt

Abstract

Embodied energy on building materials is a concept that allows the measurement of environmental impact, considering energy expenditure associated to the extraction, transport, processing, in-site assembly and performance of materials, during their expected life cycle. From this point of view, longer service life periods of given materials correspond to more sustainable practices, as they reduce the impact of energy and resource consumption and the corresponding level of emissions.

In this paper, an assessment is made of the cost / benefit associated to two different strategies for intervention on a 40 year old detached single house project in Portugal: total demolition and reconstruction vs. refurbishment and alteration. Since the building cost of both strategies of intervention was estimated to be similar, environmental impact was considered as a decision criteria. Therefore, an analysis was made of the embodied energy of original materials, new materials and materials sent to landfill, for two different scenarios: (a) integral substitution of the existing structure by a new house, and (b) partial demolition and refurbishment of existing house. The original house was characterized to provide a benchmark for the comparison of both intervention strategies. In the end, data related to energy and mass were used to strategically sustain a decision regarding the recommended type of intervention.

1 INTRODUCTION

All buildings are an assembly of matter and energy that fulfil a purpose during a period of time. When buildings fail to meet the expectations of their users – be it from a technical, from a functional, aesthetical or economical point of view – they reach the end of their service life [SL]. Such threshold is seldom reached in a single (spectacular) moment, as would be the collapse of a structure. More often than not, building components within building layers go through a progressive state of deterioration or of obsolescence, with small continuous failures through a relatively large period of time. This process can generally be delayed through maintenance and repair. However, a time may be reached when users / owners’ expectations can only be fulfilled through alterations to the initial construction. Buildings are recognized to grow
over time and experience cycles of major refurbishment to accommodate new needs: more space, better infrastructures, more functional lay-outs or up-to-date fire and security performance, to name just a few.

In this paper, a case study has been used to monitor a major refurbishment process – the transformation of a detached single house located in Portugal, for which the authors also worked as designers. The original house was 40 years old and although the construction still responded to major technical requirements (load bearing, water tightness), all infrastructures were deemed to be obsolete, as well as the internal lay-out of spaces. The owner also required more usable space and wished for 40 to 60 year extension of the building SL, which led to an architectural design that enlarged the floor area, altered the interior lay-out and increased the thermal performance of the whole. Early consultations to local based construction firms showed two possible strategies: (a) total demolition of the existing house and construction of a new one; (b) a major alteration of the existing house. Although the cost of materials of the former solution was deemed to be higher, it was nevertheless a competitive possibility because of expected time and complexity savings. The refurbishment solution was regarded as less expensive in terms of materials, but required a better skilled work force to deal with the complexity of the building process and would require a larger building period. In the end, both solutions were deemed to have roughly the same cost.

Since the layout, brief and performance specifications were the same for the two building possibilities and since cost was not a differentiation factor, embodied energy [EE] of materials and the amount of construction and demolition waste [CDW] were used in order to identify the solution with less environmental impact. From a research point of view, this process was regarded as a miniature case study for sustainable strategic thinking and real life decision making that could provide hard data generally difficult to obtain, in line with similar research examples (Gonzalez and Navarro, 2006; Huijbregts et al., 2006).

2 BACKGROUND

Matter and energy are inextricably linked: building materials are the result of years of transformation of raw materials under the action of nature or of man in processes that requires variable amounts of energy (Santos, 2010). Building materials are then assembled into components and systems that need to be transported and installed in the building site. Since the production and consumption of energy have known environmental impacts (Hammond and Jones, 2008), the embodied energy on building materials - considering the different stages of the material’s lifecycle: extraction, transport, processing, in-site assembly and performance of materials (Dixit et al., 2010) can be used as a simplified indicator of environmental impact.

After the original construction, buildings continue to change (Brand, 1997). Within each major transformation cycle, buildings consume more matter and energy (necessary to produce, transport and assemble new materials and solutions and to dispose of existing matter). This process of inputs and outputs of matter and energy is generally referred to as “building metabolism”. Empirical evidence shows that buildings may loose and gain significant amounts of matter in major refurbishment operations, which may correspond to more than 80% of the matter used in the original construction (Santos, 2010) – figures that would be incompatible in terms of body mass metabolism in any living organism.

Data on the amount of matter required for a given construction operation may be broken down gradually in systems, components, materials and bulk materials. The total mass of this
“system” can then be calculated, as well as its embodied energy. From the latter, data on the level of emissions can be obtained. In theory, extending the service life of building materials can help to maximize the energy already invested in them and to minimize the carbon emissions to the atmosphere and building waste production (Nässen et al., 2007). However, such analyses are difficult to carry out not only because the scarce information on the embodied energy in building materials, but also because emissions are not directly proportional to energy (as they depend on the energetic mix on a given location and moment).

3 METHODOLOGY

In this research three “scenarios” were studied: (a) the original construction; (b) the demolition of the original construction and the building of a new house and (c) the partial demolition of the original construction and a major refurbishment operation. For simplicity’s sake, such scenarios are referred to as (a) “existing house” or scenario “E”; (b) “new house” or scenario “N”; (c) “refurbishment” or scenario “R”.

For each scenario, data was gathered on the building characteristics: dimensions were taken in the original house and the layouts for interventions N and R were obtained. Building materials and solutions of the existing house were identified through visual inspection and probing on walls and floors. For scenario N, detailed drawings and lists of building quantities were analysed. Scenario R required information both of the original building and of the projects. Data on infrastructure was not considered, although some sources refer these may amount to 10 to 15% of the embodied energy of a house (UNCHS, 1991; Hegger et al., 2008). This decision was due to the fact that both strategies of intervention considered new systems from scratch and the disposal of all existing infrastructures – and, as such, these data were neutral in terms of decision taking.

Building data thus gathered were broken down to bulk material level in order to obtain the overall mass of the building and material flow for each scenario. This task was far more difficult than it might seem, due to the scarce available and published information. Whenever possible, technical information on available commercial solutions was used. Fixings (such as nailing, bolting, screwing, etc.) were not accounted for given their low weight – although they’re usually composed of “industrial” or “synthetic” materials, with high embodied energy values per weight (Santos, 2010). Local and regional information on building materials, products and methods was preferred to other sources. As such, when possible, national sources were preferred to international data; and European data were preferred to data obtained from more distant sources.

Once lists were produced with the material flow analysis of each scenario, embodied energy was calculated. Again, this step presented many difficulties not only because of the little available data but also because of the – sometimes very expressive – differences between data obtained from different sources, a limitation often pointed out by other researchers. Thus, although the EE factors were taken from a number of published sources, the major source is the Inventory of Carbon and Energy from Hammond and Jones (2008). Using these data ensures transparency as they are in the public realm and can be reviewed by anyone at any time. The downside to using standardised data is that they do not account for individual production processes of specific companies and local characteristics, but rather use a “broad brush” approach based on averaged data. Finally, the obtained results for both intervention possibilities were compared, considering the overall values of mass and EE (that is, including
the values of the original house).

In comparing the three scenarios it is important to bear in mind the following aspects: (a) the building mass and EE from the original house correspond to the construction as-built, around forty years ago, with a small extension at an undefined point in the past and with very little changes ever since; (b) the design solution was identical for both scenarios in terms of plan and volume, differing only in terms of its form of implementation; (c) matter and energy of strategies N and R corresponds to the sum of the values of the original house and the new construction or refurbishment to fully take into account the overall environmental impact of both intervention strategies; and finally (d) all material associated with the demolitions of the original house is disposed of in form of CDW with no valuation operation due to the still prevailing lack of habits and incentives to reuse and recycling within the Portuguese context.

4 DESCRIPTION OF SCENARIOS E, N AND R

In this section, a succinct description of each scenario is made in order to allow a better understanding of the results and conclusions.

4.1 Scenario E – the existing house

The original building dated from the late sixties and had been in use for about 40 years, until it was deemed unpractical, obsolete and performing badly. The construction had a gross floor area of 164,4 m², divided into two floors, with only an external stair: in practice, it corresponded to two separate apartments. The structure was made of slender reinforced concrete (RC) elements (direct foundations, columns and beams) and slabs composed of prefabricated reinforced beams filled with hollow ceramic elements. Walls were made of double layer of extruded hollow ceramic bricks, laid with cement mortar, rendered and painted. Ground floor walls had load bearing foundations made of stone and cement. Windows were composed of wooden frames (main house) and extruded aluminium (extension) with single glass panes. Window sills were made of local marble stone. Solar protection was obtained through PVC roller blinds (main house only). Roofing was made of ceramic tiles, over a timber structure. No insulation or waterproofing was present. Inside, floors were finished with wooden parquet or ceramic tiling (in kitchens and bathrooms); interior walls and ceilings were plastered and painted.

In the original house, a total of 246,3 tons of matter was used, corresponding to 1,50 tons/m². After applying EE values to bulk materials used in the original construction, it was found that 587,0 GJ were present, which correspond to 3,57 GJ/m². These values are in line with the literature that points out reference values of 3,70 GJ/m² (UNCHS, 1991), especially if one considers that the original house had no insulation, nor any waterproofing.

4.2 Scenario N – new house

The new house was designed based on the land area occupied by the original house for licensing purposes, extending the enclosed area to 245,4 m². This extension represented an extra 49% to the original area and was obtained with more built volume and the occupation of the attic. The design was made so that both strategies N and R could be implemented; therefore the volume of the main house was respected, except for an addition on the back side, to the garden. The structure was designed in RC elements for the foundations, columns, beams and slabs; the roof structure was composed of an I-beam steel ridge and timber rafters covered with panels of cement wood, extruded polystyrene and plywood.
External cavity walls were composed of two layers of extruded hollow ceramic bricks, with a layer of render and an ETIC system composed of 4cm expanded polystyrene. Windows and doors received a 4cm frame of marble stone. Double glazed windows were made of double sash or sliding aluminium frames, protected with external aluminium shutters. A double lock standing seam zinc roofing system, along with zinc flashings and accessories, was selected in order to minimize future maintenance requirements. Internally, walls and ceilings were plastered and painted. On the ground floor, gypsum-board painted ceilings were also used. Pavements and internal stairs were finished with birch type wooden boards. Ceramic tiling was used in bathrooms, toilets and kitchen areas. The attic floor was finished with linoleum.

Rebuilding a new house would require 341.9 tons of material (more than 39% than the original house, for roughly 49% floor space) and 1.198.3 GJ of embodied energy (more than twice the EE required to build the original house). These figures correspond to 1.39 tons/m² (a 7% lighter construction than the original) and 4.88 GJ/m² (37% higher than the original figures). These values were added to those of the original construction so that decisions could be made in the context of sustainability strategic planning. Thus, the total figures for scenario N were of 588.2 tons of used material, of which 246.31 tons corresponded to CDW transported to landfills in the area; the total use of matter corresponded to 2.40 tons/m² (60% more than the original house). Embodied energy required for this scenario was of 1.785.34 GJ or 7.271 GJ/m² (two times the EE of the original house). These results show that nowadays constructions demand more EE on materials to meet current performance standards, but are more effective in the use of energy compared to past similar buildings.

4.3 Scenario R – refurbishing

As mentioned, the major refurbishing option corresponded to the same built area, volume, internal layout and finishing solutions than those considered in the scenario N. In scenario R, most of the interior of the house was to be demolished, as well as the roof, the external stairs, all windows and doors, the small extension to the original house and all infrastructures. A new structure would be added to the existing structure, which added to the complexity of the construction, especially considering the small scale building firms associated to this type of intervention. From the original house most of the structure, foundations and the external walls were to be reused - in other words, the “structure” and most of the “envelope” building layers.

This scenario corresponded to the demolition of 115.8 tons of the original house (roughly half of its mass, to be sent to landfill as CDW) or the disposal of 373.61 GJ (64% of the original embodied energy). In relative terms, the demolition meant a loss of 0.70 tons/m² and 2.27 GJ/m², referred to the original house. Refurbishment work required to estimated input of 249.4 tons of material and 1.017.48 GJ of embodied energy. In the end, this scenario corresponded to 1.475 tons/m² and 6.535 GJ/m² of embodied energy.

5 DISCUSSION OF RESULTS

This case study confirms the cyclic nature of the service life [SL] of buildings. After 40 years in use, the original house was considered below a minimum acceptance performance level, due to aesthetic reasons, but also due to obsolescence and evolving comfort patterns. Noteworthy is the fact that the physical (technical) performance of the structure, envelope and internal layout still responded to the original requirements. A major intervention was therefore deemed necessary in order to increase the SL of the house at least another 40 years. If
scenario N (new house) was chosen, then it could be said that the SL of the original house had reached its end. If scenario R were to be chosen, then the SL of the original house would be prolonged at the cost of a major operation in which the house would lose nearly half of its mass and two-thirds of the initial embodied energy. It would also gain new mass – equivalent to a new house – and more than double its original embodied energy. Such impressive building metabolism figures have no parallel in the world of living organisms (with which buildings and their life are often compared) and should be taken in account in any Life Cycle Analysis or, for that matter, in any study concerning costs, impacts and the life of buildings.

5.1 Total building mass and material flow comparisons between scenarios

The total used mass in the refurbishing scenario was of 249.4 tons, about 27% less than the total used mass for scenario N (588.2 tons). The amount of waste to be sent to landfill is of 115.8 tons (scenario R) and 246.3 tons (in scenario N). This last value is the totality of the original house and represents 42% of all used matter in scenario N. This indicator is of great importance as in many countries the cost of disposal of CDW is heavily taxed, adding to the cost of new constructions replacing existing buildings and corresponding to potential savings in refurbishment operations. This is particularly important as Portuguese building systems, processes and technologies mostly rely on traditional heavy weight construction, with cement as binder of elements, which has little potential for reuse, recycling or energy recovery.

Further analysis of data also shows that some building layers tend to always undergo renovation and replacement (i.e. extensive demolitions and new construction) regardless of the strategy/scenario: interior layout and finishes, infrastructures and services and part of the “skin” (roofing and facades). Some building layers are less prone to change, namely the structure of the building and the parts of the skin, such as bearing walls or structures, over which the owner or user has a lower level of “control” (Santos, 2010). Thus the major difference between strategies lies in the potential value of saving (parts of) the structure and external envelope.

5.2 Relative building mass comparisons

Analysis of the ratio of mass per floor unit area reveals changing trends in the construction industry in Portugal over the past 40 to 50 years. In fact, the original construction was relatively heavy (1.50 tons/m²) compared to a similar detached single house built today (1.39 tons/m²). That can be explained by the introduction of new materials and especially new insulation solutions that allow thinner walls, whilst increasing the thermal performance of the building. This trend can be understood as a progression from a relatively low-tech building tradition to more energy efficient constructions that comply with demanding building codes.

5.3 Total embodied energy comparisons between scenarios

In line with a more complex building practice (with materials requiring more energy in its extraction, production and synthesis – such as plastics, bitumen and metals), it is easy to identify a trend of growing embodied energy consumption in different periods of time. A building today requires less “heavy matter” than a building from the past but is far more energy demanding at the construction stage because it “consumes” industrial / synthetic materials. This has direct impact on the expected SL of new buildings.

Considering the surface, age and EE of the original house, this represents a value of 0.089 GJ/m²/year. Such results show that, given the larger surface area and EE value of scenario N,
the new house should last an estimated 55 years in order to achieve similar values GJ/m²/year. Nevertheless, it is expected that the new buildings perform better, namely in terms of energy consumption at the “in use” stage, with corresponding economic gains.

5.4 Relative embodied energy comparisons between scenarios

The embodied energy required to build a m² of a detached single family house today is 36.7% higher than the energy to build a similar house in the late 60’s (4.88 GJ/m² and 3.57 GJ/m²). Present day constructions tend to be lighter, better insulated and, as noted before, make use of high energy building materials (PVC, plastics in general, silicon based products, etc.). If the EE associated to demolition were not to be accounted for, building a new house would be the most favourable option (8% less energy consuming than the sum of EE of the original house and the refurbishment works). However, considering the overall use of EE – that is, taking into account also the energy used on the demolished parts – clearly refurbishing is more energy saving (6.53 GJ/m²) than demolishing the existing house and building a new one (7.27 GJ/m²). In the end, extending the SL of the existing house it is more environmentally correct, because it preserves more matter and saves more EE in absolute and relative terms than building a new house would, regardless of the Carbon print of both strategies (which are not addressed in this paper) and not including the cost of disposal of building waste (which would be higher in a totally new construction versus a refurbishment option). These results were taken into consideration and a decision was made for scenario R, which entailed a more complex building process but lower matter consumption and greater EE savings (Figure 1).

Figure 1a: Existing original house at an early deconstruction stage; 1b detail of partial demolition and introduction of a new structure; 1c midway building process, before render and ETICs introduction on the outside: note existing walls with new windows, new roof structure and renovated overall image.

6 CONCLUSIONS

In this paper, a case study is presented in which information on the Embodied Energy and material flow is used to help a strategic building decision, considering a sustainable point of view. The case studied presented some characteristics that made it an interesting research example: the existing house (of about 40 years of age) had reached its service life limit due to functional and aesthetical reasons and two possible courses of action could be followed: (a) demolishing the existing house and building a new one or (b) a major renovation that could
extend the SL of the house for 40 years more. Both strategies had the same layout design and similar building costs, especially after weighting the complexity of the building process and the time required for it. Thus, an analysis was made of the environmental indicators of material flow and embodied energy of each strategy, along with the data of the original house. The study had some important limitations as it did not consider infrastructures and services and had to use data of several different sources (especially concerning EE) none of which from a local or regional building context: south of Europe and the Mediterranean.

The results showed insights of the metabolism of each building strategy and confirmed that such mid-life transformations of buildings tend to replace large amounts of matter (referred to the original construction), with the removal of nearly half the mass of the existing house and the introduction of the equivalent to a new house, despite the strategy considered. Results also showed that new constructions tend to weight less but consume more embodied energy than previous constructions, as newer and more synthetic materials are used for insulation and waterproofing, when compared to an older and relatively low-tech way of building.

Finally, it was concluded that refurbishment was a more sustainable strategy than new construction as it represented less matter and embodied energy consumption and less demolition waste. These data were therefore used as criteria for strategic decision making and the house was renovated with only partial demolitions and a major internal refurbishment.

It is expected that this case study and methodology may lay the ground for more wide-scope studies, in terms of building types and number of cases studied.

REFERENCES

The study of the characteristics of mortars is normally performed on samples prepared in laboratory and in metallic moulds with several configurations according to the standards requirements. However, the behaviour of mortar in the laboratory samples will not be exactly the same of mortar applied on real substrates.

After the application of the mortar on the substrate an interface will be formed and the interactions that occur after the contact of the mortar in the plastic state with the substrate will change over time due to hydration kinetics and absorption of substrate [1,2]. The behaviour of the mortar, over time, will then be influenced by the characteristics of the substrate and by the interface between these construction materials.

Which parameters will influence this behaviour? Will the influence of the mortars own characteristics be greater than the influence of the substrate properties?

These are some of the questions that we don’t know the answer yet and that we intend to answer with the research that is taking place at the University of Coimbra in collaboration with the University of Porto and LNEC. The first results obtained will be presented in this paper.

1. Introduction

The wall coverings most commonly used are still plaster and render mortars. Mortar plasters and renders are applied on brick surfaces, concrete blocks surfaces, even reinforced concrete surfaces or other ones. For the selection of a mortar the technical characteristics that are provided are generally analyzed without taking into account the influence of the kind of support intended for use. After application, the final characteristics may be different from the expected.
On the other hand mortars play also a fundamental role in the conservation of built heritage. Concerning mortars for renders it can be said that they are the "skin" of our buildings. They are the construction elements more exposed to the direct action of water, climatic agents, mechanical action of shock and erosion, chemical action of pollution and soluble salts. Being the most exposed elements they will also be the first ones to degrade and to require repair or replacement. Concerning old buildings, namely built heritage, it must be taken into account not only the functional aspects of the coatings but also the preservation of history and of our collective memory [3].

Considering these presuppositions and face to the degradation of a wall covering the first option should always be the conservation, maintenance and repair of existing mortars. Replacement should only be considered when repair is not viable.

When the need of substitution is verified, two possible ways of action are open:
- Reproduce the old mortar;
- Formulate a compatible mortar

An integrated methodology of these two ways is the best option [3].

That is, the composition of the existing mortar must be taken into account, and as far as possible, the constituents and the type of mortar must be respected, but also the most significant performance characteristics must be identified and reproduced, as far as possible.

After defining the desired characteristics for the replacing render or plaster and knowing the composition of the existing mortar the replacing mortar must be chosen by comparing the characteristics wanted with the characteristics of the available mortars. If a mortar with the intended characteristics is not available, a new composition must be studied that fits the required characteristics.

It is at this stage that an important question may arise: will the behaviour observed in the laboratory and on specimens hardened in metal moulds be the same for mortars applied on their substrates? The evolution of the characteristics of mortars over time will influence their overall behaviour?

After the application of the mortar on the substrate an interface will be formed and the interactions that occur after the contact of the mortar in the plastic state with the substrate will change over time due to hydration kinetics and absorption of substrate [1,2]. The behaviour of the mortar, over time, will then be influenced by the characteristics of the substrate and by the interface between these construction materials.

These are some of the questions that this research developed at the University of Coimbra in collaboration with LNEC and FEUP wants to answer, by testing both cement based and lime based mortars applied on different kinds of substrates.

2. EXPERIMENTAL CAMPAIGN

2.1 Objectives

The main objective of all the campaign that has been developed is to analyse the influence of the characteristics of the substrate, the application conditions and the curing conditions/accelerated aging on the behaviour of the mortars applied on them.

For the development of the campaign three types of substrates (acrylic, concrete and brick) and three kinds of mortars (cement mortar, one coat mortar and air lime mortar) were chosen.

In this paper the results obtained after accelerated aging will be presented and a comparative analysis with the obtained results after 28/60 days will be performed.
2.2 Performed tests

The substrates were characterized with the determination of the following properties: open porosity, bulk density, water absorption and water vapour permeability. The characterization of the mortars was carried out in two different ways: first several samples with different dimensions according to standards and for all the mortars were made and hardened under the standard conditions. Then the application of a layer of each mortar with a constant thickness of 1,5 cm was carried out onto the different substrates and they were then submitted to the standard curing conditions. For samples in which it was intended to later separate mortar a fiberglass mesh was introduced between the mortar and the substrate to facilitate this detachment.

After the 28/60 days of the normal curing half of the samples were submitted to accelerated aging conditions based on the standard EN 1015-21 [4]. The evaluated properties were: open porosity, bulk density, water absorption and water vapour permeability. In table 1 the used standards are presented.

<table>
<thead>
<tr>
<th>Obtained characteristics</th>
<th>Used Standards</th>
</tr>
</thead>
<tbody>
<tr>
<td>Open porosity / bulk density</td>
<td>NP EN 1936:2008 [7]</td>
</tr>
</tbody>
</table>

For the determination of drying characteristics the tests were based on the procedure Nº II.5 from RILEM [8] and the drying index was calculated according to the following formulas:

$$i_5 = \frac{\sum_{i=1}^{n} \left( t_i - t_{i-1} \right) \times \frac{Q_{i-1} + Q_i}{2} }{Q_{\text{max}} \times t_n}$$  

$$i = \frac{m_i - m_{\text{dry}}}{m_{\text{dry}}}$$  

with:

- $t_i$ – moment of test i, [h]
- $t_o$ – moment at the end of test, [h]
- $Q_i$ – amount of water inside the sample at moment i, [%]
- $Q_{\text{max}}$ – amount of water inside the sample at initial moment i, [%]
- $m_i$ – mass of the sample at the moment I [g]
- $m_{\text{dry}}$ – mass of the dry sample [g]

2.3 Obtained results

A) Substrates

As mentioned before the chosen substrates were acrylic, because it is a low absorbent material, and concrete and brick because they are current substrates in buildings. All the substrates were made with facial dimensions of 40x40cm.

The obtained results for the substrates’ properties are presented in table 1.
Table 2: Properties of the substrates

<table>
<thead>
<tr>
<th>Property/Substrate</th>
<th>Acrylic</th>
<th>Concrete</th>
<th>Brick</th>
</tr>
</thead>
<tbody>
<tr>
<td>Open porosity [%]</td>
<td>0,26</td>
<td>12,63</td>
<td>19,10</td>
</tr>
<tr>
<td>Bulk density [Kg/m³]</td>
<td>1177</td>
<td>2247</td>
<td>2026</td>
</tr>
<tr>
<td>Water absorption coefficient [kg/(m²).s^(1/2)]</td>
<td>0,0028</td>
<td>0,0045</td>
<td>0,0260</td>
</tr>
<tr>
<td>Water vapour permeability coefficient [kg/m.s.Pa]</td>
<td>2,0 x 10^{-13}</td>
<td>1,9 x 10^{-12}</td>
<td>2,3 x 10^{-12}</td>
</tr>
</tbody>
</table>

B) Mortars

The experimental campaign began with the moulding of prismatic samples with 40x40x160mm for the determination of open porosity, bulk density, water absorption and drying index, and circular samples with 1,5cm of thickness and 10cm of diameter for the determination of water vapour permeability. All the samples were kept in the first seven days at 20ºC and 95% relative humidity and after that the relative humidity was changed to 65% and the temperature remained at 20ºC. After 28/60 days (28 days for cement mortars and one-coat mortar and 60 days for air lime mortar), the properties were determined and are presented in table 3.

Table 3: Properties of the mortars hardened in the moulds

<table>
<thead>
<tr>
<th>Property</th>
<th>CM</th>
<th>OCM</th>
<th>ALM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Open porosity [%]</td>
<td>17,45</td>
<td>22,71</td>
<td>29,66</td>
</tr>
<tr>
<td>Bulk density [Kg/m³]</td>
<td>1884</td>
<td>1586</td>
<td>1546</td>
</tr>
<tr>
<td>Water absorption coefficient [kg/(m²).s^(1/2)]</td>
<td>0,0356</td>
<td>0,0065</td>
<td>0,0625</td>
</tr>
<tr>
<td>Water vapour permeability coefficient [kg/m.s.Pa]</td>
<td>0,56 x 10^{-11}</td>
<td>1,36 x 10^{-11}</td>
<td>1,72 x 10^{-11}</td>
</tr>
</tbody>
</table>

CM - Cement mortar; OCM - One-coat-mortar; ALM - Air-lime mortar

The following step was the application of the mortars onto the different substrates which was made by the application of a layer with 1,5cm of thickness. In the first 28/60 days of curing the conditions were the same aforementioned.

After this curing half of the samples were submitted to an accelerated aging during 8 days based on the standard EN 1015-21 [4] and subjected to two series of conditioning of four cycles each one.

1. 1st series: 60±2°C during 8h±15min; 20±2°C/65±5% RH during 30±2min; -15±1°C during 15h±15min; 20±2°C/65±5% RH during 30±2min.
2. 2nd series: partial emersion in water (5mm) at 60±2°C during 8h±15min; 20±2°C/65±5% RH during 30±2min; -15±1°C during 15h±15min; 20±2°C/65±5% RH during 30±2min.

To the determination of the properties, the mortars were detached from substrates, samples of mortar with different dimensions were made and were tested according to applicable standards.
The obtained results to the mortars removed from the substrates and tested after 28/60 days of curing are presented in table 4 and the results obtained for the mortars subjected to the accelerate aging are presented in table 5.

Table 4: Properties of the mortars tested after normal curing (28/60 days)

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Mortar</th>
<th>$P_O$ [%]</th>
<th>$\rho_b$ [Kg/m$^3$]</th>
<th>$W_A$ [kg/(m$^2$).s$^{1/2}$]</th>
<th>DI</th>
<th>$\delta$ [kg/m.s.Pa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acrylic</td>
<td>CM</td>
<td>16,30</td>
<td>1947</td>
<td>0,0377</td>
<td>0,432</td>
<td>0,595 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>OCM</td>
<td>16,49</td>
<td>1543</td>
<td>0,0188</td>
<td>0,150</td>
<td>1,418 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>ALM</td>
<td>16,80</td>
<td>1605</td>
<td>0,0170</td>
<td>0,201</td>
<td>1,326 x$^{-11}$</td>
</tr>
<tr>
<td>Concrete</td>
<td>CM</td>
<td>15,41</td>
<td>1952</td>
<td>0,0347</td>
<td>0,390</td>
<td>0,676 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>OCM</td>
<td>22,16</td>
<td>1624</td>
<td>0,0251</td>
<td>0,130</td>
<td>1,580 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>ALM</td>
<td>13,07</td>
<td>1575</td>
<td>0,0135</td>
<td>0,203</td>
<td>1,406 x$^{-11}$</td>
</tr>
<tr>
<td>Brick</td>
<td>CM</td>
<td>13,56</td>
<td>1980</td>
<td>0,0137</td>
<td>0,557</td>
<td>0,601 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>OCM</td>
<td>16,10</td>
<td>1678</td>
<td>0,0154</td>
<td>0,190</td>
<td>1,214 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>ALM</td>
<td>13,16</td>
<td>1626</td>
<td>0,0114</td>
<td>0,221</td>
<td>1,556 x$^{-11}$</td>
</tr>
</tbody>
</table>

CM - Cement mortar; OCM - One-coat-mortar; ALM - Air-lime mortar; $P_O$ - Open porosity; $\rho_b$ - Bulk density; $W_A$ - Water absorption coefficient; $\delta$ - Water vapour permeability coefficient; DI - Drying index

Table 5: Properties of the mortars tested after accelerated aging

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Mortar</th>
<th>$P_O$ [%]</th>
<th>$\rho_b$ [Kg/m$^3$]</th>
<th>$W_A$ [kg/(m$^2$).s$^{1/2}$]</th>
<th>DI</th>
<th>$\delta$ [kg/m.s.Pa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acrylic</td>
<td>CMaa</td>
<td>15,26</td>
<td>1997</td>
<td>0,0342</td>
<td>0,324</td>
<td>0,567 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>OCMaa</td>
<td>8,94</td>
<td>1631</td>
<td>0,0132</td>
<td>0,210</td>
<td>1,206 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>ALMaa</td>
<td>19,15</td>
<td>1595</td>
<td>0,0264</td>
<td>0,206</td>
<td>1,025 x$^{-11}$</td>
</tr>
<tr>
<td>Concrete</td>
<td>CMaa</td>
<td>14,79</td>
<td>2012</td>
<td>0,0258</td>
<td>0,380</td>
<td>0,690 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>OCMaa</td>
<td>24,69</td>
<td>1546</td>
<td>0,0276</td>
<td>0,135</td>
<td>1,382 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>ALMaa</td>
<td>21,87</td>
<td>1570</td>
<td>0,0202</td>
<td>0,221</td>
<td>1,165 x$^{-11}$</td>
</tr>
<tr>
<td>Brick</td>
<td>CMaa</td>
<td>11,88</td>
<td>1989</td>
<td>0,0099</td>
<td>0,456</td>
<td>0,551 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>OCMaa</td>
<td>11,80</td>
<td>1720</td>
<td>0,0079</td>
<td>0,269</td>
<td>0,976 x$^{-11}$</td>
</tr>
<tr>
<td></td>
<td>ALMaa</td>
<td>18,67</td>
<td>1612</td>
<td>0,0203</td>
<td>0,198</td>
<td>1,048 x$^{-11}$</td>
</tr>
</tbody>
</table>

CMaa - Cement mortar; OCMaa - One-coat-mortar; ALMaa - Air-lime mortar; $P_O$ - Open porosity; $\rho_b$ - Bulk density; $W_A$ - Water absorption coefficient; $\delta$ - Water vapour permeability coefficient; DI - Drying index
3. DISCUSSION OF THE RESULTS

For a better analysis of the comparative behaviour of all the mortars some graphics were made and presented in figures 1, 2 and 3 where all the results mentioned above are displayed.

**Figure 1: Bulk density and open porosity of all the samples**

BDm - Bulk density in moulds; BDa - Bulk density in acrylic substrate; BDc - Bulk density in concrete substrate; BDb - Bulk density in brick substrate; OPm - Open Porosity in moulds; OPa - Open Porosity in acrylic substrate; OPc - Open Porosity in concrete substrate; OPb - Open Porosity in brick substrate

**Figure 2: Water absorption coefficient [kg/(m\(^2\)).s\(^{1/2}\)] of all the samples**

In what concerns to bulk density and open porosity it can be seen that both the substrates and the accelerated aging influence them. It is noticed that when the porosity of the substrate increases the porosity of the mortars decreases and their bulk density increases. Regarding the
influence of the accelerated aging it can be said that for cement mortar it has introduced a reduction of porosity and a growth of bulk density in all mortars, contrary to what happened with air lime mortars were all the mortars had a growth of porosity and a reduction of bulk density. To one-coat mortar a clear trend cannot be identified.

Figure 3: Water vapour permeability coefficient $[x10^{-11} \text{ (kg/m.s.Pa)}]$ and drying index of all the samples

For water absorption again the influence of the substrates and accelerated aging is observed. In this case it can be noted that generically in cement mortars and air lime mortars water absorption decreases for samples applied in substrates and increases for one-coat-mortar. With respect to the influence of accelerated aging in cement mortars and one-coat-mortars it causes the decrease of water absorption and in air lime mortars causes its increase. With respect to the behaviour towards water vapour it can be confirmed that cement mortar has the lowest water vapour permeability and lowest drying capacity and on the opposite side is air lime mortar. The substrates tend to increase water vapour permeability and drying capacity for cement mortar and one-coat-mortar and decrease in the case of air lime mortar. The influence of the accelerated aging goes towards decreasing the water vapour permeability and the drying capacity.

4. CONCLUSIONS

The presented results are part of the investigation that is being developed. Some conclusions can already be drawn.

Without any doubt the characteristics of the substrates and accelerated aging influence, in different ways, the characteristics of all mortars. The characteristics that are less influenced
are open porosity and bulk density. Porosity of applied mortars decreases when compared with samples prepared in metallic moulds, decreasing as the substrate becomes more porous, and bulk density varies in the opposite direction, as expected. The accelerated aging causes the reduction of open porosity to cement mortar and one-coat-mortar and an increase to air lime mortar.

Concerning the water behaviour it is noticed that water absorption coefficient generally decreases as the porosity of the substrate increases, although this trend is not so clear for all types of mortars; the accelerated aging causes its decrease for cement mortar and one-coat-mortar, probably due to progress of cement hydration, and increase for air lime mortar.

Finally it can be said that for cement mortar the values obtained for water vapour permeability for mortars on the substrates were always higher than those for mortar samples hardened in metal moulds, contrary to what happened to air lime mortar. As for one-coat-mortar there is no defined trend.

5. ACKNOWLEDGEMENTS

The authors would like to thank FCT- Portuguese Foundation for Science and Technology for its substrate through the project “EXPL/ECM-COM/0928/2012 - Incorporation of ceramic residues in repair mortars”.

The authors would like to thank the FCT- Portuguese Foundation for Science and Technology for its substrate through the project “PTDC/EC M-C OM/3080/2012-Development and optimization of a higro-adjustable system for drying out buildings after a flood”.

6. REFERENCES

Abstract

PVC membranes have been in use since approximately 50 years. The paper describes three case studies of plasticized PVC sheets in roofing and tunnel waterproofing with exposure durations of more than forty years.

Case 1 is a roofing membrane produced in 1967. It was applied in fully adhered mode on a hydropower station in the southern part of Switzerland. This membrane is still functional after 45 years in direct weathering exposure. Samples were collected in 2001 and 2012. Although some degradation was found, the mechanical properties of this membrane remain on a level that it will fulfill its function for several more years.

Case studies 2 and 3 report on PVC sheets collected from two road tunnels in Switzerland after 41 and 44 years of use. Testing was done in cooperation with the Technical University of Cologne. The membranes had marginally aged. The mechanical data are still above the requirements for new membranes. Contemporary tunnel construction is based on the assumption of one hundred years of service life. The measured data suggest that this assumed period will be reached by the membranes with high probability.

1 INTRODUCTION

The patent for the industrial production of PVC was granted in 1913. However, membranes for roofing and waterproofing applications based on plasticized PVC are in use since the early 1960's only. Publications on the durability of such membranes are scarce, especially reports from the early years of application.

A comprehensive field study on 25 roofs in North America and 20 roofs in Europe was conducted in 2001/2002 and reported with various aspects (Graveline et at. [1], Whelan et al. [2], Cash [3] and Beer et al. [3,4]). Some papers on the hail resistance of aged PVC membranes were published by Cullen [6], Fricklas [7], Foley et al., [8] and Beer et al. [9].
One of the roofs of the 2001 study was revisited in 2012. The results of this meanwhile 45 year old roof are dealt with in case study 1.

For tunnel waterproofing no publications on applications twenty years or older could be found. This is understandable since tunnels have a design service life of one hundred years and access to the waterproofing membrane is almost impossible once a tunnel is finished. Due to security updates of two road tunnels of age 41 and 44 years samples could be collected. The results are presented in cases studies 2 and 3.

2 CASE STUDY 1 – A 45 YEAR OLD PVC ROOFING MEMBRANE

The roof of the hydropower station Nuova Biaschina at Personico/Switzerland was covered with a membrane of plasticized PVC with a fiber glass mat reinforcement. It was produced in 1967 and applied in fully adhered mode with a contact adhesive (Figure 1). A first sampling took place in 2001. The results of the subsequent lab testing were reported earlier [4].

In a second sampling in 2012 pieces of 1m by 1m were cut out and the sampling area was recovered with a freshly produced piece of membrane. Joining the 45 year old membrane with the new one by means of hot air welding was possible without any difficulty (Figure 2).

The sample was tested at the original manufacturer’s R&D center. The results of this testing are depicted in Figures 3-8 with initial values and data after 34 and 45 years respectively.

Thickness is reduced gradually from 1.2 mm to 1.04 mm (Figure 3, EN 1849-2). This goes in parallel with plasticizer reduction to 63.4 % compared to the initial content (Figure 4). This loss of plasticizer leads to some stiffening of the membrane which reflects in the shift of the low temperature folding from -20 °C to 5 °C (Figure 5, EN 495-5). The 45 year value of this cold flexibility property did not change compared to the 34 year value. Dynamic puncture resistance was reduced from the initial 450 mm falling height of the impact tool to 300 mm after 34 years and stayed then constant after 45 years (Figure 6, EN 12691). The 300 mm represent the standard requirement for new membranes.
In tensile testing the elongation at break was reduced from 220% to 100% (Figure 8, EN 12311-2/B). Although this reduction is significant the membrane can still be elongated by double of its length until it breaks. This drop can be explained by the reduced flexibility. On the other hand the flexibility reduction leads to an increased tensile strength (Figure 7).

Figure 3: Thickness as f(time).
Figure 4: Plasticizer content as f(time).
Figure 5: Low temperature folding as f(time).
Figure 6: Impact resistance as f(time).
Figure 7: Tensile strength as f(time).
Figure 8: Elongation at break as f(time).

3 CASE STUDY 2 – A 41 YEAR OLD PVC TUNNEL MEMBRANE

The Tunnel Reussport is part of the highway bypass of the city of Lucerne in the center of Switzerland. It is integrated into the E35, a European main traffic axis North-South from Benelux and Germany to Italy. This bypass is one of the most frequented highway sections in Switzerland. Every day about 85’000 vehicles pass the tunnel. It is a 600 meters long mining
The tunnel and consists of twin tubes with three lanes each. The tunnel has a rock superstructure of up to 30 meters in an urban area. It was built in 1970 in the partial waterproofing style, i.e. the umbrella type with water drainage along the tunnel side but without bottom waterproofing (Figure 9).

Due to some heavy accidents all the alpine tunnels are being refitted to comply with increased security requirements. One of them is the creation of person escape ports (Figure 10). At the occasion of such excavations the tunnel waterproofing membrane was accessible and samples could be collected.

![Figure 9: Schematic build-up of the Reussport Tunnel.](image9)

![Figure 10: Excavation of escape port in the Reussport Tunnel.](image10)

The refurbishment started in 2009 and was finished in June 2013. The waterproofing membrane is a single ply membrane made of PVC-P and contains a glass fleece inlay. Its thickness was originally 1.5 mm. It is semi-transparent with a beige color. The samples received were visually in good condition although traces from stone indentations could be found on the bottom side of the membrane. The membrane had been manufactured in 1970. A retain sample of that particular production was still available for comparative testing with the aged sample from the tunnel. Apart from some holes and scratches arising from the excavation procedure the samples were in a very good state with surprisingly high flexibility.

The samples were tested at the University of Applied Sciences, Cologne/Germany and at the manufacturer’s R&D center. Results and their discussion are presented in sections 6 and 7.

4 CASE STUDY 3 – A 44 YEAR OLD PVC TUNNEL MEMBRANE

The tunnel Allmend, Thun, is part of the highway A8 connecting the city of Berne with the City of Lucerne (Switzerland). It was built in 1968 in open cut construction mode. It underpasses a military training ground. The tunnel Allmend consists of twin tubes of two lanes each and is 960 m long (Figure 11). Although subject to a first refurbishment in 1991 the tunnel is partially damaged due to its service life of 45 years and requires renovation as well as new security installations. During the planning phase of the refurbishment project samples of the waterproofing membrane were taken by an engineering company to evaluate...
their actual condition. The engineering consultants handed over membrane samples to the manufacturer for in-depth analyses after 44 years of successful and damage-free service life.

The membrane was manufactured in 1968. It is of black color (Figure 12). As with the Reussport Tunnel a retain sample from that time is available (Figure 7). Six samples of size A3 were received and were tested by the same laboratories as in case study 2 above. The samples are reinforced single ply membranes made of PVC-P, the reinforcement consisting of a fine polyester mesh. They do not show any damage and are in very good and flexible condition.

![Figure 10: Portal of the Allmend tunnel](image)

![Figure 11: Sample from the Allmend tunnel.](image)

5 TEST PROCEDURE CASE STUDIES 2 AND 3

The samples from the Reussport tunnel contained traces of adhering concrete on the backside. For testing purposes areas with low concrete residues were used and the residual concrete was removed. Samples from both tunnels were tested according to actual European standard procedures.

The ability of welding to new membranes was determined by hot air welding samples of PVC membranes from the two tunnels to fresh membrane pieces as actually provided to the market (Figure 12). After cooling down of the seams their strengths were tested by peeling. The criterion of a good seam is that break occurs not within but outside the seam.

![Figure 12: Two types of new membranes heat welded to a sample from the Reussport tunnel.](image)

The results of the sample testing from both tunnels are listed in Tables 1 and 2. All tests were carried out using EN standard test methods with the exception of the plasticizer content determination which is an internal test procedure of the manufacturer.
## 6 TEST RESULTS CASE STUDIES 2 AND 3

### Table 1: Properties of Samples from Tunnel Reussport

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Method</th>
<th>Requirement¹)</th>
<th>Retain Sample 1970</th>
<th>Sample From Reussport Tunnel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>DIN EN 1849-2</td>
<td>-</td>
<td>1.50 mm</td>
<td>1.25 mm</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>DIN EN ISO 527-3</td>
<td>12 MPa ¹)</td>
<td>17.0 MPa</td>
<td>15.8 MPa</td>
</tr>
<tr>
<td>Elongation at Break</td>
<td>DIN EN ISO 527-3</td>
<td>&gt; 250 % ¹)</td>
<td>300 %</td>
<td>278 %</td>
</tr>
<tr>
<td>Impact Resistance</td>
<td>DIN EN 12691 Method A</td>
<td>750 mm ¹)</td>
<td>1'000 mm</td>
<td>750 mm (2/5 1'000 mm)</td>
</tr>
<tr>
<td>Folding at Low Temperature</td>
<td>DIN EN 495-5</td>
<td>- 20 °C ¹)</td>
<td>- 20 °C pass</td>
<td>- 20 °C pass</td>
</tr>
<tr>
<td>Water Tightness</td>
<td>DIN EN 1928</td>
<td>no leakage at 10 kPa/24h ²)</td>
<td>pass</td>
<td>pass</td>
</tr>
<tr>
<td>Plasticiser Content</td>
<td>Manufacturer's Test</td>
<td>-</td>
<td>100 %</td>
<td>91.1 % ³)</td>
</tr>
<tr>
<td>Seam Test with New Membrane</td>
<td>DIN EN 12316-2</td>
<td>break outside of seam ¹)</td>
<td>pass</td>
<td>pass</td>
</tr>
</tbody>
</table>

¹) Requirements by ZTV/ING, ²) Requirements by SIA V280, ³) value vs retain sample

### Table 2: Properties of Samples from Tunnel Allmend

<table>
<thead>
<tr>
<th>Property</th>
<th>Test Method</th>
<th>Requirement¹)</th>
<th>Retain Sample 1968</th>
<th>Sample From Allmend Tunnel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>DIN EN 1849</td>
<td>-</td>
<td>1.45 mm</td>
<td>1.17 mm</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>DIN EN ISO 527-3</td>
<td>12 MPa ¹)</td>
<td>17.5 MPa</td>
<td>17.5 MPa</td>
</tr>
<tr>
<td>Elongation at Break</td>
<td>DIN EN ISO 527-3</td>
<td>&gt; 250 % ¹)</td>
<td>240 %</td>
<td>226 %</td>
</tr>
<tr>
<td>Impact Resistance</td>
<td>DIN EN 12691 Method A</td>
<td>750 mm ¹)</td>
<td>1'000 mm</td>
<td>750 mm (4/5 1'000 mm)</td>
</tr>
<tr>
<td>Folding at Low Temperature</td>
<td>DIN EN 495-5</td>
<td>- 20 °C ¹)</td>
<td>-30 °C</td>
<td>-20...-25 °C</td>
</tr>
<tr>
<td>Water Tightness</td>
<td>DIN EN 1928</td>
<td>no leakage at 10 kPa/24h ²)</td>
<td>pass</td>
<td>pass</td>
</tr>
<tr>
<td>Plasticiser Content</td>
<td>Manufacturer's Test</td>
<td>-</td>
<td>100 %</td>
<td>87.7 % ³)</td>
</tr>
<tr>
<td>Seam Test with New Membrane</td>
<td>DIN EN 12316-2</td>
<td>break outside of seam ¹)</td>
<td>pass</td>
<td>pass</td>
</tr>
</tbody>
</table>

¹) Requirements by ZTV/ING, ²) Requirements by SIA V280, ³) value vs retain sample
7 DISCUSSION

1.1 Roofing Membrane

The 45 year old roofing membrane was found to be in a satisfactory state – still fulfilling its waterproofing function without any leakage after this extended period of outdoor exposure. A significant feature is the fact that the sampling area could be closed without difficulty by re-welding a new piece of membrane onto the aged membrane. This is important because it ensures that possible mechanical damages to the aged membrane could be patched.

Over the whole exposure period the membrane has lost plasticizer and thus has become less flexible compared to its initial state. This reflects in lowered values of thickness, elongation at break and low temperature flexibility. On the other hand the tensile strength has increased.

Despite the aged state the membrane still has flexibility, mechanical properties and the ability to over-weld that give no indication for replacement in the near future. The course of the aging process as depicted in Figures 3-8 suggests that this membrane will fulfill its function for a service life for several more years.

1.2 Tunnel Membranes

After 41 and 44 years the two tunnel membranes showed very minor signs of aging. Still between 87 and 91 percent of the original plasticizer load is retained. The mechanical properties are remarkably high and exceed with one exception the state of the art requirements for new membranes. The exception is the elongation at break of the Allmend sample. However, in this case the retain sample from 1968 had an elongation of 240% which is slightly below today's specification of 250%. The aged sample had an elongation value decrease of 6 percent only.

The mechanical data after more than forty years in the tunnel can be regarded as good as new. Taking this and the easy re-welding ability into account it can be expected that these membranes might have a service life of several more decades.

A more detailed report on these tunnel membrane examinations is given in [10].

8 CONCLUSIONS

This study gives evidence that properly formulated and applied PVC membranes can have service lives clearly beyond forty years.

Roofing and tunnel membranes made of plasticized PVC with on-site service lives of more than forty years have been investigated. All membranes are fully functional and can be joined to new membranes by hot air welding.

The examined roofing membrane shows signs of aging after 45 years of direct weathering exposure. The course of aging and the still good state of the membrane lead to the conclusion that it will at least reach a service life of fifty years.

The outstanding condition of the two PVC tunnel membranes after 41 and 44 years of real exposure in tunnels leads to the conclusion that they have hardly aged. Consequently the still excellent functionality of these products has brought the engineers of the two tunnel upgrade projects to the conclusion that a replacement during the tunnel renovation was not necessary.

Unlike other material classes that show rapid decay after long phases of almost no deterioration PVC membranes possess the fastest decrease of properties in the first years of
application. This initial decrease is followed by an exponential slow down. Based on this practical experience it does not seem presumptuous to assume on the basis of an exponential degradation slow down that these tunnel membranes can meet the actual design expectation of 100 years of service life for tunnels.

ACKNOWLEDGEMENTS

The authors would like to thank the road construction authorities of the Swiss Federation and of the Canton of Lucerne for allowing the taking of samples from the Reussport tunnel and Basler & Hofmann Engineering, Zurich for submitting samples from the Allmend tunnel. Thanks also go to AET, Bellinzona for permitting access and sample cutting from the Nuova Biaschina roof.

REFERENCES

PULL-OUT TESTS OF CAST-IN-PLACE ANCHOR STUDS WITH DIFFERENT EMBEDMENT DEPTHS

F. Delhomme (1), T. Roure (2), B. Arrieta (2), R. Trunfio (1) and A. Limam (1)

(1) University of Lyon, INSA-Lyon, LGCIE, F-69621, Villeurbanne, France; fabien.delhomme@insa-lyon.fr
(2) EDF SEPTEN Compagny, Civil Engineering, Villeurbanne, France.

Abstract
The equipment of French nuclear power plants is fixed on reinforced concrete structures with base plate with headed fasteners. EDF decided to carry out an experimental research program in partnership with LGCIE in order to optimize the service life design of the headed fasteners and identify margin. This article introduces the results of static tension tests on an anchorage composed of an anchor plate welded to four headed rods. The anchors are cast in place in a reinforced concrete block. The experimental campaign is carried out on real size anchors and anchors with reduced embedment depths. Tests on single headed rods are also carried out in order to analyse the group effects. The slide of the anchor plate, the strain of the head rods, the strain of the stirrups and the surface concrete block displacements are measured. The collapse of the anchorages is caused by a failure of the steel rods or a concrete breakout failure for a loading ranging between 60000 and 30000 daN. This experimental campaign will provide a data base enabling the development of numerical models in order to improve the design.

1 INTRODUCTION
In French nuclear power plants, various pieces of equipment are often fastened on reinforced concrete structures with cast-in-place anchorage, thus avoiding any drilling of the walls or slabs which would complicate the sealing of the construction. Their main function is to transfer normal loads (tension and compression) and possible shear loads relative to efforts at the base of the structure. The three main anchoring systems are the straight rod, the hooked rod and the headed rod. The tensile load is transferred through bonding between the steel rod and the concrete and/or abutment of the anchor plate or the hook on concrete. Load capacities of mechanisms depend on the type and dimensions of anchor used. Under tension, the three main failure mechanisms are the breaking of the rod, the sliding of the rod and concrete breakout cone [1, 2].
In EPR nuclear power plants, headed rods are usually used. The quantity of anchorages to be installed is increasing and their installation can be difficult for these concrete structures which have a high rate of reinforcement. EDF decided to carry out an experimental research program in partnership with LGCIE on their cast-in-place anchorage. The main goals are the following:

- Optimizing the service life design and identifying safety margins with design codes and standards [3, 4, 5, 6] recommended for rods with low embedment depths and thus, not really suited to the nuclear context.
- Reducing their volume by using straight rods consisting of ribbed bar.
- Checking load capacity and durability.
- Obtaining a database of the behaviour of anchorages in different configurations in order to validate numerical models.

All in all, about 60 tests over 3 years are expected, varying the following parameters: the type of anchor rod (ribbed bar or headed smooth bar), the type of loading (tensile, shearing, static and cyclic), the state of the concrete block (cracking or not), the embedment depth and edge distance. In the article, the results on the variation of the embedment depth for the anchors with headed rods are presented and analysed.

2 BACKGROUND

A lot of research has already been carried out in order to estimate the ultimate strength of anchor bolts in concrete. The most commonly observed failure mode, if the steel strength is sufficient and at shallow embedment depths, is concrete cone failure. The two main categories of model for describing this failure mode are concrete cone [7, 8] and concrete capacity design (CDD) [5, 6, 9, 10] models. Concrete cone models assume a projected circular area on the concrete surface based on a failure cone with a side inclination of 45° to the concrete surface. CDD models assume a projected square area on the concrete surface side dimensions of three times the anchor embedment depth and a failure surface within the concrete of approximately 35° to the concrete surface.

The experimental studies are usually realized on a single anchor rod and rarely on an anchor plate equipped with several rods. For cast-in-place anchorages, most tests have been carried out on studs of short length, and finally, few results are available for anchorages with large dimensions. Among these, Lee et al. [11] did tests on headed anchors with large deep embedment in concrete, ranging between 638 and 1143 mm. The objective was to obtain a failure by tensile breakout of concrete for different embedment depths. The authors showed that the breakout cone with the concrete surface varied from 20 to 30 degrees. Rodriguez et al. [12, 13] observed that the effect of the reinforcement placed parallel to the free surface of the block (perpendicular to the anchor axis) is negligible. To affect the loading capacity, the reinforcement must lie with the breakout cone, must be oriented parallel to the applied load, and must be developed in the surrounding concrete.

Eligehausen and Balogh [14] and Rodriguez et al. [12, 13] carried out many tests on studs fixed in cracking concrete block. According to the crack width and the number of line cracks cutting the anchor, the decrease in loading capacity ranges from 23 to 40% of the bearing capacity in uncracked concrete block. Mayer and Eligehausen [15] also carried out tests on anchor plate with four rods in cracked concrete. The line cracks cut 1, 2, 3 or 4 rods. The authors concluded that the anchor strength reduction is approximately equal to the strength...
reduction for a single anchor in a crack, and that the overall performance is only slightly affected by the positioning of anchors relative to the cracks. All the authors note a large scattering of the results in case of tests in cracked concrete.

In existing works, the applied loadings are mainly in static or fatigue [16] but rarely representative of a seismic loading [17]. However, Solomos and Berra [18] carried out impact tests on studs with the Hopkinson bar technique and showed that, for strain rates ranging from 1 s\(^{-1}\) to 10 s\(^{-1}\), dynamic loading capacity is increased between 1.5 and 1.8 times compared with static capacity.

3 TEST SETUPS

The experimental campaign is carried out on real size anchorages (scale 1). The anchor plate dimensions are 250x250x25 mm\(^3\). They are fastened by four headed anchor rods made up of steel (S235) smooth bar (20 mm in diameter) with at their end an anchor washer (60 mm in diameter and 20 mm thick). The steel yield strength is 365 MPa and the yield strain is 1830.10\(^{-6}\). The anchorages used in nuclear power plants have an embedment depth \(h_{ef}\) equal to 310 mm. Four other depths are also tested (80, 100, 120, 130 mm) in order to have a tensile breakout of concrete. The test program is summarized in Table 1. Moreover, tests on single rods with the same embedment depths are done in order to study the group effects. The anchors are cast-in-place in a reinforcing concrete block (2250 x 1850 x 600 mm\(^3\)) which is large enough to avoid splitting failure and edge effects. The minimal reinforcement recommended by EDF is installed. It consists of one layer of ribbed bar (20 and 25 mm in diameter) in both directions on the upper and lower part of the block. Some stirrups (12 mm in diameter) are also installed only to maintain the two layers of reinforcement. The reinforcing rate is 0.64 %. The concrete grade used is C40/50.

Table 1: Description of tension test specimens

<table>
<thead>
<tr>
<th>Type of tested specimen</th>
<th>Test</th>
<th>Concrete compressive strength at time of testing (MPa)</th>
<th>Effective embedment (h_{ef}) (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anchor plate</td>
<td>T1-310</td>
<td>49.8</td>
<td>310</td>
</tr>
<tr>
<td></td>
<td>T2-310</td>
<td>55.5</td>
<td>310</td>
</tr>
<tr>
<td></td>
<td>T3-80</td>
<td>55.2</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>T4-100 (70) *</td>
<td>55.9</td>
<td>70</td>
</tr>
<tr>
<td></td>
<td>T4-120</td>
<td>55.9</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>T4-130</td>
<td>55.9</td>
<td>130</td>
</tr>
<tr>
<td>Single anchor rod</td>
<td>T3-80-U</td>
<td>55.2</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>T3-100-U</td>
<td>55.2</td>
<td>100</td>
</tr>
<tr>
<td></td>
<td>T3-120-U</td>
<td>55.2</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>T3-130-U</td>
<td>55.2</td>
<td>130</td>
</tr>
</tbody>
</table>

*In test T4-100(70), the embedment depth was initially 100 mm. However, in test T3-120-U, the failure by a concrete breakout cone wrenched the concrete cover near two rods. Consequently, the mean embedment depth (70 mm) of four rods was used.
The concrete block is fixed by means of two metal sections connected to the test slab with four prestressing bars (Figure 1). No confining pressure is applied around the anchorage. The hydraulic jack is fixed to the anchorage by two symmetrical rods. The quasi-static tensile loading was displacement controlled with a loading rate of $1 \text{ mm min}^{-1}$. The load is applied until the anchor fails.

The measurements are stored at an acquisition frequency of 2 Hz. The data recorded for tests on anchor plates with headed rod are the following:

- The displacements of the anchor plate with four LVDT in each corner (P1, P2, P3 and P4).
- The strain of the stirrups under the anchor plate (J1) in order to check the appearance of concrete cone failure.
- The strain in the middle of the four rods (J01, J02, J03 and J04).
- The displacements and strain of the upper concrete block surface with the two-dimensional digital image correlation technique.

The data recorded for tests on a single headed anchor rod are the following:

- The slide of the rod with two LVDT (P1 and P2).
- The strain in the middle of the rod with two gages diametrically opposite (J01 and J02).

![Figure 3: Tension test setup](image)
4 MAIN RESULTS OF PULLOUT TESTS

4.1 Tension tests on single anchor rods: T3-80-U, T3-100-U, T3-120-U, T3-130-U

In test T3-80, with an embedment depth of 80 mm, failure is caused by the concrete breakout cone. The concrete cone cuts no stirrups. The estimation of the angle of the cone with the concrete surface is difficult because of the defects related to the test setup (verticality of the anchor, dissymmetry of the load applied) and the upper layer of reinforcement located close to the anchor washer. The mean diameter of the concrete breakout cone at the block surface is 0.45 m (Figure 2) and the mean angle is approximately 25 °.

In tests T3-100-U, T3-120-U, T3-130-U, failure is caused by the breaking of the rod near the fastening with the anchor washer. The load-displacement curves (Figure 3-a) show an increase of the load after the ultimate load is achieved. This is caused by the partial wrenching of the welding between the rod and the washer. The repositioning of the washer involves an increase in the load without reaching the maximum load.

Figure 3-b shows the mean strain of the anchor rod. Only in test T3-80-U, the rod didn’t reach its steel yield strength with a maximum strain of 1430-10^-6. In all tests, the rod strain fits the behavior curve of the steel obtained in a tension test on a sample.
4.2 Tension tests on anchor plates: T3-80, T4-100(70), T4-120, T4-130, T1-310, T2-310

The anchorages used by EDF with an embedment depth of 310 mm (T1-310 and T2-310) failed through the breaking of rods near the anchor washer without any visible damage to the concrete on the surface. In tests T3-80, T4-100(70), T4-120, failure is caused by the concrete breakout cone and the anchor rods did not reach their yield strength. The concrete cone cuts no stirrups. The failure is brittle with displacements of the anchor plate lower to 0.8 mm (Figure 4-a).

Figure 4-b shows the strain of the stirrup under the anchor plate. The significant increase in strain corresponds to the beginning of the cracks involving the uplift of the upper layer of reinforcement. In test T3-130, the anchorage failed through the breaking of the steel rod near the anchor washer. However, the strain of the stirrup show that a failure by the concrete breakout cone also appeared.

![Figure 4: (a) Measured displacements of the anchor plate at the concrete block surface – (b) Measured strain of stirrup for tests on anchor plates.](image)

Figures 5-a and 5-b show that the upper layer of reinforcement caused a flattening of the concrete breakout cone in concrete cover. The internal cracks propagate following a horizontal plan along the layer of reinforcement. The final collapse is due to a concrete breakout cone or a flexure failure of the concrete cone with the development of cracks at the corner of the anchor plate. However, the reinforcement does not seem to modify the ultimate load of the anchorage which corresponds to the creation of the first cracks.
5 CONCLUSION

This experimental campaign enables the optimum embedment depth for the headed anchor tested in uncracking concrete to be identified. The 130 mm embedment depth is the frontier depth between a concrete breakout cone and a failure of the steel rod. The longitudinal reinforcement causes a flattening of the concrete breakout cone with a propagation of the cracks in the concrete cover along the layer of reinforcement. However, the reinforcement does not seem to modify the ultimate load of the anchorage.

A similar experimental campaign is being carried out on anchor rods consisting of a ribbed bar in order to analyze, in particular, bond failure mechanisms. Moreover, the future tests, taking into account the state of cracking of the concrete block, the type of loading and the edge distance, will enable the safety margins for different test configurations to be identified. The total data set will enable the models of concrete behavior to be calibrated as well as the contact laws in the finite element codes used. The goal is to calculate the ultimate strength of these anchorages in untested configurations and improve their service life and durability.

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REFERENCES

SERVICE LIFE ASSESSMENT OF CONCRETE STRUCTURES BASED ON SITE TESTING

Roberto J. Torrent(1) and Luis Fernández Luco(2)

(1) Materials Advanced Services Ltd., Buenos Aires, Argentina, info@m-a-s.com.ar
(2) University of Buenos Aires, Buenos Aires, Argentina, lfernandez@fi.uba.ar

Abstract

The paper proposes a method, called ExpkRef, to assess the service life of structures exposed to carbonation-induced corrosion, based on two principles: a) measuring on site, non-destructively, two fundamental durability properties: air-permeability and depth of the cover concrete; b) taking as reference the conditions (given in EN Standards for defined Exposure Classes) leading to an expected service life of 50 years. It is claimed that this approach constitutes a starting point for developing more realistic and robust service life prediction models.

1 INTRODUCTION

Traditionally, Concrete Codes and Standards have applied the "Deemed-to-satisfy" approach [1] to specify durability requirements. Based on the accumulated experience in many countries, a set of primarily prescriptive rules have been established which, when rigorously observed, would result in a service life typically of 50 years (e.g. [2]). Today, many important structures are designed for service lives of 100, 150 or even more years, which clearly exceed the reach of existing experience with reinforced concrete and, therefore, requires some extrapolation via modelling.

Several models have been developed to predict/estimate the service life of reinforced concrete structures, particularly for those under risk of steel corrosion induced by chlorides or carbonation. For carbonation, the most widely used model is Duracrete [3], which assumes a purely diffusive ("Fickian") mechanism of ingress of CO\(_2\); it is taken as reference.

In this paper, the "Exp-Ref" model, already presented for chlorides [4], is developed for the case of carbonation-induced corrosion.

2 THE DURACRETE MODEL FOR CARBONATION

The Duracrete model is based on assuming that the penetration of the carbonation front in concrete can be predicted through Eq. (1):

\[ x = \sqrt{2 \cdot C_s \cdot t \cdot D_0 \cdot (t_0 / t)^n \cdot k_c \cdot k_e} \] (1)
x = penetration of carbonation front (mm) after t (years) of exposure
Cs = surface concentration of CO₂ (ppm)
D₀ = coefficient of diffusion of concrete to CO₂ considered/measured at age t₀ (typically 28 days)
n = "ageing exponent", reflecting the rate of decay of the coefficient of diffusion with time, due to hydration beyond age t₀
kₑ = curing factor, smaller the longer the moist curing
kₑ = environment factor, depending on the exposure condition and cement type

Since Duracrete adopts a semi-probabilistic approach, Eq. (1) is further modified by using "design" values for most variables, involving characteristic values and partial factors.

The time of initiation of corrosion Tᵳ is the time (t) when the carbonation front (x) has reached the outermost reinforcing steel (c). Therefore, from Eq. (1), Tᵳ results:

\[ Tᵳ = \frac{c^2}{2 \cdot C_s \cdot D₀ (t₀/Tᵳ)^n \cdot kₑ \cdot kₑ} \]

Tᵳ = time of initiation of corrosion (years)
c = cover depth (mm)

The application of (2) has some limitations that make the predictions uncertain and/or subjective, namely:

a) The actual cover depth (c) is taken from the specified value, although it is known that it may differ considerably [5]

b) The coefficient of diffusion (D₀) at age t₀ is established on the basis of accelerated tests made on laboratory specimens, applying non-standard test methods.

c) The surface concentration of CO₂ (Cs) is assumed constant at 5.10⁻⁴ kg/m³ (258 ppm), stating that it may be "higher for tunnels or other confined spaces"

d) The ageing exponent (n) varies between 0 (for laboratory tests under RH ≤65%) and 0.43, depending on the binder type and exposure condition

e) The environmental factor (kₑ) is defined only for OPC and OPC+GGBS binders

f) The reduction of D₀ with time (due to further hydration) is a gradual process taking place at the same time when the carbonation front is progressing. Therefore, applying in (2) the full reduction at t = Tᵳ to calculate the penetration of carbonation, overestimates Tᵳ

An important criticism that can be made to this model is that the main materials' characteristics (D₀ and c) are either theoretical ("Theorecrete") or based on laboratory tests ("Labcrete"). The adopted values may deviate significantly from those actually found in the real structure ("Realcrete").

Another weakness of these approaches is that several elusive parameters (Cs, n, kₑ, kₑ), needed for the prediction, can hardly be measured or established accurately and are, hence, rather arbitrary. As they exert a strong influence on Tᵳ, the estimation of service life is by no means robust and may become subjective and prone to manipulation.
3 CONCEPT OF "REALCRETE" AND "COVERCRETE"

The difference between the "as-built" quality ("Realcrete") and that reflected by the results of laboratory tests conducted on cast specimens, prepared, compacted and cured under almost perfect conditions, i.e. "Labcrete", is well known. The effect on durability of much too frequent bad practices such as: insufficient mixing time, bad compaction (especially in the space between the steel bars and the form), and lack or absence of moist curing (affecting harder the most exposed outer concrete layers) is discussed in [6,7]. In [7], the problem of cover to reinforcement is also addressed, highlighting the negative consequences for durability of too thin or too thick cover depths (cracking).

The concrete cover ("Covercrete") is the defence barrier of the structural element against the penetration of external aggressive agents. We find, therefore, the unfavourable situation that this defence barrier is the weakest in terms of quality. Moreover, the cast specimens used for laboratory “penetrability” tests are not representative of that of the “Covercrete”. In fact, the only way of knowing the “penetrability” of the vital “Covercrete” is by mean of site tests.

The same applies to the thickness of the cover concrete that protects the steel. The actual cover seldom coincides with the nominal value [5] and is rarely checked on the finished structure, despite the fact that there are electromagnetic and radar covermeters capable of making a sufficiently accurate assessment of its value [8].

4 THE 'EXP-REF' SERVICE LIFE PREDICTION APPROACH

The "Exp-Ref" method for service life prediction for carbonation-induced corrosion of steel, similar as for chlorides [4], consists of three main elements:

- Non-destructive experimental (hence "Exp") assessment of the 'penetrability' and thickness of the "Covercrete" through the application on site of standardized test methods: air-permeability (and concrete moisture check) [9] and covermeters [10]
- Correlation between the measured coefficient of air-permeability and carbonation rate
- Definition of a reference condition (hence "Ref") with a definite service life attributed

4.1 Air-Permeability as Site Durability Indicator.

So far, the only standard method used to specify and control the "penetrability" of the "Covercrete" on site is the "Air-Permeability on the Structure" [9]. This entirely NDT method is capable of measuring the coefficient of air-permeability (kT) on site in up to 6 minutes [11], producing meaningful results if the procedures prescribed in [9] are followed.

4.2 Correlation between air-permeability and natural carbonation rate

Correlations have been established between kT and accelerated carbonation tests [12,13]. In this case, the correlation is based on test results of mostly OPC concretes, the kT values of which were measured at 28 days, and later exposed to natural carbonation in a laboratory-controlled environment, 20°C and 50% RH [15,16] and 60% RH [14]. The carbonation depth was measured at ages of 500 days [15], 2 years [16] and 3.5 years [14].

Fig. 1 presents the results of carbonation rate (CR), expressed as the carbonation depth (x) divided by the square root of exposure time (t), plotted against the coefficient of air-permeability (kT) measured at 28 days. Eq. (3) gives the best correlation (R = 0.89) to the results, with CR in (mm/√y) and kT in (10^-16 m²).
This type of relation has been found also for old structures and used to assess the service life of a Museum in Tokyo [17] and of precast elements for the Port of Miami Tunnel [18].

\[ CR = \frac{x}{\sqrt{t}} = 1.8 \cdot \ln (174 \cdot kT) \quad \text{or} \quad CR = 0 \quad \text{if} \ kT < 1/174 \quad (3) \]

Now, the carbonation rate can be derived from Eq. (1), remembering that, for laboratory tests with RH ≤ 65%, is \( n = 0 \) and \( k_e \) and \( k_c \) (already included in \( kT \)) can be assumed as 1.0.

\[ CR = \frac{x}{\sqrt{t}} = \sqrt{2 \cdot C'_s \cdot D_0} \quad (4) \]

where \( C'_s \) is the \( \text{CO}_2 \) concentration in the laboratory environment.

Combining (3) and (4) we can write:

\[ 2 \cdot D_0 = \frac{3.24}{C'_s} \cdot \ln^2 (174 \cdot kT) \quad (5) \]

Introducing (5) in (2), and eliminating \( k_c \), because the effect of curing is already included in the site measurement of \( kT \), we have for a concrete under a given exposure class:

\[ T_i = \frac{C'_s}{C_s} \cdot \frac{c^2}{3.24 \cdot \ln^2(174 \cdot kT) \cdot (t_0/T_i)^n \cdot k_c} \quad (6) \]

Eq. (6) allows us to calculate the initiation time \( T_i \), now as function of the air-permeability (kT) and the cover depth (c), both measured on the structure. The problem is that the uncertain parameters \( C_s, n, k_c \) are still present in the formula.

4.3 Reference Condition
After many years of development, experts from most European countries have agreed on a common classification of exposures [2] covering all the conditions likely to be found in Europe (see Table 1 for a description of carbonation-induced corrosion exposure classes).
Secondly and more important, they agreed that concretes with a w/c ratio below a certain w/c$_{\text{max}}$ limit and a cover depth above a certain c$_{\text{min}}$ limit [2,19], if well processed according to [20], are expected to reach a service life of 50 years under a defined Exposure Class. These limits are shown in the first two rows of Table 2.

Table 1 - Exposure Classes for carbonation-induced corrosion environments [2]

<table>
<thead>
<tr>
<th>Class Designation</th>
<th>Description of Environment</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>XC1</td>
<td>a - Dry</td>
<td>- Inside buildings with low air humidity</td>
</tr>
<tr>
<td></td>
<td>b - Permanently wet</td>
<td>- Permanently submerged in water</td>
</tr>
<tr>
<td>XC2</td>
<td>Wet, rarely dry</td>
<td>Many foundations</td>
</tr>
<tr>
<td>XC3</td>
<td>Moderate humidity</td>
<td>Inside buildings w/moderate or high air humidity; external sheltered from rain</td>
</tr>
<tr>
<td>XC4</td>
<td>Cyclic wet and dry</td>
<td>Concrete surfaces subject to water contact, not within exposure class XC2</td>
</tr>
</tbody>
</table>

Table 2 - Requirements for 50 years service life [2,19]

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>XC1a</th>
<th>XC1b</th>
<th>XC2</th>
<th>XC3</th>
<th>XC4</th>
</tr>
</thead>
<tbody>
<tr>
<td>w/c$_{\text{max}}$</td>
<td>0.65</td>
<td>0.60</td>
<td>0.55</td>
<td>0.50</td>
<td></td>
</tr>
<tr>
<td>c$_{\text{min}}$ (mm)</td>
<td>15</td>
<td>25</td>
<td>30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>w/c$_{\text{ref}}$</td>
<td>0.63</td>
<td>0.58</td>
<td>0.53</td>
<td>0.48</td>
<td></td>
</tr>
<tr>
<td>kT$_{\text{ref}} (10^{-16} \text{ m}^2)$</td>
<td>1.41</td>
<td>0.79</td>
<td>0.45</td>
<td>0.25</td>
<td></td>
</tr>
<tr>
<td>c$_{\text{ref}}$ (mm)</td>
<td>25</td>
<td>35</td>
<td>40</td>
<td></td>
<td></td>
</tr>
<tr>
<td>T$_p$ (y)</td>
<td>45</td>
<td>10</td>
<td>10</td>
<td>25</td>
<td>2</td>
</tr>
<tr>
<td>T$_{\text{ref}}$ (y)</td>
<td>5</td>
<td>40</td>
<td>40</td>
<td>25</td>
<td>48</td>
</tr>
<tr>
<td>$\beta$ (y/mm²)</td>
<td>0.24</td>
<td>1.94</td>
<td>0.79</td>
<td>0.39</td>
<td>0.43</td>
</tr>
</tbody>
</table>

To comply with those limits, the target values of w/c and c (reference values), should have a margin respect to the limiting values: w/c$_{\text{ref}}$ = w/c$_{\text{max}}$ - 0.02 and c$_{\text{ref}}$ = c$_{\text{min}}$ + 10 mm.

A relation between gas-permeability and w/c ratio, proposed in [21] and validated for kT results [4] is adopted:

$$\log kT_{\text{ref}} (10^{-16} \text{ m}^2) = -3 + 5 \cdot \frac{w}{c_{\text{ref}}} \quad \text{(Eq. 2.1-107 of [20])} \quad (7)$$

Therefore, a concrete structure with a cover depth c$_{\text{ref}}$ and air-permeability kT$_{\text{ref}}$ is expected to have a service life (SL$_{\text{ref}}$) of 50 years, serving as the reference condition (see rows 3-5 in Table 2).

Moreover, we know that:

$$\text{SL} = T_i + T_p \quad \text{(8)}$$
In the case of exposure to chlorides, it is acceptable to assume $T_p = 0$, due to the availability of $O_2$ and moisture and the low electrical resistivity of the chloride-contaminated concrete [4]. For carbonation, this is not the case, and $T_p$ has to be taken into account in the assessment of service life.

Fig. 2 shows the relative carbonation and carbonation-induced corrosion rates as function of the relative humidity of the environment [22]. The location of the 5 exposure classes is also shown schematically. The need to subdivide class XC1 into XC1a and XC1b is now evident, given the different resulting carbonation and corrosion rates. Based on this qualitative description and the quantitative model proposed in [23], the corrosion propagation time $T_p$ (for cracking onset) can be estimated for each class (see row 6 of Table 2).

The $T_{iref}$ values, for the reference service life $SL_{ref} = 50$ y, are shown in row 7 of Table 2.

Now, applying Eq. (6) to the reference condition, we have:

$$T_{iref} = \frac{C_s}{C_s} \cdot \frac{c_{ref}^2}{3.24 \cdot \ln^2(174 \cdot kT_{ref}) \cdot (t_0 / T_{iref})^p \cdot k_e}$$ (9)

Dividing (6) by (9) and assuming that $C_s$ and factor $k_e$ are the same for the same exposure class, and that the decay factor $(t_0 / T_i)^n$, after so many years of hydration, is also the same for the reference condition and for the investigated case, we can write:

$$T_i = \frac{c^2}{c_{ref}^2} \cdot \frac{\ln^2(174 \cdot kT_{ref})}{\ln^2(174 \cdot kT)} \cdot T_{iref}$$ (10)

The service life $SL$ can be computed from Eqs. (11, 12), using the $\beta$ and $T_p$ values of the corresponding exposure class, indicated in rows 6 and 8 of Table 2.

$$SL = \beta \cdot \frac{c^2}{\ln^2(174 \cdot kT)} + T_p$$ (11)
\[ \beta = T_{\text{ref}} \cdot \ln^2(174 \cdot kT_{\text{ref}}) / c_{\text{ref}}^2 \quad (12) \]

Eq. (11) provides an estimate of the service life of a concrete structure SL (years), exposed to a given carbonation-induced corrosion class, as function of the measured cover depth c (mm) and the coefficient of air permeability kT \( (10^{-16} \, \text{m}^2) \), both measured on site. Obviously, the higher the value of c and the lower the value of kT, the longer the service life SL.

A transformation of Eq. (12) allows to estimate the penetration of the carbonation front, as function of the exposure class and the measured value of kT \( (10^{-16} \, \text{m}^2) \):

\[ x = (t / \beta)^{1/2} \cdot \ln(174 \cdot kT) \quad (13) \]

- \( x = \) carbonation depth (mm)
- \( t = \) time (y)
- \( \beta = \) \( (y/mm^2) \), value given in row 8 of Table 2 for each exposure class; from (13) the rate of carbonation for a given kT is inversely proportional to \( \beta^{1/2} \)

For instance, using the values of kT measured on the Port of Miami Tunnel (for assumed classes XC1a/XC3), the carbonation depth with the ExpkRef method can be estimated, comparing them with the values predicted by other models [18], Table 3. The values predicted by "Exp-Ref" (for classes XC1a/XC3) fit well to the other methods, slightly on the safe side.

| Predicted Carbonation Depth (mm) @ 150 years after method: | Analytical Based on kT site measurements | 
| --- | --- | --- | --- |
| Duracrete | Parrott | Old Structures | Exp-Ref (XC1a / XC3) |
| **72 h Curing (kT= 0.027)** | 24 | 14 | 38 | 38 / 30 |
| **18 h Curing (kT= 0.057)** | 35 | 19 | 48 | 57 / 45 |

5 CONCLUSIONS

The Exp-Ref approach, based on measurements conducted directly on the structure ("Realcrete"), in particular of the air-permeability and thickness of the "Covercrete", has been applied to carbonation-induced corrosion. Since the reference condition, on which the model is based, corresponds to 50 years of service life, the extrapolation is for shorter periods than for a model starting from time zero.

The proposed method has two main advantages over other methods:
- a) it is concerned with the quality actually achieved in the as-built structure, including important factors for the durability performance of the structure such as concrete production, placement, compaction, finishing and curing, as well as proper placement and fixing of the steel reinforcement
- b) there are no coefficient or factors to be chosen freely by the user, except the definition of the Exposure Class

It is a starting point towards more realistic and robust service life prediction models.
REFERENCES

ENVIRONMENTAL SUSTAINABILITY PERFORMANCE OF RESIDENTIAL BUILDINGS' THERMAL RETROFITS

Ikbal Cetiner (1) and Ecem Edis (2)

(1) Istanbul Technical University, Faculty of Architecture, Istanbul – cetinerikb@itu.edu.tr
(2) Istanbul Technical University, Faculty of Architecture, Istanbul – ecem@itu.edu.tr

Abstract
Environmental sustainability is one of the greatest concerns of the world, and building sector has a considerable effect on achieving a sustainable environment because of the impacts associated with but not limited to the manufacturing of building materials, construction of the buildings, energy use in the operation period, and demolition and waste disposal. Thermal retrofitting of existing buildings is a measure in this sense which will reduce environmental impacts associated with energy use in the operation period, and also that will prevent impacts associated with construction of new buildings. In this respect, a research project was completed on thermal retrofitting of residential buildings in Istanbul (Turkey), and a database was built for selection of efficient retrofit alternatives. As a further study, using building types determined in this research, environmental sustainability performance of building retrofits in two other cities of Turkey with different climatic conditions, namely Ankara and Izmir, were studied in order to evaluate the effect of climatic conditions on environmental sustainability performance.

In the paper, the completed research project is briefly explained at first, methods and procedures of the current study and the results of three cities are then presented, and finally the efficiencies of retrofits are comparatively discussed for different cities.

Keywords: Environmental sustainability, retrofit, residential building, climate.

1 INTRODUCTION
Depletion of natural resources including both energy and others such as minerals or water, and pollution of environment caused by all kinds of human activities are two of the significant problems in the world. Taking necessary measures, first, for preserving current condition of the environment, and then, for reversing the harm we caused is a policy accepted all over the world. Buildings, as objects providing the necessary interior environment we need to perform our activities, and construction industry, which creates the necessary built-environment we need, have a considerable and widely accepted effect on achieving the aforementioned policy.
Considering that thermally retrofitting the existing (residential) buildings, rather than demolishing them to construct new ones, is a measure that will help both preserving natural resources and decreasing pollution caused by manufacturing, construction and space heating activities, various research studies had been made in building community. Some of them take the subject into account theoretically/strategically [1, 2, 3], and some others directly analyse performances of retrofitting alternatives [4, 5]. Similarly, a research project considering the case in Turkey was designed, and completed in 2011 by the authors [6]. One of the main objectives of the study was to evaluate both economic and environmental efficiency of various thermal retrofit alternatives considering buildings’ characteristics such as remaining service life, window to wall ratio (WWR) or plan type. A database was generated for Istanbul, as the output of the study that can be used either by the owners or constructors, for deciding on what type of thermal retrofits would be efficient for their building. Enlarging the database for different cities of Turkey was planned as well. Considering that climatic conditions affect buildings’ energy consumption [7, 8, 9], initially, the environmental sustainability performance of thermal retrofits are being investigated for three different cities of Turkey, namely Istanbul, Ankara and Izmir. These cities are selected as they have the highest urban population in Turkey in the given order [10], and represent three of the four climatic regions defined in the mandatory Turkish Standard on thermal insulation of buildings [11], where Istanbul is primarily classified under Marmara type with a temperate climate as a transition region, Ankara is under terrestrial type with cold winters, and hot-dry summers, and Izmir is under Mediterranean type with hot-dry summers, and warm-humid winters [12]. Climate types of these cities according to different classification schemes can be found in [13].

In this paper, in relation with this further research study, brief information on the research project completed in 2011 is given initially for providing necessary background information. Methods and procedures used in the comparative evaluation of environmental sustainability performances at different cities are then explained, and findings are presented. Finally, effectiveness of these thermal retrofits is comparatively discussed for different cities.

2 THERMAL RETROFITTING OF RESIDENTIAL BUILDINGS IN ISTANBUL

In the research study, a method was developed for sustainability assessment of thermal retrofits, and a database was generated based on the use of predefined building types and predefined retrofit alternatives for detached residential buildings with natural gas fired central space heating system. For this purpose, 60 buildings with these characteristics, located at six different neighbourhoods of Istanbul were randomly selected, and information such as distances to other buildings, number of stories, number of apartments at one storey, and assembly of building elements was then gathered through field surveys and project analyses. As the result of this investigation, two main building types, namely Types A and B in terms of plan shape, and two sub-types for each of them in terms of the floor area of one storey were identified. For determining the thermal retrofits, material manufacturers and construction firms working on retrofitting were contacted for identifying the mostly preferred construction techniques and thermal insulation materials, which were identified to be the exterior application of either extruded polystyrene (XPS), expanded polystyrene (EPS) or stone wool (SW) at the exterior walls, and laying glass wool (GW) on the attic floor at the roofs. As additional retrofit alternatives; (i) replacement of single-pane wooden framed windows with double-pane windows of either wooden or PVC frame, (ii) thermally insulating the floor
between heated apartments and the unheated basement, and (iii) thermally insulating the walls enclosing the staircase with the same materials used at the exterior walls were defined as well.

In the evaluation of environmental sustainability, life cycle assessment (LCA) method was used. Environmental impacts of buildings without any retrofit were determined considering the space heating energy required during their remaining lives. For the retrofitted cases, raw materials and energy required for the manufacturing of retrofit materials, and material transportation and construction energy were considered additionally [14]. In the evaluation of economic sustainability, life cycle cost (LCC) method was used, and costs associated with the aforementioned items for the environmental sustainability were considered. Energy consumption of building types were determined by EnergyPlus software, and environmental impacts were determined by SimaPro software and its Ecoinvent database.

In the determination of environmental and economic performances of retrofit alternatives, the same approach was used and ‘the total impact of a building type without any retrofit’ was compared with ‘the total impact of the same building type when retrofits are applied’ [15]. Equation used for determining environmental performance is given as an example in Equation (1). The overall performances of retrofit alternatives are then determined by Equation (2).

\[
NR_{i,j} = \frac{(NI_i - NI_j) \times 100}{NI_i} \quad \text{Equation (1)}
\]

\[
SP_{i,j} = \frac{(NR_{i,j} \times mn) + (CR_{i,j} \times mc)}{100} \quad \text{Equation (2)}
\]

Where, \(NR\) is environmental performance, \(NI\) is environmental impact (Ecopoints), and the indices \(i\) and \(j\) are building type and retrofit alternative planned to be used respectively in Equation (1), and \(SP\) is sustainability performance (-), \(NR\) is environmental performance (-), \(CR\) is economic performance (-), \(m\) is importance ratio (%) in Equation (2).

3 SUSTAINABILITY PERFORMANCE OF THERMAL RETROFITS IN DIFFERENT CITIES OF TURKEY

3.1 Method and procedures

In the completed research project, as aforementioned, two main building types were determined through the field studies, which were Type A with a square-like plan and Type B with a rectangular plan, and there were two sub-types of each in terms of floor area. For the rectangular type, there were also two sub-types in terms of orientation of the building. Three different WWRs were determined for each building type, which were 10%, 20% and 30%. Service life of buildings was accepted to be 50 years, and buildings at the ages of 15, 20, 25 and 30 were studied. As the buildings at those ages were generally middle-rise buildings, buildings were accepted to have six stories, one of which was unheated basement. In the current study on evaluating the effect of exterior environmental conditions, the same building types determined for Istanbul were used, and one sub-type of each main type with a WWR of 20% for the ages of 15 and 30 were studied (Table 1). For the rectangular type, only the one, whose long side was oriented to north-south, was studied.

<table>
<thead>
<tr>
<th>Building type</th>
<th>Plan type</th>
<th>Long side facing</th>
<th>Floor area</th>
<th>WWR</th>
<th>Age</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>Square-like</td>
<td>N/A</td>
<td>210.25 sq.m</td>
<td>20%</td>
<td>15, 30</td>
</tr>
<tr>
<td>B1-1</td>
<td>Rectangular</td>
<td>North-South</td>
<td>231 sq.m</td>
<td>20%</td>
<td>15, 30</td>
</tr>
</tbody>
</table>
In the completed research project, typical building element assemblies of existing buildings were determined by analysing their projects available at the archives of municipalities, and the thicknesses of thermal insulation materials were determined considering the information gathered from construction firms on generally applied thicknesses. In the current study, on the other hand, as performing field studies, project analyses and construction firms interviews at different cities would be time consuming, building element assemblies determined for the existing buildings at Istanbul are used for all other cities, and thermal insulation thicknesses are determined considering the U values defined for building elements in the mandatory Turkish Standard on thermal insulation of buildings [11]. Therefore, computations associated with Istanbul were renewed considering the thermal insulation thicknesses determined. All buildings investigated during the field studies had reinforced concrete (R.C.) structural frame. Considering the thermal bridges at the structural members, necessary thicknesses for the thermal insulations were calculated taking into account the assembly at the location of R.C. beams rather than the one at infill walls. In addition, in this paper, impacts of retrofitting with two different thermal insulation materials, namely EPS and SW, and renewal of windows with double-pane PVC frame are presented only (Table 2).

Table 2: Information on the retrofitted building elements, except renewal of window system.

<table>
<thead>
<tr>
<th>Building element</th>
<th>Assembly(a) ((d; \lambda))</th>
<th>U value(b) - insulation material ((m; d))(c)</th>
</tr>
</thead>
</table>
| Vertical exterior envelope above ground | Exterior rendering \((3; 1.4)\)  
Thermal insulation \(d\)  
R.C. beam \((25; 2.1)\)  
Interior plaster \((2; 0.87)\) | Istanbul: 0.60  
EPS/SW; 5  
Ankara: 0.50  
EPS/SW; 6  
Izmir: 0.70  
EPS/SW; 4 |
| Roof                                | Thermal insulation \(d\)  
R.C. floor slab \((10; 2.1)\)  
Interior plaster \((2; 0.87)\) | Istanbul: 0.40  
GW; 8  
Ankara: 0.30  
GW; 11  
Izmir: 0.45  
GW; 7 |
| Floor above unheated basement      | Wooden parquet \((2; 1.12)\)  
Cement screed \((3; 1.4)\)  
R.C. floor slab \((10; 2.1)\)  
Thermal insulation \(d\)  
Interior plaster \((2; 0.87)\) | Istanbul: 0.60  
EPS/SW; 5  
Ankara: 0.50  
EPS/SW; 6  
Izmir: 0.70  
EPS/SW; 4 |
| Projected floor                    | Wooden parquet \((2; 1.12)\)  
Cement screed \((3; 1.4)\)  
R.C. floor slab \((10; 2.1)\)  
Thermal insulation \(d\)  
Interior plaster \((2; 0.87)\) | Istanbul: 0.60  
EPS/SW; 5  
Ankara: 0.50  
EPS/SW; 6  
Izmir: 0.70  
EPS/SW; 4 |
| Walls enclosing staircase          | Interior plaster \((2; 0.87)\)  
Thermal insulation \(d\)  
Hollow clay brick \((9; 0.45)\)  
Interior plaster \((2; 0.87)\) | Istanbul: 0.60  
EPS/SW; 3  
Ankara: 0.60  
EPS/SW; 3  
Izmir: 0.60  
EPS/SW; 3 |

Notes and abbreviations

- \(a\): Assembly either from top to down or exterior to interior.
- \(b\): Max. thermal transmittances (W/m²K) used in the calculations considering values in [11].
- \(c\): Thermal conductivity of EPS, RW and SW are taken as 0.035 W/m.K.
- \(d\): Location of thermal insulation when retrofitted. Thicknesses for different cities are given in the cells of the same row.

\(d\): Thickness (cm)  \(\lambda\): Thermal conductivity (W/m.K)  \(m\): Thermal insulation material
3.2 Results

Annual space heating energy consumptions of building types with and without retrofitting are given in Figure 1 for different cities of Turkey. In order to understand the effect of building orientation on heating energy consumption, building B1-2, whose long side is facing east-west, is studied as well. Analysis of heating energy consumptions shows that;

- Thermal retrofit of all building types decreases the energy consumption at all cities;
- Annual consumption is the greatest at Ankara followed by Istanbul and Izmir, when buildings of same type with same retrofitting conditions are compared with each other;
- Annual heating energy consumption of building B1-2 is always higher than that of building B1-1 at all cities.

![Energy Consumption Chart](image)

Figure 1: Annual space heating energy consumption of building types with and without retrofitting and located at different cities of Turkey.

As the objective is to evaluate the effect of climatic conditions on the environmental performance of building retrofits, information on the exterior air temperature and solar radiation are given in Figure 2 for the cities considered. General analysis of these shows that;

![Temperature and Solar Radiation Chart](image)

Figure 2: Monthly statistics for dry bulb temperatures (°C), and global solar radiation on horizontal surface (Wh/m²) [16].
Monthly minimum exterior air temperature is always lower in Ankara than in other cities;
Monthly global solar radiation on horizontal surface is always higher in Izmir than in other cities, during both summer and winter seasons.

Environmental performances of two buildings types retrofitted with two different insulation materials are given in Figure 3 for different cities of Turkey and for different building ages. Analysis of environmental performances shows that;

- Environmental performance is always higher when EPS is used, when compared with the use of SW at the same building;
- Environmental performance of a building at the age of 15 is always higher than the same building at the age of 30;
- Environmental performance of building A1 is always higher than building B1-1’s, except the ones located in Izmir, when buildings at the same age retrofitted with the same material are compared with each other;
- Both for buildings A1 and B1-1, the highest performance is achieved in Izmir followed by Istanbul and Ankara, when buildings at the same age retrofitted with the same material are compared with each other.

Figure 3: Environmental performance of buildings types retrofitted with different insulation materials at various cities of Turkey.

As mentioned in Section 2, environmental performance used in this study is dimensionless, and reflects the percentage of performance gain (or loss in some cases). Therefore, environmental impact scores for different end point damage categories are given in Figure 4 for building A1 located in Istanbul as an example. Analysis of these scores shows that;

- Ecosystem quality impact is in negligible amounts for all cases;
- The highest impact occurs in relation with resource use followed by climate change and human health;
The impact of building without any retrofit is always higher than the retrofitted ones for all damage categories;

- The impacts of a 30 years old building are lower than that of a 15 years old building, as the remaining life of the former one is shorter (i.e. 20 years) than the latter (i.e. 35 years), and thus the amount of heating energy consumption during its remaining life is smaller.

\[\text{Figure 4: Building A1 located in Istanbul, with and without retrofit - Endpoint scores for damage categories.}\]

\[\text{4 DISCUSSION AND CONCLUSION}\]

In general, it is observed that thermal retrofitting increases the environmental performance of residential buildings located at different cities of Turkey with different climatic conditions. These performance values range between 55-64 points, and the highest improvement is achieved in climate change and resource use categories.

Environmental performances of buildings in Istanbul and Ankara are similar to each other, although minimum air temperature of Ankara is lower than that of Istanbul, up to 18°C. As the mandatory Turkish Standard [11] defines different thermal transmittance values at these cities, where limit values are lower in Ankara, similar performance gains can said to be meaningful. In Izmir, on the other hand, environmental performances of buildings are higher than those of located in other cities, especially for building B1-1, although higher limit thermal transmittance values are defined for Izmir in the aforementioned standard. Maximum air temperature of Izmir is usually higher than others’, up to 10°C, especially during summer time, and the amount of solar radiation is also higher than others’ up to ca. 1100 Wh/m². As the summer time conditions are more severe in Izmir, it can be assumed that primary driver of the limit transmittance values in Izmir is the cooling loads occurring during summer. Therefore, increase in the sustainability performance is found meaningful as space heating energy consumption is only considered in this study.

Another significant variation observed in the results is the performance of building B1-1 at different cities. In Istanbul and Ankara, it has a lower performance than building A1’s, while it is the opposite in Izmir. Building B1-1’s long facades are facing north-south, and the annual energy consumption of its retrofitted state is the lowest in Izmir, while in building B1-2 with long facades oriented east-west, the annual heating energy consumption of its retrofitted state is the highest. Therefore, the increase observed in environmental performance of building
B1-1 may be explained by high solar radiation in Izmir, and increased solar heat gain through south façade due to increased façade area. However, further analysis considering different WWRs and floor areas will be helpful to strengthen this assumption.

In conclusion, this initial study on understanding the effect of weather conditions on environmental performance of thermal retrofit of residential buildings showed that, in addition to heating energy consumption, which is important in the case of Istanbul and Ankara, cooling energy consumption, which is especially important in the case of Izmir, should be also studied to make a holistic comparison among different climates.

REFERENCES

Abstract
The specifications of the construction materials and the normalization of test methods are essential in perfecting the manufacturing processes, in supporting selection and acquisition and in those products quality control.

The properties of factory made rendering and plastering mortars used on walls, ceilings, columns and partitions are normalized. The specifications include the essential definitions and the final performance requirements. They also comprise the relevant characteristics to designate the different categories of mortars. Each category of mortars entails a set of essential requirements in which the evaluation and test method is identified.

However, in the last few years, the market has evolved and the properties of mortars have been being altered and adapted to satisfy new consumption demands. Repair mortars, plasters for interior temperature or interior relative humidity control are being created. The development of a cement and lime based plaster containing phase change materials (PCM) is an example of a new coating material for walls and ceilings designed for controlling interior temperatures of buildings.

With this background, the purpose of this paper is to present the current specifications and test methods established by CEN in the ambit of external renders and internal plasters, and suggest methods for the evaluation of particular requirements for the new developed materials.

With this paper, the authors wish to bring attention to the need of broadening the scope of the present normalization, hoping to contribute in this way to the evolution of the manufacturing processes and to the improvement of the finished products quality.

1 INTRODUCTION
Mortars consist in a mix of one or more inorganic binders, aggregates, water and sometimes admixtures and/or additions, used as external renders or internal plasters [1]. They can be used as cladding on walls, ceilings, columns and partitions.

In this paper distinct types of renders and plasters are identified. Each type is adequate to different fields of use and exposure conditions, and entails a set of properties and essential requirements in which the evaluation and test method is identified. The properties and
requirements imposed by current standardization and the test methods used to evaluate them are summarized.

This paper presents the development of a new composite material based on the incorporation of phase change materials (PCM) in cement based plastering, to be used as inner wall covering in order to increase thermal comfort inside buildings. An experimental campaign is settled for the characterization of this new plaster and the results are compared to those obtained for conventional reference mortars, which are frequently used as interior coatings.

This research work also aimed to set adequate bases for the durability assessment of PCM mortars.

2 RENDERING AND PLASTERING MORTARS

2.1 Types, classes and properties

The European standard EN 998-1 [1] is applicable to rendering or plastering mortars for internal or external use, respectively, on walls, ceilings, columns and partitions, including either factory-made or semi-finished mortars. In the range of mortars to which it applies, six distinct types are identified according to properties and/or intended use (see Table 1).

Table 1: Types of rendering/plastering mortar according to properties and/or use [1].

<table>
<thead>
<tr>
<th>Abbreviated terms</th>
<th>Types of rendering/plastering mortars</th>
</tr>
</thead>
<tbody>
<tr>
<td>GP</td>
<td>General purpose rendering/plastering mortar</td>
</tr>
<tr>
<td>LW</td>
<td>Lightweight rendering/plastering mortar</td>
</tr>
<tr>
<td>CR</td>
<td>Coloured rendering mortar</td>
</tr>
<tr>
<td>OC</td>
<td>One coat rendering mortar for external use</td>
</tr>
<tr>
<td>R</td>
<td>Renovation mortar</td>
</tr>
<tr>
<td>T</td>
<td>Thermal insulating mortar</td>
</tr>
</tbody>
</table>

Different fields of use and exposure conditions require mortars with different properties and performance levels [1]. For this purpose, categories related to three different properties, namely: compressive strength; capillary water absorption; and thermal conductivity; are used. Thus, hardened mortars shall be classified according to categories presented on table 1 of EN 998, part 1, charter 5 [1].

The remaining properties are not divided into categories, whoever properties relevant to the intended use and/or type of product shall be declared. The characterization tests must be performed laboratory following specific rules to this effect, with particular emphasis to EN 1015 [2] in its various parts (see §.2.2.).

2.2 Requirements

According to the foregoing, to be marketed and applied in Portugal, mortars shall meet the requirements imposed by current standardization. In the development of a new mortar, of GP or LW type, the final product shall meet the requirements shown in Table 2.
Table 2: Summary of requirements for GP and LW hardened mortars [1].

<table>
<thead>
<tr>
<th>Properties</th>
<th>Test Method</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry bulk density [kg/m$^3$]</td>
<td>EN 1015-10 [2]</td>
<td>Declared range of values</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Declared range of values ≤ 1300 kg/m$^3$</td>
</tr>
<tr>
<td>Compressive strength [N/mm$^2$]</td>
<td>EN 1015-11 [2]</td>
<td>CS I to CS IV</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CS I to CS III</td>
</tr>
<tr>
<td>Adhesion [N/mm$^2$] and fracture pattern FP: (A, B or C)</td>
<td>EN 1015-12 [2]</td>
<td>≥ Declared value and fracture pattern (FP)</td>
</tr>
<tr>
<td>Thermal conductivity [W/(m·ºC)]</td>
<td>EN 1745 [3], Table A.12</td>
<td>Tabulated mean value (P = 50 %)</td>
</tr>
<tr>
<td>Reaction to fire (class)</td>
<td>EN 13501-1 [4]</td>
<td>The appropriate reaction to fire class shall be declared.</td>
</tr>
<tr>
<td>Durability ($\lambda_{10,dr}$) (intended use in external elements)</td>
<td>Until a European method of test is available, the freeze-thaw resistance shall be evaluated and declared to the provisions valid in the intended place of use of the mortar.</td>
<td></td>
</tr>
</tbody>
</table>

Beyond the limits imposed on the properties shown in Table 2, the applicability of renderings and plasterings, entails checking the fulfillment of the responsibilities of the manufacturer, the planner and the worker.

The manufacturer shall ensure that the CS, W and T categories are identified, wherever applicable to the type of mortar under study. General propose (GP) or light weight (LW) mortars types can belong to any categories regarding mechanical strength (CSI to CSIV) and capillary water absorption (W0 to W2), respectively. Categories T1 and T2 only apply to thermal insulation mortars: T type mortars. The manufacturer shall also verify that the remaining properties identified in the standard are declared and whether the product complies with the announced properties.

The planner has the responsibility to ensure that it was chosen a suitable product for its intended application. Finally, the worker must respect the rules of preparation and application of the product (substrate conditions, application techniques, thicknesses, etc.) disclosed, in general, in the datasheets or product in the bibliography of art.
3 PCM PLASTERING MORTAR

3.1 Summary

The main goal of this section is to evaluate the feasibility of a new composite material, with inclusion of micro-encapsulated paraffin in cement based mortars, to be used in interior coatings for buildings. In order to be marketed within the European Union (EU) plastering mortars should respond to European Norm EN 998-1[1]. Therefore, the development of a plastering mortar should comply with characteristics presented in Table 2 where test parameters and target properties for mortars are identified. In the scope of this work, apart from performing tests on the properties mentioned above (Table 2) while developing a PCM mortar, complementary properties like enthalpy and specific heat were also evaluated.

3.2 Components and formulation

The achieved mixture (used later on for further characterization) resulted of a set of preliminary analyses performed with the goal of evaluating the technical viability of the incorporation of microencapsulated PCM (industrially manufactured) into cement/lime based plastering mortar. Due to initial problems associated to the high cracking proneness of mortars containing PCM, the mixture composition was adjusted by an iterative process of trial and error, until the samples of the hardened mortar presented no visible cracking after the first hours of drying in controlled environment (with a temperature of 22±2°C and a relative humidity of 50±5%). The trial samples, with 10 cm×10 cm×1 cm, resulted of spreading of the fresh mortar over a brick surface. Besides all conventional components used in common plastering mortars: cement, lime, sand and/or industrial fillers and different kinds of additions (resins, fibers, cellulose deviants and others) the trial mortars involved the use of PCM. The following text provides further information on the several constituents mentioned above, encompassing their main characteristics. The cement used is a type I class 42,5R according to NP-EN 197-1 [5]. The industrial aggregate used was calcium carbonate with 0.5–1.5mm granulometric distribution. Resins (polymer ethyl-vinyl acetate - VAE) were applied in some of the trial formulations, with the primary function of improving adherence to the substrate (brick or concrete substrate) on low cement concentrations. However, they are known to reduce the blend’s elasticity. The added fibers – polyacrylonitrile (PAN) 6 mm fibbers – and aluminium powder, are meant to compensate and redistribute the internal tension in the mortar, thus turning the shrinkage crack formation invisible. Cellulose deviants (cellulose ether) are used for high consistency development, as they impart good workability and enhance water retention considerably [6]. A micro-encapsulated paraffin powder (based on polymethyl methacrylate, highly cross-linked paraffin mixture), with commercial designation of “Micronal DS5008x”, is used as PCM, with 23°C of melting point, an enthalpy of 100–110 kJ/kg and 300 kg/m3 density, approximately [7].

After a trial-and-error procedure that consisted in casting several test specimens (formulations A to L shown in Table 3, together with the corresponding compositions), adequate behavior (i.e., absence of drying cracks) was obtained with formulations J, K and L. The formulation of a reference mortar, named as REFM, is also shown in Table 3, to better illustrate the similitude between formulation L and the formulation of a common plastering, which is commercially available for interior coating.
Table 3: Mix proportions of formulations A to L and REFM [8].

<table>
<thead>
<tr>
<th>Formulation</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
<th>K</th>
<th>L</th>
<th>REFM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement (hydraulic binder)</td>
<td>40,00</td>
<td>40,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>10,00</td>
<td>10,00</td>
<td>10,00</td>
<td>10,00</td>
<td>10,00</td>
<td>12,00</td>
<td></td>
</tr>
<tr>
<td>Lime (hydraulic binder)</td>
<td></td>
<td></td>
<td>20,00</td>
<td>20,00</td>
<td>10,00</td>
<td>10,00</td>
<td>5,00</td>
<td>5,00</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2,48</td>
</tr>
<tr>
<td>Sand (filler)</td>
<td>10,00</td>
<td>9,90</td>
<td>24,92</td>
<td>49,92</td>
<td>29,92</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>65,00</td>
</tr>
<tr>
<td>Calcium carbonate (industrial filler)</td>
<td>20,00</td>
<td>49,87</td>
<td>49,85</td>
<td>42,80</td>
<td>42,70</td>
<td>52,80</td>
<td>52,65</td>
<td>57,65</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>20,00</td>
</tr>
<tr>
<td>Resins (VAE, Tg = 15ºC)</td>
<td>0,02</td>
<td>0,02</td>
<td>0,02</td>
<td>0,02</td>
<td>0,02</td>
<td>0,02</td>
<td>0,02</td>
<td>0,02</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fibers (PAN, with 6mm)</td>
<td>0,20</td>
<td>0,20</td>
<td>0,20</td>
<td>0,20</td>
<td>0,20</td>
<td>0,20</td>
<td>0,20</td>
<td>0,20</td>
<td></td>
<td></td>
<td></td>
<td>0,05</td>
<td></td>
</tr>
<tr>
<td>Fibers (Aluminium powder)</td>
<td>0,10</td>
<td>0,10</td>
<td>0,10</td>
<td>0,10</td>
<td>0,10</td>
<td>0,10</td>
<td>0,10</td>
<td>0,10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0,09</td>
</tr>
<tr>
<td>Cellulose ether (µ = 8000 a 12000 mPa.s 2%, 2ºC)</td>
<td>0,10</td>
<td>0,08</td>
<td>0,08</td>
<td>0,08</td>
<td>0,05</td>
<td>0,05</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0,09</td>
</tr>
<tr>
<td>Cellulose ether (µ = 2000 a 3000 mPa.s 2%, 2ºC)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0,05</td>
<td>0,05</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PCM (&quot;Micronal DS5008&quot;)</td>
<td>50,00</td>
<td>50,00</td>
<td>50,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td>25,00</td>
<td></td>
</tr>
<tr>
<td>Calcium stearate (water repellent agent)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0,29</td>
</tr>
</tbody>
</table>

3.3 Characterization and classification: Thermal properties

The characterization of the developed plastering mortar had two main goals: the compliance with normative required values and consequent classification of the PCM mortar; and the identification of the thermal complementary properties given by the presence of the PCM (like enthalpy and specific heat). The hardened mortars of the selected formulation L, now renamed as PCMM (PCM Mortar), were submitted to a set of characterization tests required by European Norm EN 998-1 [1]. The measured properties for PCMM are summarized in Table 3, where the corresponding test method/normalization is shown (the normalizations required for the developed tests are EN 1015 [2] and EN 1745 [3]). In regard to the properties shown in Table 3, it should be remarked that the water vapor permeability coefficient and the specific heat were estimated from the values presented on Table A.12 of EN 1745:2002 [3], as function of the measured density of the composite mortar (1170 kg/m³).

A heat flow meter apparatus was used to determine thermal conductivity of the new composite material according to ISO 8301 [9]. The thermal conductivity measured with this experimental technique was 0.295 W/m K. Also, the melting temperature and the fusion enthalpy were experimentally determined through Differential Scanning Calorimetry (DSC) tests with recourse to a Diamond DSC from Perkin Elmer. A sample of the supplied PCM mortar (powder) of approximately 7.7 mg was tested and an enthalpy of 22.6 kJ/kg was registered. This result came close to what would be expected (25 kJ/kg), assuming that the enthalpy is linearly proportional to the fraction of paraffin amount in the sample (25%), bearing in mind that the PCM is the only material in the mixture to experiment phase
transition (from solid to liquid) in the range of studied temperatures (5–50°C). From these results it may be concluded that the PCM keeps its characteristics when integrated into hardened cement based mortars. Therefore, from now on, it will be considered that the composite mortar containing 25% of PCM has a melting point of 23°C and a melting enthalpy of 25 kJ/kg in a 2°C temperature range.

The comparison of characteristics between PCMM and REFM is presented in Table 4. These results confirm the expected similarity in behavior for the developed plastering mortar in view of the chemically inert characteristics of the PCM, which was introduced in the mix by replacing aggregates.

Table 4: Results of the test performed to the composite mortar with formulation L (PCMM) and comparison to reference mortars (REFM) [8].

<table>
<thead>
<tr>
<th>Test parameter</th>
<th>PCMM (LW)</th>
<th>REFM (GP)</th>
<th>Classification of PCMM / Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry bulk density [kg/m³]</td>
<td>1170</td>
<td>1400</td>
<td>LW / Declared range of values: ρ ≤ 1300 kg/m³.</td>
</tr>
<tr>
<td>Compressive strength [N/mm²]</td>
<td>2.40</td>
<td>2.25</td>
<td>CS I / Similar behaviour.</td>
</tr>
<tr>
<td>Adhesion [N/mm²] and fracture pattern FP: (A, B or C)</td>
<td>0.35</td>
<td>0.30</td>
<td>Similar behaviour.</td>
</tr>
<tr>
<td>Capillary water absorption (intended use in external elements)</td>
<td>4.89</td>
<td>≤ 4.0</td>
<td>W 0 / Similar behaviour.</td>
</tr>
<tr>
<td>Water vapour permeability coefficient (µ) (intended use in external elements)</td>
<td>5 - 10</td>
<td>&lt; 15</td>
<td>EN 1745 [3], Table A.12.</td>
</tr>
<tr>
<td>Thermal conductivity [W/(m.°C)]</td>
<td>0.29</td>
<td>0.61</td>
<td>Similar behavior when compared to LW mortars.</td>
</tr>
<tr>
<td>Latent heat [kJ/kg]</td>
<td>≈ 25</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Melting temperature [°C]</td>
<td>≈ 23</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Melting temperature range [°C] (90%)</td>
<td>23 - 25</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Specific heat (solid) [kJ/(kg K)]</td>
<td>1.0</td>
<td>1.0</td>
<td>EN 1745 [3], Table A.12.</td>
</tr>
</tbody>
</table>

Thus, we obtained a new composite material similar to a common mortar with changes in density and thermal conductivity without compromising mechanical performance. There were no differences between the values of the compressive strength or adhesion of the PCMM developed when compared to the reference mortar considered (REFM).

4 DURABILITY ASSESSMENT

According to EN 998 [1], mortar’s durability shall be assessed by testing adhesion and water permeability after freeze/thaw cycles. This way, it can be expected the essential
properties of mortars to be adhesion and water permeability and therefore its fatigue can be observed through the failure of these requirements.

When the mortars are transformed and adapted to support a larger heat storing capacity, as in the case of the new developed composite material presented in this paper, the PCMM, other requirements are introduced beyond the essential ones already mentioned.

The new plastering with PCM will be used and applied with the purpose of storing energy, contributing to the interior temperature stabilization of buildings (residences, offices and/or commercial spaces).

The durability of PCMM cannot be evaluated just by the adhesion and water permeability trials response. The enthalpy (or latent heat) and the melting temperature range become essential requirements as well. The fusion enthalpy relates to the heat storing capacity during the transition between solid to liquid state; if there is wear and/or enthalpy reduction caused by degradation agents, these phenomena should be studied and evaluated. The melting temperature, at which the solid to liquid transition occurs, is another essential requirement, as heat storing for indoor temperatures stabilization is only useful within the interior comfort temperature range (between 20°C to 25°C).

After identifying the essential requirements, it is necessary to know the degradation agents responsible for its failure. Considering the developed PCMM, Table 5 is presented with a summary of the ‘essential requirements’, ‘degradation agents’ and ‘minimum performance requirements’.

Table 5: Properties, degradation agents and performance requirements, for durability assessment of PCM plastering mortars.

<table>
<thead>
<tr>
<th>Essential requirements (Properties)</th>
<th>Degradation agents and requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive strength [N/mm²]</td>
<td>Freeze/Daw ≥ 0.5 N/mm² (EN 1015-11 [2])</td>
</tr>
<tr>
<td>Adhesion [N/mm²]</td>
<td>Weathering cycles ≥ 0.5 N/mm² (EN 1015-12 [2])</td>
</tr>
<tr>
<td>Capillary water absorption</td>
<td>Solar radiation W0 (not specified) (EN 1015-18 [2])</td>
</tr>
<tr>
<td>(intended use in external elements)</td>
<td>Chemical agents</td>
</tr>
<tr>
<td>Latent heat [kJ/kg]</td>
<td>Equal to initial declared range (DSC test)</td>
</tr>
<tr>
<td>Melting temperature range [°C]</td>
<td>≥ 90% of the initial declared value (DSC test)</td>
</tr>
</tbody>
</table>

The values shown in the table above (Table 5) represent a proposal for the assessment of the durability of a mortar with special heat storage capacity. Some of the mentioned tests are already in progress in order to assess and better characterize the PCMM developed.
5 CONCLUSIONS

The research reported in this work aimed at three main objectives: (i) to present the current specifications and test methods established by CEN in the ambit of external renders and internal plasters; (ii) to develop and characterize a new construction material, based in incorporating microencapsulated paraffins into cement based mortars, to be used in inner wall surfaces in order to increase thermal comfort inside buildings; (iii) and to suggest methods for the evaluation of essential requirements for the characterization of durability of new developed material.

Rendering and plastering mortars were split into types and categories according to properties and/or intended use. Requirements and characterization test methods were presented for general propose (GP) and lightweight (LW) mortars as in the normative documents.

A thermally enhanced mortar was achieved with 25% of PCM on the mass fraction, after a process of trial-and-error that encompassed several previous attempts that revealed cracking problems. The main thermal characteristics of the developed mortar are: enthalpy of 25kJ/kg in a melting range from 23ºC to 25ºC; and thermal conductivity of 0.3 W/(m.ºC). It should be stressed that the proposed PCM incorporation in mortars did not compromise the properties that are desirable for their application as plastering materials.

Bearing in mind that durability is one of the essential requirements of components or materials used in construction, the foundations for the evaluation of PCM-based plastering mortars, regarding its special characteristics, were established. For each PCMM’s properties, different degradation agents were identified and the performance requirements to be determined after the separate or combined action of those agents were defined.

1 REFERENCES

ANISOTROPIC EXPANSION OF ASR AFFECTED CONCRETE PRISM SPECIMENS

Bishnu P. Gautam (1) and Daman K. Panesar (1)

(1) Department of Civil Engineering, University of Toronto, Toronto, Ontario, Canada
b.gautam@mail.utoronto.ca; d.panesar@utoronto.ca

Abstract

Alkali aggregate reaction (AAR), the predominant type of which is alkali silica reaction (ASR), is a major deterioration problem in some concrete structures. Expansion is a common manifestation of ASR. However, the expansion behaviour has been reported to be influenced by the direction of casting of concrete. The scope of this paper is to present the expansions of reactive concrete prisms in the longitudinal direction (perpendicular to the casting direction) and the two transverse directions (one parallel to casting direction and the other perpendicular to the casting direction). Although the results revealed that the transverse expansion measurements were greater than the longitudinal expansion, no influence of the casting direction on the transverse expansion measurements was observed. In fact, the transverse expansions in both directions were identical indicating isotropic ASR expansion irrespective of the casting direction. The difference in the longitudinal and transverse expansions is attributed to the aspect ratio of the specimen and is supported with basic theoretical analysis.

1 INTRODUCTION

Alkali aggregate reaction (AAR) has been identified as one of the major deterioration problems in some concrete structures. The most prevalent type of AAR is the alkali silica reaction (ASR). After the first identification of ASR in 1940s by Stanton [1], the chemistry of the reaction has been extensively studied and adequately understood. ASR is a chemical reaction between the reactive silica available in certain types of aggregate and the alkalis in the concrete’s pore fluid. The consequences are the initiation of cracks in the reactive aggregates, crack propagation to the paste matrix, volumetric expansion of the concrete, stresses due to expansive pressure, and degradation of mechanical properties. The damage caused by the reaction depends on the rate of formation of the reaction product, its volume concentration within concrete, and the stresses concrete is subjected to [2–4].

One recognized complexity in understanding the effect of ASR on concrete structures is its apparent anisotropic expansion in the absence of any directional restraints such as reinforcement or stress. A study by Smaoui et al. [5] reported that ASR expansion of cylinders and prisms of various orientations, is highly anisotropic with respect to the direction.
of casting of concrete (Figure 1 a,b,c). Several prisms (cast horizontally and cast vertically), cylinders and concrete blocks were measured for expansion in different directions and the variations in expansion were interpreted in context with the casting direction [5]. The study concluded that the expansion was always significantly greater when measured in the direction parallel to the casting direction. While the study did not investigate how casting direction affected the expansion, a possible explanation was given that the anisotropy is associated with the accumulation of water under the aggregate particles.

Multon et al. [6] reported on the effect of casting direction by measuring the expansion along the length of concrete cylinders and concrete prisms. It should also be noted that Multon et al. [6] did not compare the effect of casting direction on expansion of prisms vs. prisms or cylinders vs. cylinders, rather they compared cylinders vs. prisms. For the cylinders, they measured the expansion parallel to the casting direction (Figure 1a). For the prisms, they measured the expansion perpendicular to the casting direction (Figure 1c). The results revealed that the expansion of the cylinders measured parallel to the casting direction was greater than the expansion of the prisms measured perpendicular to the casting direction.

Dunant and Bentz [7] attempted to clarify the possibly anisotropic expansion by combining an experimental study with a numerical simulation of the micro-mechanical effects of ASR. However, they only considered scenarios perpendicular to the casting direction.

The aim of this study is to investigate the effect of casting direction on the variability of expansion in order to identify the dependency, if any. Expansion of concrete prisms, as shown in Figure 1c, in the longitudinal direction and in the two transverse directions is reported. The longitudinal expansion measurement of the prisms is perpendicular to the casting direction. The transverse expansion measurements perpendicular to the casting direction are measured on two concrete surfaces that have been cast against the formwork and so are referred to as the ‘form-form’ transverse measurement. The transverse expansion measurements parallel to the casting direction are measured on one concrete surface that has been hand finished and the other surface cast against the formwork and so is referred to as the ‘finish-form’ transverse measurement.
2 MATERIALS AND METHODS

The specimens considered in this study are four concrete prisms of dimensions, 285 mm long and a square cross-section of 75 mm. They were cast and cured in accordance with the concrete prism test, ASTM C1293 [8]. Compaction was conducted by rodding. One day after casting, the prisms were demolded and the initial length reading was taken. They were then stored in a hermetic plastic pail inside a 38 °C chamber. The bottom of the pail was filled with one litre of de-ionized water. Plastic supports were used to raise the bottom of prism by 2 cm above the water surface. The side of the bucket was covered with absorbent paper to facilitate water circulation by wick action. Subsequent to the initial measurement, the prisms were measured for expansion at 7, 28, 56, 91, 182, 273, and 365 days. The pail was taken outside the 38 °C chamber for acclimatization to room temperature before taking the measurements.

2.1 Mix design

The concrete mix design was based on ASTM C1293 [8], and is shown in Table 1. High alkali general use cement was used with total alkali content of 1 % Na₂O equivalent by weight of cement. The alkali content was boosted to 1.25% Na₂O equivalent by adding NaOH pellets. The water to cementitious material ratio was 0.44.

Table 1: Volumetric mix design of concrete (per m³)

<table>
<thead>
<tr>
<th>Material</th>
<th>Mass (kg)</th>
<th>Specific Gravity</th>
<th>Volume (L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total</td>
<td>2440</td>
<td>2.44</td>
<td>1000</td>
</tr>
<tr>
<td>Cement</td>
<td>420</td>
<td>3.15</td>
<td>133</td>
</tr>
<tr>
<td>Coarse aggregate</td>
<td>1113</td>
<td>2.69</td>
<td>414</td>
</tr>
<tr>
<td>Water</td>
<td>185</td>
<td>1.00</td>
<td>185</td>
</tr>
<tr>
<td>Alkali pellet (NaOH)</td>
<td>1.3</td>
<td>1.40</td>
<td>0.9</td>
</tr>
<tr>
<td>Sand</td>
<td>721</td>
<td>2.70</td>
<td>267</td>
</tr>
</tbody>
</table>

Note: Mass of sand and coarse aggregate shown are for saturated surface dry condition. Moisture correction was made after determining the moisture content prior to mixing.

2.2 Coarse aggregate and sand

The coarse aggregate used was Spratt aggregate from Stittsville near Ottawa, Ontario, Canada generously supplied by the Ontario Ministry of Transportation. The Spratt limestone containing reactive silica minerals is classified as highly reactive aggregate, and has been widely studied in Canada and abroad [9]. Spratt is a crushed aggregate from a quarry, and thus, has a more definite geological formation (in comparison to river gravel) and can been regarded as a standard reactive aggregate for ASR studies. Sand used in this study was obtained from Orillia, Ontario, Canada and kindly supplied by Lafarge Canada Inc.. The sand was evaluated to be non-reactive according to the accelerated mortar bar test as per ASTM C1260 [10].
2.3 **Expansion measurement in the longitudinal direction**

The longitudinal expansion was measured as per ASTM C1293 [8] using a comparator against the steel studs which are fixed on both ends of the prism (Figure 2). The comparator has a resolution of 1 µm, and was calibrated against a reference invar bar. The expansion at a given age was calculated as the difference between the comparator reading at the given age and the initial comparator reading taken at the day of demolding.

![Figure 2: Comparator measurement for expansion in the longitudinal direction](image)

2.4 **Expansion measurement in the transverse directions**

Figure 3a illustrates a digital micrometer and the orientation of a prism during transverse expansion measurement. The digital micrometer with a resolution of 1 µm was calibrated against a micrometer standard before taking the prism measurements. Transverse expansion measurements were taken against the small brass targets, which were epoxy-glued on the sides of the prisms as shown in Figure 3a. Two targets were glued in each of the four faces of a prism; thus, each prism had two measurements in each transverse direction. Steel guides were prepared as shown in Figure b to assist in accurately fixing the targets.

![Figure 3: a) Micrometer; b) steel guides for affixing the brass targets](image)

3 **RESULTS**

The mean expansion measurements ± one standard deviation in the longitudinal, transverse (finish-form), and transverse (form-form), of the prisms are plotted in Figure 4. Each data point for the longitudinal expansion is the mean of four measurements whereas each data point for the transverse expansion is the mean of two measurements.
point for the transverse expansions is the mean of eight measurements, including two measurements in each prism. The longitudinal expansion measurements were taken in the direction perpendicular to the casting direction (Figure 1c). As previously mentioned, two transverse measurements were taken and are referred to as ‘form-form’ and ‘finish-form’. The ‘form-form’ transverse measurement was taken perpendicular to the casting direction on two formed concrete surfaces. The ‘finish-form’ transverse measurement was taken parallel to the casting direction on one finished and the other formed surface.

![Graph showing average longitudinal and transverse expansions of the concrete prisms.]

Figure 4: Average longitudinal and transverse expansions of the concrete prisms

4 DISCUSSION

4.1 Experimental analysis of the anisotropic expansion

Table 2 compares the expansion results with the results obtained from a multi-laboratory study for the concrete prisms made with Spratt aggregate as reported by Fournier et al. [11]. The corresponding coefficients of variation (COV) are also presented. A perfect agreement between the two sets of data having 99 % correlation could be regarded as a corroboration of selecting Spratt as the standard reactive aggregate for this study.

Table 2: Comparison of longitudinal expansion with results from a previous study

<table>
<thead>
<tr>
<th>Age (days)</th>
<th>Multi-laboratory results (Fournier et al. [11])</th>
<th>Present study (Gautam and Panesar)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average expansion</td>
<td>COV</td>
</tr>
<tr>
<td>91</td>
<td>0.097 %</td>
<td>25 %</td>
</tr>
<tr>
<td>182</td>
<td>0.180 %</td>
<td>10 %</td>
</tr>
<tr>
<td>273</td>
<td>0.204 %</td>
<td>8 %</td>
</tr>
<tr>
<td>365</td>
<td>0.229 %</td>
<td>2 %</td>
</tr>
</tbody>
</table>
The longitudinal expansion of the prisms was 0.215 % in one year. For reference it should be noted that the limiting expansion that is used to confirm an aggregate as non-reactive is 0.04 % in one year in concrete prism test as per ASTM C1293 [8]. The expansions in the transverse directions were observed higher than in the longitudinal direction as shown in Figure 4 and Table 3. The ratio of expansion in the transverse directions to the longitudinal direction was 3.72 at 28 days, and decreased to 1.31 by 365 days. The expansions in the two transverse directions were almost identical even though the transverse expansion of the finish-form surfaces was observed to be marginally greater than that of form-form surfaces at all ages.

Table 3: Expansion ratios and coefficient of variation in the transverse expansions

<table>
<thead>
<tr>
<th>Age (days)</th>
<th>Ratio of expansions</th>
<th>COV (of eight measurements)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average transverse</td>
<td>Transverse</td>
</tr>
<tr>
<td></td>
<td>to longitudinal</td>
<td>(finish-form)</td>
</tr>
<tr>
<td></td>
<td>(finish-form) to</td>
<td>(finish-form)</td>
</tr>
<tr>
<td></td>
<td>(form-form)</td>
<td>(form-form)</td>
</tr>
<tr>
<td>28</td>
<td>3.72</td>
<td>1.33</td>
</tr>
<tr>
<td>56</td>
<td>2.09</td>
<td>1.09</td>
</tr>
<tr>
<td>91</td>
<td>1.66</td>
<td>1.04</td>
</tr>
<tr>
<td>182</td>
<td>1.45</td>
<td>1.04</td>
</tr>
<tr>
<td>273</td>
<td>1.23</td>
<td>1.05</td>
</tr>
<tr>
<td>365</td>
<td>1.31</td>
<td>1.03</td>
</tr>
</tbody>
</table>

In comparison to the COV (max. 5 %) for the longitudinal expansion measurements reported in Table 2, the COV corresponding to the transverse measurements was markedly larger (14 - 55 %) as shown in Table 3. Smaoui et al.[5] also reported relatively larger COV for transverse expansions compared to the longitudinal expansion measurements. This may be partly attributed to the fact that transverse measurements correspond to a relatively smaller section of concrete (75 mm in this study) being measured compared to 285 mm for the longitudinal expansion.

4.2 Theoretical analysis of the anisotropic expansion

The relatively greater expansion in the transverse directions than in the longitudinal direction could be attributed to the aspect ratio of the specimen. This section presents a theoretical analysis of the likely scenario of stresses in the concrete prisms as illustrated by Blight and Alexander [12].

Consider a prism with external dimensions \( L \times B \times H \) as shown in Figure 5. Assume a uniform cover concrete of thickness ‘c’, the core of concrete will be \( l \times b \times h \), where \( l=L-2c \) etc.. Initially, the cover concrete will suffer from ASR expansion first, due to moisture availability and the core is assumed to be relatively less affected. As the reaction front penetrates to the concrete core, it expands which subjects (i) the cover concrete to tensile stress \( f_t \) and, (ii) the core to compressive stress \( f_c \). At equilibrium, the compressive force in the core should balance the tensile force in the cover concrete as shown in Figure 5a and:

\[
\text{Longitudinal (perpend. to cast dir.)} \quad (BH - bh)f_{tL} = bhf_{cL} \quad (1)
\]
The equilibrium equation for the transverse stresses parallel to casting direction (Figure 5b) is shown in equation (2) and perpendicular to the casting direction in equation (3):

\[
\text{Transverse (parallel to cast dir.) } \quad (BL - b) f_{ct} = bl f_{cr}
\]

\[
\text{Transverse (perpen. to cast dir.) } \quad (HL - h) f_{ct} = bl f_{cr}
\]

If a prism is square, \( B = H \) and \( b = h \). The transverse stresses in the direction perpendicular and parallel to the casting direction will be identical. For a prism with \( L = 285 \) mm, and \( B=H=75 \) mm, Table 4 presents the ratio of tensile-to-compressive stress calculated for different values of \( c \) since the most representative value for this study is unknown. In any case, the ratio of tensile-to-compressive stress is always larger in the transverse directions than in the longitudinal direction as shown in Table 4. Assuming a relatively uniform expansive pressure in the core, this indicates that the prism is likely to have more longitudinal cracks (perpendicular to the larger tensile stress) than transverse cracks. Similarly, more expansion can be expected in the transverse directions than in the longitudinal direction.

This theoretical analysis explains and supports the observations that (i) expansions in the two transverse directions of square prism specimens were similar, (ii) the transverse expansions were greater than the longitudinal expansion, and (iii) cracks in the prisms reported by Smaoui et al. [5] were predominantly longitudinal.

![Figure 5: Stress state in a prism specimen during expansion; a) longitudinal; b) transverse](image)

**Table 4: Ratio of tensile-to-compressive stress for a square prism (285 x75 x 75 mm)**

<table>
<thead>
<tr>
<th>Cover c (mm)</th>
<th>Length l (mm)</th>
<th>Section b &amp; h (mm)</th>
<th>Ratio of tensile-to-compressive stress</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Longitudinal ( (f_{ct}/f_{cr}) )</td>
</tr>
<tr>
<td>5</td>
<td>275</td>
<td>65</td>
<td>3.0</td>
</tr>
<tr>
<td>10</td>
<td>265</td>
<td>55</td>
<td>1.2</td>
</tr>
<tr>
<td>15</td>
<td>255</td>
<td>45</td>
<td>0.6</td>
</tr>
</tbody>
</table>

5 CONCLUSIONS AND FUTURE WORK

This study measured the expansion of the ASR concrete prisms in the longitudinal and the two transverse directions.

- The expansions were statistically significantly similar in both the transverse directions (parallel and perpendicular to the casting direction) and consequently, no significance of anisotropy related to the casting direction was observed in this study.
The expansion in the transverse directions was markedly greater (3.72 times) than in the longitudinal direction. With age however, the difference between longitudinal and transverse expansions reduced whereby at one year the transverse expansion was only 1.31 times that of the longitudinal expansion. Aspect ratio of the specimen is believed to have contributed to the variation.

Based on the outcome of this study, a cube specimen would provide identical expansions in the three mutually perpendicular directions. A new experiment has been launched which involves cube specimens and is expected to be free from any differential effect of the aspect ratio of a specimen.

REFERENCES

THE INFLUENCE OF CRACK WIDTH ON THE DURABILITY OF REINFORCED CONCRETE STRUCTURES

A. Blagojević (1), D. A. Koleva (2), J. C. Walraven (1)

Delft University of Technology, Faculty of Civil Engineering and Geosciences,
(1) Department of Structural Engineering - Concrete Structures, Delft – a.blagojevic@tudelft.nl; j.c.walraven@tudelft.nl
(2) Department of Materials and Environment, Delft – d.a.koleva@tudelft.nl

Abstract

According to structural design codes, including recent ones, crack width within serviceability limits plays a dominant role, hence contributes to structures-related costs. This research intends to shed more light on the role of crack width in relation to durability. An investigation is carried out, where crack width, crack frequency, concrete cover and type of loading are the dominant variables under study. The test series consists of reinforced concrete beams (1500×100×150 mm), exposed to wetting and drying cycles in order to simulate aggressive environment i.e. weekly, a 2 days ponding (3.5 % NaCl solution) and 5 days drying cycles are altered. Longitudinal cracks lead to a considerable increase of the speed of corrosion. Crack frequency is recognised to play a role with regard to reinforcement corrosion. Crack width, however, could be a significant durability-related parameter as well, which is as adopted in most codes of practice. Indeed, if crack width is an important factor, it seems to be more appropriate to use the crack width at maximum service load, rather than at the frequent load or the quasi-permanent load. Justification of this latter approach is within the goals of this study, aiming to bring more accuracy in the current codes of practice.

1 INTRODUCTION

Durability of concrete structures is one of the most important issues with respect to the design of new structures and/or extending service life of existing ones. Several deterioration mechanisms (e.g. chloride- or carbonation-induced corrosion, sulphate attack, alkali-silica reaction) can significantly shorten the service life of reinforced concrete. Understanding the most dominant deterioration mechanisms, occurring during the service life of a structure is very important. Chloride-induced corrosion of the steel reinforcement is one of the major threats to durability, especially for structures in marine environment and those exposed to de-icing salts. In these environments chloride ions penetrate into the concrete bulk matrix and
reach the vicinity of the steel reinforcement. At certain critical chloride concentration, steel corrosion is initiated (reported thresholds vary between 0.2 and 2.0 wt.% per cement weight). In marine environment the splash and tidal zones are those with highest corrosion risk where alternating wetting and drying cycles ease chloride penetration. For structures exposed to de-icing salts, such as bridge decks, car parks, etc., the corrosion risk is mainly caused by the very high concentrations of chloride ions in the water after ice melting. Similarly, the result is steel corrosion initiation and further compromised integrity and service life.

2 RESEARCH SIGNIFICANCE

In codes for structural concrete the maximum crack width under a defined load level is assumed to be a major criterion for durability. In Eurocode EN 1992-1-1 [1] the maximum crack width for reinforced concrete structures is determined under the quasi-permanent load combination, while for prestressed concrete structures - under the frequent load combination. The EN 1992-1-1 is flexible with regard the choice of authorities in each country to determine the loading level and the maximum allowable crack width for the relevant environmental class. No indication is given for the most appropriate choice. The crack width criteria in the various codes show substantial differences. For example, Eurocode 2 recommends a value of 0.3 mm for the maximum allowable crack width in reinforced concrete members exposed to aggressive environment under quasi-permanent load combination, whereas ACI Committee 224 [2] prescribes 0.15 mm under the design service load. An aspect that should be considered as well is that the crack width is measured at the surface of the structure, whereas it decreases into the direction of the reinforcement. This is not logic since the crack width at the reinforcement should be a governing parameter, although this cannot easily be measured. The present guidelines can even lead to erroneous conclusions, since if the concrete cover is increased to account for increased durability, the surface crack width will also increase, which erroneously suggests that durability is decreased. In other words, increasing the concrete cover requires more reinforcement in order to limit the surface crack width to its allowable value. Only in a small number of codes this anomaly is avoided by defining the maximum allowable surface crack width as a function of the concrete cover, like the former Dutch code NEN 6720 [3] and the fib Model Code for Concrete Structures 2010 [4].

A more important and logic question is whether the crack width matters at all for durability. Already in 1989, acknowledging fundamentals, the CEB Bulletin 182 [3] states that for the corrosion process to occur and proceed, a separation of anodic and cathodic areas is necessary. In a simplified way, the reinforcing steel in the cracked region would generally act as the anodic area, whereas the cathodic process will mostly occur on the steel surface within the un-cracked areas or “between the cracks”. In the case of crack widths up to 0.4 mm, “self-healing”, resulting from calcium-bearing compounds, dirt- and rust deposits in the cracks is frequently observed. Hence, further penetration of aggressive substances, water and oxygen through these cracks are suppressed and the rate of the corrosion process decreased. On the other hand, chloride-induced corrosion is an autocatalytic process with the following sequence: once chloride ions are present at the steel surface, localised corrosion initiates; corrosion products form above and in the vicinity of the area of corrosion initiation; this limits further chloride ions penetration in this same location. However, chloride ions remain trapped within the “pits” and the corrosion process proceeds in depth of the reinforcement. Within this process, Fe^{2+}, Cl^{-} and H^{+} ions are present in the pits and pH can locally drop < 5, resulting in
fast corrosion propagation. Therefore, the mechanism of chloride-induced corrosion, especially in alkaline environment, is mainly dependent on the continuous ingress of aggressive substances and hence crack width, frequency and type are largely contributing factors to corrosion-induced degradation of civil structures.

In the Model Code for Service Life Design [6] the role of crack width is even totally ignored, stating that cracks are not relevant. Nevertheless, even in the most recent codes, tables with crack width limits are given, indicating that obviously doubts about the role of crack width for structural performance still exist. These uncertainties mainly refer to:

- Can we confidently state that steel corrosion is significantly minimised due to self-healing, dirt of rust deposits in various cracks? Concrete structures are often subjected to variable loads, which hampers self-healing. Hence, the presence and development of cracks can not be ignored, even if self-healing is accounted for.
- If a crack width is large, this means that at the reinforcement level, considerable micro-cracking occurs, enabling additional access of oxygen, water and chloride ions to areas away from the main crack. In the case of close-by positioned principle cracks, the result will be the formation of a scaffold of micro-cracks, extending along substantial areas between the principle cracks. Logically, these microstructural changes will contribute to a significant extent for ions and water penetration i.e. will determine the electrochemical phenomena on the steel surface. Therefore, their existence cannot be ignored.

Aiming at gaining more insight into the above-raised questions, an experimental program was designed in order to generate knowledge and contribution to existing codes and practice.

3 EXPERIMENTAL PROGRAM

Taking into consideration durability provisions for chloride-induced corrosion of reinforced concrete structures, the following issues are prescribed by codes: exposure class, concrete mixture, maximum allowable crack width and minimum concrete cover. Exposure classes for concrete structures are for instance defined in Eurocode 2 [1]. The experimental program designed for this work considers severe (aggressive) environment via alternating wetting and drying cycles. Informative examples of this exposure class (XD3) are car park slabs, pavements, parts of bridges exposed to spray containing chlorides etc. Since a research project generally has a limited time-frame, simulation of environmental conditions generally includes accelerated testing, but can also be designed as realistic as possible. In this project, cracked reinforced concrete beams were exposed to alternating wetting and drying cycles i.e. simulation of aggressive environment was achieved through a weekly 2 days ponding cycle (3.5% NaCl solution), followed by a 5 days drying phase.

3.1. Concrete mixture and casting procedure

The research project (this work being part of it) aims at investigating the influence of cracks on chloride-induced corrosion during a limited period of time. Consequently, a relatively low value of the concrete strength class was aimed at for the concrete mixture, C20/25 (w/c ratio 0.60 and cement content 260 kg/m3). Concrete composition is not subject to investigation, therefore only one concrete mixture was employed for all specimens.

Reinforced concrete beams 1500×100×150 mm were cast with one ribbed bar, embedded at the desired position (schematic presentation in Fig.1). High yield reinforcing steel B500A was used, diameter 12 mm. The bars were thoroughly cleaned and degreased prior to casting.
An internal pseudo-reference electrode (platinized titanium) was fixed next to the reinforcing bar using strips. The part of the beam, which was later exposed to a chloride solution, is situated at the bottom in the mould. The concrete was vibrated in three layers. Titanium mesh was embedded at the opposite side, at 15 mm from the cover, in order to act as a counter electrode for corrosion measurements. Several cubes were cast for each batch to control the compressive strength. After casting, all specimens were covered with plastic sheets for 72 hours under laboratory conditions. Next, all specimens were cured in climate-controlled room (20±2°C and 95±5% RH) until 28 days of age, when cubes were tested to check compressive strength, while the reinforced concrete beams were exposed to air drying under lab conditions for an additional 7 days. The 28-day age compressive strength was 31 ±2MPa.

3.2. Concrete cover

Three different concrete covers were applied: 20 mm, 30 mm and 40 mm. Larger concrete cover implies larger barrier for chloride ions penetration, consequently, a thicker concrete cover provides better corrosion protection for the steel reinforcement. On the other hand, a larger concrete cover leads to higher surface crack width.

![Figure 1: Experimental set-up][7]

Four-point bending was applied in order to induce predefined crack widths in the specimens. Steel beams were required for the purpose to place supports (steel rods) on them and to use a downer bracket in interaction with threaded rods, bolts and upper bracket to apply force on the beam. The experimental set-up is shown in figure 1.

![Figure 2: Overview of test series in Stevin Lab II][8]
The different series of mean crack widths and number of cracks were tested in combination with concrete covers and type of loading – the experimental schedule is presented in Table 1. The crack widths were measured by a digital microscope and the average value was compared with the desired target value. In order to control the number of cracks in some series undesired cracks were sealed by epoxy. The containers for the chloride contaminated water were made of PVC and placed on the top of the beam. The cracks in depth along both sides of the beams were sealed by silicone. An overview of the test series is shown in figure 2.

Table 1: Series of variable combinations

<table>
<thead>
<tr>
<th>Series</th>
<th>Concrete cover (mm)</th>
<th>Mean crack width (mm)</th>
<th>Number of cracks</th>
<th>Type of loading</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>C=20</td>
<td>0</td>
<td>0</td>
<td>Unloaded</td>
</tr>
<tr>
<td>2B</td>
<td>C=20</td>
<td>0.15+longitudinal</td>
<td>9</td>
<td>Sustained load</td>
</tr>
<tr>
<td>3A</td>
<td>C=20</td>
<td>0.15</td>
<td>9</td>
<td>Sustained load</td>
</tr>
<tr>
<td>4B</td>
<td>C=20</td>
<td>0.15</td>
<td>6</td>
<td>Sustained load</td>
</tr>
<tr>
<td>5A</td>
<td>C=30</td>
<td>0</td>
<td>0</td>
<td>Unloaded</td>
</tr>
<tr>
<td>6A</td>
<td>C=30</td>
<td>0.15</td>
<td>8</td>
<td>Sustained load</td>
</tr>
<tr>
<td>7A</td>
<td>C=30</td>
<td>0.10-0.20</td>
<td>8</td>
<td>Variable load</td>
</tr>
<tr>
<td>11A</td>
<td>C=40</td>
<td>0</td>
<td>0</td>
<td>Unloaded</td>
</tr>
<tr>
<td>14B</td>
<td>C=40</td>
<td>0.15-0.25</td>
<td>6</td>
<td>Variable load</td>
</tr>
<tr>
<td>15A</td>
<td>C=40</td>
<td>0.15</td>
<td>6</td>
<td>Sustained load</td>
</tr>
<tr>
<td>15B</td>
<td>C=40</td>
<td>0.25</td>
<td>6</td>
<td>Sustained load</td>
</tr>
<tr>
<td>16A</td>
<td>C=40</td>
<td>0.2</td>
<td>6</td>
<td>Sustained load</td>
</tr>
</tbody>
</table>

3.3. Measurement of steel corrosion

The half-cell potential (HCP) is a generally employed method, indicating active or passive state of the steel reinforcement. According standards and guidelines, potential values more anodic than -270 mV (vs SCE for example) account for passive steel. Different reference electrodes can be used, including external and embedded ones. In this experiment platinized titanium (Pt/Ti) and silver-silver chloride (Ag/AgCl) were applied as an internal and an external reference electrode respectively. Potentiostat Autolab (Metrohm BV) was used for all corrosion tests. Linear polarization resistance (LPR) was performed in the range of ± 20mV around open circuit potential, scan rate of 0.1 mV/s (a common three-electrode arrangement was used, where the steel bar was the working electrode and the Ti mesh the counter electrode). For comparative purposes, LPR was conducted using both embedded (Pt/Ti) and external (Ag/AgCl) reference electrodes. Linear regression was applied to derive polarization resistance ($R_p$) in eq.1, whereas the Stern-Geary equation (eq. 2) was employed to calculate the corrosion current ($I_{corr}$). The corrosion current density ($i_{corr}$) was calculated through considerations of geometrical constants (eq. 3 and 4).

\[
R_p = \frac{\Delta E}{\Delta I} \quad (1)
\]

\[
I_{corr} = \frac{B}{R_p} \quad (2)
\]

\[
i_{corr} = \frac{I_{corr}}{A} \quad (3)
\]

\[
A = \pi D l = 550 \text{ cm}^2 \quad (4)
\]
where, \( B \) is 26 mV for active and 52 mV for passive state in reinforced concrete \(^8\), \( D \) is diameter of the steel rebar 12 mm and \( l \) is a length of the polarized steel area of 146 cm.

4 TEST RESULTS AND DISCUSSION

4.1. The influence of longitudinal cracks

Beam 1A was not loaded and it was uncracked as well as beams 5A and 11A. Beam 2B was loaded until reaching the intended maximum crack width. Flexural cracks were created along with few longitudinal ones. Next, the load was decreased so that a mean flexural crack width of 0.15 mm was reached. The cracks in beam 3A were created by applying the load until the mean crack width was 0.15 mm. Nine flexural cracks were created in beam 3A and beam 2B. Longitudinal cracks have not been created in beam 3A. In beam 4B after loading a few cracks have been deactivated by epoxy i.e. 6 active cracks were left. The corrosion tests showed a very good reproducibility (99%) when both reference electrodes were used (figs. 3 and 4). In the uncracked beam, the reinforcement is still passive. The highest corrosion current was recorded in beam 2B, fig. 3. Corrosion products are visible at the top of the cover after 26 weeks of exposure. In all aforementioned cracked beams, corrosion initiation was very fast i.e. during the first ponding cycle of 2 days. The corrosion process further slowed down in beam 4B. The higher number of cracks in beam 3A led to slightly higher corrosion current, compared to beam 4B. The corrosion currents are still increasing in beam 2B due to easy access of chlorides, moisture and oxygen to a large steel surface area as a result of the longitudinal crack. So, longitudinal cracks can be even more dangerous than flexural cracks with regard to corrosion of steel reinforcement.

![Graph](image1.png)

Figure 3: Evolution of \( i_{corr} \) - beams with 20 mm concrete cover after 26 weeks of exposure

4.2. The influence of concrete cover

In order to induce a mean crack width of 0.15 mm in beams 3A, 6A and 15A the number of cracks without any crack sealing was 9, 8 and 6 respectively. Corrosion rate measurements on beams with 0.15 mm mean crack width but different concrete cover and number of cracks are shown in figure 4. Beam 15A (concrete cover 40 mm, 6 cracks) has the same mean surface crack width, 0.15 mm, as beam 3A (concrete cover 20 mm, 9 cracks). The corrosion current density for beam 3A is significantly higher than that in 15A. The corrosion activity for beam 6A is in between, which is logic considering the number of cracks and the concrete cover. In this case according to the theory of crack formation a larger concrete cover leads to a smaller number of cracks and as such a lower corrosion activity. In beams 15A (6 cracks)
and 6A (8 cracks) the corrosion current density seems to stabilize after about 20 weeks. In beam 3A, with 9 cracks, stabilizations has not been reached after 26 weeks.

![Graph of corrosion current density vs. time](image)

**Figure 4:** Evolution of $i_{corr}$ - beams with 0.15 mm mean crack width but different concrete cover and number of cracks

### 4.3. The influence of type of loading

Beams 6A and 7A contain equal number of 8 cracks and the same concrete cover of 30 mm. Beam 6A is under sustained constant load, the mean crack width is 0.15 mm. The mean crack width in beam 7A is 0.10 mm. Beam 7A was reloaded once a week in order to reach a mean crack width of 0.20 mm for 18 hours (6 h during wetting and 12 h during drying cycles). After reloading, beam 7A was relaxed to the previous loading where the mean crack width was again 0.10 mm for the rest of the week (150 h). Taking into account the duration of the loaded and the reloaded state of beam 7A, it can be concluded that the mean crack width is 0.10 mm for 88% of the exposure time and 0.20 mm for 12% only of the exposure time. Consequently, the mean crack width under the quasi-permanent load combination would be slightly higher than 0.10 mm, but certainly lower than 0.15 mm. It can be noted, figure 5 (left), that higher corrosion current was recorded in the case of beam 7A, compared to beam 6A. Although the mean crack width is lower in beam 7A, the corrosion activity is higher due to the variable load and additional micro-cracks, formed under the maximum variable load. These additional micro-cracks do not close completely after reducing the load level. Obviously, the maximum variable load would largely determine the steel corrosion state. Hence, with regard the role of crack width in durability aspects, if any, the maximum service load should be accounted for and not the quasi-permanent or the frequent load. Should be noted that in the case of variable load (Beam 7A) no stabilization of the corrosion current density was reached after 26 weeks.

The corrosion activity for four beams with the same concrete cover and number of cracks, but different crack widths is shown in fig. 5 (right). Beams 15A, 16A and 15B were subjected to a sustained constant load where the mean crack widths were 0.15 mm, 0.20 mm and 0.25 mm respectively. It can be seen that the wider mean crack width leads to higher corrosion currents under a constant load, constant concrete cover and constant number of cracks. Beam 14B was subjected to an alternating load for the same duration of reloading as applied for the aforementioned beam 7A. However, the mean crack width in this beam was 0.15 mm; under reloading - 0.25 mm. It can be seen that the corrosion activity in beam 14B was much higher than that in beam 15A, where the mean crack width was constantly 0.15 mm. The corrosion activity in beam 14B was even slightly higher than that in beam 15B, subjected to a constant load, where the mean crack width was always 0.25 mm. It can be concluded that alternating
opening and closing of cracks exerts larger difference in corrosion activity, compared to cracks with a constant width. Obviously crack opening and closing under an alternating load leads to increased chloride ingress and a higher corrosion rate. This occurs due to the fact that a lot of microcracks form and the width of the bending cracks varies, preventing the previously discussed self-healing and crack blocking.

![Graph showing corrosion activity](image)

**Figure 5: Evolution of $i_{corr}$ - beams with the same concrete cover and number of cracks but the different type of loading and crack widths**

5 CONCLUSIONS

The following conclusions can be drawn based on the preliminary experimental results:

- In the presence of longitudinal cracks the corrosion process progresses faster than in the case of pure bending cracks. Therefore, longitudinal cracks can be much more dangerous than pure bending cracks for the service life of civil structures.
- The crack frequency (number of cracks per unit-length) influences corrosion activity. For the same mean crack width a larger concrete cover leads to a smaller number of cracks and as such a lower current density in the early deterioration stage.
- In the early corrosion stage the maximum load level governs the corrosion rate.
- In the early corrosion stage load variation leads to an increase of the corrosion rate.
- In the case of alternating loads, longitudinal cracks, or a high crack frequency even after 26 weeks, no stabilization of the corrosion current density was observed.

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TIME-DEPENDENT PASSIVE INFRARED THERMOGRAPHIC INSPECTION OF FACADES

Ecem Edis(1), Inês Flores-Colen(2) and Jorge de Brito(3)

(1) Istanbul Technical University, Faculty of Architecture, Istanbul, Turkey, ecem@itu.edu.tr
(2) DECivil-IST, Technical University of Lisbon, Lisbon, Portugal, ines@civil.ist.utl.pt
(3) DECivil-IST, Technical University of Lisbon, Lisbon, Portugal, jb@civil.ist.utl.pt

Abstract
In passive thermography, where natural boundary conditions are used to trigger surface temperature variations, thermographic data is usually analysed qualitatively, and thus accuracy issues may arise, and defects cannot be characterised exactly. On the contrary, in active thermography applications where artificial heat sources are used, quantitative analysis methods are performed and defects can be accurately characterised. However, in building thermography, especially in the case of exterior inspections of middle to high-rise buildings, passive thermography is generally preferred.

In current thermography literature, attempts to use quantitative methods in passive thermography have been made to enhance thermograms and/or increase the results accuracy. Time-dependent data are analysed for this purpose. In this respect, a ceramic building facade with pre-identified delamination problems was inspected by passive thermography, and time-dependent thermal behaviour of the defective areas was analysed by simple image subtraction, principal component analysis and non-negative matrix factorization, as different quantitative methods, for evaluating the potential of time-dependent analysis and comparing different analysis approaches.

In the paper, following an introduction, brief information on quantitative approaches used in the analyses is given as a theoretical background. Information on the aforementioned inspection and results obtained by different quantitative approaches are explained afterwards. Finally, the potential of these approaches in detecting the delamination problem is discussed and compared.

Keywords: Passive infrared thermography, adhered ceramic cladding, delamination, time-dependent analysis.
1 INTRODUCTION

In infrared thermography (IRT), ‘passive’ and ‘active’ approaches define how observable surface temperature variations are provided. In the passive approach, natural boundary conditions are used [1], and thermographic data is usually analysed qualitatively. In the active approach, artificial heat sources are used [1], and quantitative analysis of the thermography data is performed. Attempts to use computational methods of quantitative analysis in the passive IRT applications are also present to enhance thermograms and/or increase the results’ accuracy.

In adhered ceramic claddings that are widely preferred in some countries such as Portugal, delamination between the tile and the substrate, and the resulting detachment of the tile is one of the durability problems observed [2], and it also creates safety risks. Therefore, detection of delamination before any detachment occurs is important to take necessary preventive (maintenance) measures and IRT provides a non-destructive approach. In building IRT, especially for exterior inspections of middle to high-rise buildings, passive approach is generally preferred, but accuracy issues may also arise due to its qualitative nature. The use of methods of quantitative analysis in passive building thermography applications, such as time-dependent inspection/analysis, is an approach that may increase its accuracy.

A ceramic building facade in Lisbon (Portugal), with pre-identified delamination problems was therefore inspected by passive IRT for nine hours with half-an-hour intervals. The objective was to evaluate the potential and accuracy of time-dependent analysis in detecting delamination in adhered ceramic claddings, and also to comparatively evaluate different computational methods. For this purpose, the time-dependent thermal behaviour of the defective areas was analysed by the computational methods of ‘simple image subtraction (SIS)’, ‘principal component analysis (PCA)’ and ‘non-negative matrix factorization (NMF)’.

In the paper, brief information on the computational methods used in the study for time-dependent analysis is given initially as a theoretical background. Information on the in situ inspection and results obtained by different methods are explained afterwards. Finally, the potential of these methods in detecting the delamination problem is discussed and compared.

2 THEORETICAL BACKGROUND

In IRT, some computational methods such as ‘thermal contrast’ may be used either for qualitative or quantitative purposes (i.e. defect visualisation or characterisation respectively) [3]. In defect characterisation, the use of solar irradiation for stimulating observable surface temperature variations (i.e. a passive approach), requires additional research [4], especially on test walls that have artificial defects with known characteristics, to obtain the necessary data to interpret the indications observed during in situ inspections. Grinzato et al. [4] provided information on a test wall representing ancient construction of Italy with delamination problems. However, the wall finishing was plaster, and the orientation of the facade was not clearly indicated. Chew [5] provided information on delamination problem observed in the adhesive of ceramic tiles, but the test samples were placed facing east. Therefore, considering the current state of available information on passive IRT of adhered ceramic claddings, it was decided to use qualitatively computational methods for image enhancement.

Thermal contrast computation, pulse phase thermography, SIS, PCA, and NMF are some of the computational methods listed by researchers [6,7,8] for image enhancement and/or time-dependent analysis of IRT data. The last three methods were selected for the analysis of in situ IRT inspection data, and are explained briefly in the following paragraphs.
SIS is used both for image enhancement to remove unwanted effects such as uneven heating [7] and for defect detection by analysing the variation between the thermal behaviour of sound and defective areas [8]. For both purposes, pixel-by-pixel temperature differences of two thermograms are calculated. It is called ‘temporal reference technique’ when consecutive thermograms of the same area are used and ‘spatial reference technique’ when thermograms of different areas, one of which is the example of a sound element, are used [3].

PCA is a statistical method that reduces the dimensionality of a data set consisting of interrelated variables, and defined by equation:

\[ A = U R V^T \]  
Where \( A \) is an \( n \times m \) matrix, \( R \) is an \( n \times n \) diagonal matrix representing singular values of matrix \( A \), \( U \) is an \( m \times n \) matrix, and \( V^T \) is the transpose of an \( n \times n \) matrix [9]. Principal components (PCs) are a new set of transformed variables that are uncorrelated, and the first few PCs retain most of the variation present in all of the original variables [10]. Rajic [9], one of the first researchers to study its use in IRT, reported advantages in detecting delamination.

NMF is an approximate factorization method that can be used for the statistical analysis of multivariate data, and is represented by equation:

\[ V \approx WH \]  
Where \( V \) is a non-negative \( n \times m \) matrix, \( W \) and \( H \) are non-negative \( n \times r \) and \( r \times m \) matrix factors respectively. \( m \) represents the number of examples in the data set, and \( r \) is usually chosen to be smaller than \( n \) or \( m \), so that \( W \) and \( H \) are smaller than the original matrix \( V \) [11].

3 TIME-DEPENDENT IRT OF AN ADHERED CERAMIC CLADDING

3.1 In situ inspection and analysis procedures

The adhered ceramic cladding inspected to evaluate the potential of time-dependent analysis and to compare different computational methods was the south façade of a building in Lisbon (Portugal). The building had a reinforced concrete structural frame and hollow clay brick cavity walls. Only two areas of interest with pre-identified delamination problem, located at the ground floor level, and clad with glazed tiles of grey in colour was inspected (Figure 1). In this paper, only the results of area B are given.

![Figure 1: Ground floor south façade of the building and locations of inspected areas.](image)

IRT inspection was performed on August 1st, 2013, between 10:00-19:00. Thermograms of areas of interest were taken with half-an-hour interval using a Fluke TiR27 camera in 7.5-14 µm spectral range, with a detector of 240x180 pixels, and thermal sensitivity of 0.045 °C. The two
areas of interest were determined considering the results of a previous research study [12,13]. The current conditions of these areas were identified by tapping control by hand and/or rubber-head hammer. The tiles were accepted consist of nine smaller test areas, and whenever a sound change indicating a delamination problem was noticed, that test area was recorded as defective. Relative surface moisture contents of these areas were also measured using a surface moisture meter; Tramex Survey Encounter, using the ‘plaster’ option. Environmental conditions (e.g. air temperature, solar radiation) were also recorded during the IRT inspection.

Pre-processing was performed on IRT data prior to time-dependent analysis to correct variations caused by the change in camera viewpoint during each shot. Corrections were performed using Matlab R2012 software, and synthetic thermograms were generated in the end, using the pixel-by-pixel temperature data imported from the IR camera’s software. SIS, PCA and NMF were also performed using Matlab.

In PCA and NMF (Table 1), apart from their comparative performance, the effect of inspection timing on the results was investigated. The assumed convective heat gain and loss periods were determined for this purpose considering measured exterior air temperature data. IRT data obtained with 1-hour interval was analysed by PCA and NMF for the convective heat gain and loss periods and for the whole inspection period to evaluate the effect of inspection timing. In addition, in NMF calculations, two different ‘r’ values, which were selected in relation with ‘m’ (i.e. \( r_1 = \frac{1}{2}m \) and \( r_2 = m-1 \)), were used to observe its effect on the results. In SIS, the effect of time interval between IRT shots on the results was investigated for the convective heat gain and loss periods (Table 1). Additionally, in SIS7, the objective was to analyse the use of thermographic data obtained during opposite exterior environmental situations; i.e. during air temperature increases and decreases. For this purpose, the air temperature graph of the Portuguese Institute of Sea and Atmosphere (IP-MA - www.ipma.pt) in the Gago Coutinho weather station of Lisbon for the day of the IRT inspection was analysed. Considering the inspection period, thermograms taken 4 hours after the observation, of minimum and maximum air temperatures, were then selected for the analysis.

Table 1: Information on analyses performed.

<table>
<thead>
<tr>
<th>Code</th>
<th>Period&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Time&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Interval&lt;sup&gt;b&lt;/sup&gt;</th>
<th>Code</th>
<th>Period&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Time&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Interval&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
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<td>10:00, 10:30</td>
<td>½ hour</td>
<td>PCA1, NMF1a</td>
<td>heat gain</td>
<td>10:00-14:00</td>
<td>1 hour, 3</td>
</tr>
<tr>
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<td>heat gain</td>
<td>10:00, 11:00</td>
<td>1 hour</td>
<td>PCA2, NMF2a</td>
<td>heat loss</td>
<td>15:00-19:00</td>
<td>1 hour, 3</td>
</tr>
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<td>2 hours</td>
<td>PCA3, NMF3a</td>
<td>whole</td>
<td>10:00-19:00</td>
<td>1 hour, 5</td>
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<td>½ hour</td>
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<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Notes: <sup>a</sup>: Time of IRT shots; <sup>b</sup>: Time interval between IRT shots and ‘r’ value when applicable.

3.2 In situ inspection and analysis results

Test areas identified to have delamination during tapping controls are given in Figure 2(a). Exemplary synthetic thermograms showing the surface temperature variations at different hours of inspection are also given in Figure 2(b-d). During the early solar and convective heat
gain period (e.g. 10:00), most areas with delamination were visible at the thermograms with a surface temperature increase. As the amount of convective and/or solar heat gain increased, the visibility of thermal indications decreased, and finally disappeared (i.e. 17:00). During the late afternoon period (i.e. 19:00) without any solar load on the wall, indications started to be visible at some but not all the defective areas with a decrease in surface temperature.

Moisture map (Figure 3(a)) showing records taken on the day of the IRT inspection reveal that surface moisture contents of tiles ranged between 26% and 80%. It was also noticed that the moisture meter registered lower moisture contents at most areas with delamination. Apart from this, no resemblance between moisture map and thermograms was observed.
Results of SIS are given in Figure 3(b-h). With these results, SIS2 and SIS3 had apparent false indications as shown in Figure 3(c and d) respectively. SIS1 had indications at most areas with delamination, and they showed that, during the heat gain period, the surface temperature increase was greater at the defective areas than at sound areas. The same trend was observed at SIS2 and SIS3, where areas with false indications were ignored. SIS4, concerning the heat loss period, had conflicting indications at some areas with delamination as shown in Figure 3(e). An area with delamination revealed a slower surface temperature decrease while the opposite occurred at another area. Other analyses of the same period (i.e. SIS5 and SIS6) revealed a faster surface temperature decrease at the defective areas than that at sound areas. In SIS7, where surface temperatures during increasing and decreasing exterior air temperature spells were analysed, all areas with delamination showed higher surface temperature than their surroundings. However, indications with no relation to delamination were also present.

Selected PCs of PCA are given in Figure 4(a-c). As a relative analysis method, depending on the characteristics of the analysed data and behaviour of the defect, indications showing possible defective areas are visible at different PCs [after 6, 8]. In this case, PCA1 PC1, PCA2 PC2 and PCA3 PC2 had indications resembling areas with delamination. The visual comparison of delamination map with these PCs showed that all areas with delamination were visible with varying intensities, but there were also some indications that were not related to delamination. During the heat gain and loss periods, the variance of areas with delamination was greater than that of sound areas, while the opposite occurred in the whole inspection period.

![Figure 4: Area B: Results of (a-c) PCA and (d-h) NMF](image)

The first elements of matrix factors (MFs) of NMF are given in Figure 4(d-h) except NMF1b. Visual comparison of delamination map and MF1s showed that, apart from NMF1 (a
and b), indications showing all areas with delamination were present at all MF1s. Similarly to the case in PCA, there were also some indications that were not related to any delamination. At MF1s of NMF1 (a and b), close to the ground level, as pointed out on Figure 4(d), there were no indications at some areas with delamination.

4 DISCUSSION

Thermograms of passive IRT showed that areas with delamination were visible at early heat gain period in the case of the south facing façade with continuing solar irradiation on the surface. The visibility of the indications gradually decreased due to heat gain, and was not surely visible again during the remaining hours of inspection (i.e. until 19:00). Therefore, it can be said that the timing of the inspection, in relation to the orientation of the façade and the amount of heat gain, has an important effect on the accuracy of results in the case of localized analyses in time.

The SIS of different inspection periods and time intervals showed that false, missing or conflicting indications could occur, which might lead to false interpretations. Only two of the seven analyses, i.e. SIS6 and SIS7, did not show such problems. Therefore it can be said that, in the time-dependent analysis of thermograms, the potential of SIS is questionable.

The PCA of different inspection periods showed that all areas with delamination were visible in all analyses, but at different PCs, depending on the characteristics of the data set and the defect. Therefore, it can be said that PCA has a potential for time-dependent analysis of thermograms, and inspection timing does not have an apparent effect on the results, but expert knowledge may be needed for the selection, and thus interpretation of the appropriate PC.

The NMF of different inspection periods and with different ‘r’ values showed that nearly all areas with delamination were visible in all analyses. Only a relatively small area with delamination had no indications in the heat gain period analyses. A ‘linear’ coefficient of determination (R²) was used to analyse the effect of ‘r’ value on the results, where R² values of 0.999, 0.934 and 0.873 were determined for NMF1 (a and b), NMF2 (a and b) and NMF3(a and b) respectively, indicating a strong correlation. Therefore, it can said that NMF has a potential for time-dependent analysis of thermograms, and timing of inspection and ‘r’ value have a negligible effect on the results obtained for the case analysed.

5 CONCLUSION

Passive IRT of a south facing adhered ceramic cladding at Lisbon (Portugal) with pre-identified delamination problem was performed on August 1st, 2013 during day time; between 10:00 and 19:00. The main objectives of the inspection were ‘evaluating the potential of time-dependent analysis against localized one’, and ‘evaluating the comparative performance of different computational methods in time-dependent analysis, which were selected to be SIS, PCA and NMF’. Tapping control and surface moisture content measurements were performed to determine the current state of the cladding, and their results were used to evaluate the potential of time-dependent analysis with different methods. The following conclusions are drawn from the comparative analysis of all results;

- The time of inspection and inspection conditions have an important effect on the accuracy of thermographic indications when using a single thermogram, i.e. localized analysis, is preferred, while time-dependent analysis is not affected by inspection timing, depending on the analysis method applied;
- SIS has a risk of generating false or conflicting indications, and some areas with
delamination are not visible. Therefore, supplementary inspections with other techniques are necessary to determine the precise extent of the problem before starting any preventive (maintenance) operation;

- PCA and NMF have a great potential for time-dependent analysis of IRT data, as all areas with delamination, with one exception, were visible in all results. However, care should be taken when applying the technique since indications similar to delamination may be present in the results.

As further research studies, the following are proposed: additional inspections at different seasons of claddings facing different orientations, destructive tests at areas with similar indications as delamination, further analysis to determine optimum time periods for the inspections will be helpful in evolving the applicability and accuracy of time-dependent analysis of IRT data in detection of delamination, especially by PCA and NMF.

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REFERENCES

STUDY OF WOOD WASTE ASH AS A POZZOLANIC ADDITION

Ana M. Matos (1), Telma Ramos (2) and J. Sousa-Coutinho (2) (3)

(1) LEMC, Department of Civil Engineering, Faculty of Engineering of University of Porto
(2) LABEST, Department of Civil Engineering, Faculty of Engineering of University of Porto
(3) Associate Professor at Department of Civil Engineering, Faculty of Engineering of University of Porto

Abstract
Portland cement production is an energy-intensive industry, responsible for about 5% of the global anthropogenic carbon dioxide emissions. The present work deals with comparing effect on strength and durability of wood ash from a timber processing industry and a wood bottom ash from a grate operated biomass power plant. Various tests enabled physical and chemical characterization of these materials, including pozzolanic activity, chemical analyses and laser particle size distribution. Strength and durability properties were assessed on mortar, including carbonation, ASR and chloride penetration. Both wood ashes used as cement replacement in mortar indicated marginal or no loss in terms of strength, expected reduction in carbonation resistance and improved ASR resistance with increased replacement. Chloride resistance reduced for the non pozzolanic wood ash but increased for the pozzolanic ash. Wood waste ash is a complex waste material demanding prior assessment of effect in mortar and concrete for use as cement replacement for sustainability in construction.

1 INTRODUCTION
Every year more than 1 m$^3$ of concrete is produced per person worldwide [1] with Portland cement being the key ingredient, but producing the greatest environmental burden. Presently around 3 billion tons of Portland cement are consumed worldwide [2] and for the production of every 600 kg of cement, approximately 400 kg of carbon dioxide gas is released [3].

While cement production in its beginnings only focused on ordinary Portland cement, later cements with several main constituents were produced by replacing parts of the clinker content by supplementary cementitious materials [4].

Though timber processing waste is a sustainable and renewable source of fuel for energy production, the thermal process leads to significant amounts of fine wood waste ash as a by-product material which, if not managed properly, may result in serious environmental and health problems [3], [4].

In plants with efficient fluidized bed furnaces, ash produced is predominantly fine fly ash with only a small fraction of coarse ash retained within the combustion chamber but when grate fired furnaces are used, wood ash produced is coarser in nature and tend to settle inside the combustion chamber as bottom ash [5].
However research on use of coarse bottom ash from wood waste as partial cement replacement for concrete is scarce.

Several research programs have been carried out to incorporate fine wood waste fly ash as a cement replacement material [3], [5], [6], [7], [8], [9]. Results have indicated that wood waste fly ash can be effectively used as a cement replacement material for the production of structural grade concrete of acceptable strength and durability [4] and even self-compacting concrete [9].

The present work deals with comparing effect on strength and durability of wood ash from a timber processing industry and a wood bottom ash from a grate operated biomass power plant. Various tests enabled physical and chemical characterization of these materials, including pozzolanic activity, chemical analyses and laser particle size distribution. Strength and durability properties were assessed on mortar, including carbonation, ASR and chloride penetration.

2 EXPERIMENTAL PROGRAM

2.1 Materials, mortar production and fresh properties

Typical commercial CEM I 42.5 R Portland cement was used in this study. CEN reference sand for mortar production complying with NP EN 196-1 [12] was used.

Biomass consisting of wood and forest waste is collected in the woods surrounding Mortágua power plant in an area of a few kilometres in radius. This forest waste wood is then prepared and incinerated in a furnace to produce energy which is introduced in the public power gridlines (EDP-Energias de Portugal, S.A.). Combustion of the wood produces two types of materials – fly ash and bottom ash. Samples of wood bottom ash collected showed abundant coarse particles consisting of unburnt organic matter such as branches and pieces of wood. Dry wood bottom ash was sieved and particles under 4 mm were separated and ground in the laboratory in a ball mill, for 24 hours and named GWA [13].

Therefore, three types of equivalent mortars were produced, a control mortar (CTL1) with 100% of Portland cement, a second type with 5% cement replacement GWA (GWA5), and a third type with 10% replacement (GWA10) following the procedure described in NP EN 196-1 [17] (see Table 2). Prior testing had shown that higher replacements dosages are inadequate.

Wood waste ash was obtained from a timber processing industry where wood waste is burnt at high temperatures as fuel for energy production (thermal and electrical). To ensure similar fineness to cement, grinding was further performed for 10 min in a planetary mill [14].

Three mortar types were prepared following the procedure described in NP EN 196-1 [17] a control mix with 100% cement (CTL2), mixes with 10% (WWA 10) and 20% (WWA 20) of wood waste ash as cement replacement (see Table 2).

Chemical composition and physical characteristics of cement, WWA and GWA are given in Table 1 as well as standard requirements. Scanning Electron Microscopy (SEM) on a WWA, GWA and images can be seen in Figure 1.

Several test specimens were produced, demoulded the following day and then cured in water at 20º C in a fog room until testing.
Table 1 - Chemical and physical characteristics of cement and WWA and requirements for fly ash (NP EN 450-1 [10]) and pozzolanic materials in Portugal (NP 4220 [11]).

<table>
<thead>
<tr>
<th></th>
<th>Chemical composition (% mass)</th>
<th>Particle size distribution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>LOI</td>
<td></td>
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<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>SiO₂</td>
<td>20,43</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Al₂O₃</td>
<td>5,16</td>
</tr>
<tr>
<td></td>
<td>Fe₂O₃</td>
<td>3,32</td>
</tr>
<tr>
<td></td>
<td>SiO₂+Al₂O₃+Fe₂O₃</td>
<td>88,32</td>
</tr>
<tr>
<td></td>
<td>CaO</td>
<td>62,16</td>
</tr>
<tr>
<td></td>
<td>MgO</td>
<td>2,22</td>
</tr>
<tr>
<td></td>
<td>Na₂O</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>K₂O</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Na₂Oeq</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>TiO₂</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>P₂O₅</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>SO₃</td>
<td>3,84</td>
</tr>
<tr>
<td></td>
<td>Cl</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Pozzolanicity</td>
<td>-</td>
</tr>
</tbody>
</table>

|                  | D 10% (mμ)                    | 0,80                        | 0,95  | 1,10                       |
|                  | D 50% (mμ)                    | 7,91                        | 4,99  | 6,70                       |
|                  | D 90% (mμ)                    | 27,10                       | 23,62 | 27,50                      |
Table 2 - Mixture proportions of mortars.

<table>
<thead>
<tr>
<th>Mortar type</th>
<th>CTL1</th>
<th>GWA5</th>
<th>GWA10</th>
<th>CTL2</th>
<th>WWA10</th>
<th>WWA20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Portland cement, g</td>
<td>450</td>
<td>427.5</td>
<td>405</td>
<td>450</td>
<td>405</td>
<td>360</td>
</tr>
<tr>
<td>GWFA, g</td>
<td>0</td>
<td>22.5</td>
<td>45</td>
<td>0</td>
<td>45</td>
<td>90</td>
</tr>
<tr>
<td>Standard sand, g</td>
<td>1350</td>
<td>1350</td>
<td>1350</td>
<td>1350</td>
<td>1350</td>
<td>1350</td>
</tr>
<tr>
<td>water, g</td>
<td>225</td>
<td>225</td>
<td>225</td>
<td>225</td>
<td>225</td>
<td>225</td>
</tr>
<tr>
<td>Flow diameter, mm</td>
<td>211.1±1</td>
<td>216.2±1</td>
<td>216.5±0</td>
<td>201 ± 2</td>
<td>203 ± 2</td>
<td>199 ± 3</td>
</tr>
</tbody>
</table>

2.2 Mechanical strength
Flexural and compressive strength testing was undertaken at 7, 28, 90 and 180 days following the standard procedure in NP EN 196-1 [12].

2.3 Carbonation, Alkali silica reaction (ASR) and Chloride ion diffusion
Resistance to carbonation was generally assessed in accordance with the procedure described in RILEM CPC-18 [15] on three specimens for each mortar type in the WWA study but only one in the GWA work. Curing procedure and exposure to accelerated carbonation were different in each study (GWA and WWA).

The potential risk of alkali-silica reaction in concrete was monitored in accordance with the accelerated mortar bar test following ASTM 1567 [16], where two mortar prisms (25 x 25 x 250 mm) were used for each mortar type. In this test method, higher temperature (80 °C) and increased alkalinity (1 N NaOH) accelerates the reaction. ASTM C 1567 [16] method is used in this study to understand the expansion characteristics of plain and modified mortar. In fact this test method can be used to evaluate the effectiveness of additions in reducing the expansion due to ASR.

Two slight modifications were performed when following the procedure in ASTM. Instead of 1:2.25:0.47 cement: aggregate: water ratio required in ASTM, the ratio used was 1:3:0.5 which is considered in NP EN 196-1 [12]. ASTM C 1567 [16] also requires a specific particle size distribution of the (reactive) aggregate, but the particle size distribution used was the one...
considered in NP EN 196-1[12] CEN sand, which is similar. This sand was found to be reactive. After demoulding specimens were kept I water at 80º C for a day and then in 1N NaOH solution at 80º C for 14 days. Expansion is shown in Table 3.

The resistance to chloride permeability was evaluated by the CTH Rapid Method according NT Build 492 [17] which is a non-steady state migration method based on a theoretical relationship between diffusion and migration which enables the calculation of the chloride diffusion coefficient (Dns) from an accelerated test. An external electrical potential is applied axially across the specimen and forces the chloride ions outside to migrate into the specimen. After a certain test duration, the specimen is axially split and a silver nitrate solution is sprayed on to one of the freshly split sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation, after which the chloride migration coefficient can be calculated from this penetration depth as follows:

\[
D_{ns} = \frac{0.0239(273 + T)L}{(U - 2)t} \left( x_d - 0.0238 \sqrt{\frac{(273 + T)Lx_d}{U - 2}} \right)
\]  

where Dns is the non-steady-state migration coefficient (×10 \(-12\) m²/s), U is the absolute value of the applied voltage (V), T is the average value of the initial and final temperatures in the anodic solution (ºC), L is the thickness of the specimen, usually 50mm, xd is the average value of measured chloride penetration depth (mm), and t is the testing period (hours).

3 DISCUSSION

Both types of ash are low calcium (<10%CaO) and high silica content ashes. Oxide contents of each ash type are matched up to requirements for coal fly ash in accordance with NP EN 450-1 [10] and pozzolan requirements in accordance with NP 4220 [11], as can be observed in Table 1. The total value of the three oxides, SiO\(_2\), Al\(_2\)O\(_3\) and Fe\(_2\)O\(_3\) are over the minimum threshold required for coal fly ash and remaining values agree with requirements. Chlorides are well below requirements and loss of ignition falls in the limits [10] for Class A coal fly ash. However GWA tested negative for Pozzolanicity.

In terms of mortar, workability was not affected for the range of replacement considered. Therefore superplasticizer will not be necessary, leading to economic advantages in future concrete production.

Mechanical and durability properties are presented in Table 3. For each of the mortar properties analysed enhancement due to replacement of Portland cement by GWA and WWA was calculated by comparing the result with control mortar. A positive (+) result means there was enhancement concerning that property and a negative (-) result means that mortar with wood ash presented a worse result than the control mortar.

Strength in compression decreased with increase of cement replacement by GWA up to 28 days followed by an increase until 90 days. Elinwa and Mahmood studied compressive strength of Portland cement concrete (OPC) concrete mixes with different sawdust wood ash contents and with different periods of water curing. Compressive strength decreased with increase of percentage of ash and increased, as expected, with curing time [6]. Findings by
other authors for replacement between 5% and 30%, also show that compressive strength of concrete with wood ash is reduced relative to neat OPC concrete for all curing times [3]. However for WWA flexural and compressive strength showed similar results for WWA mortar compared to CTL mortar at 7, 28 and 90 days, which must be due to pozzolanic reaction of the high silica content in WWA, which tested positive for pozzolanicity (Table 1). Therefore, these results concerning WWA appear more promising than findings by other authors.

Carbonation depth for blended cement mixtures was greater than for the Portland cement mixture. It was found that carbonation increased along with the increase in wood ash content, which is consistent with the trend observed in concrete for various pozzolanic materials and probably due to CH reduction and consequent pH reduction.

Results obtained for ASR expansion related to CTL mortar indicated potential deleterious expansion according to ASTM 1567 (higher than 0.1% after 14 days in NaOH) for this particular combination of sand and cement.

Both GWA and WWA were effective in reducing ASR to expansion below the low risk target 0.1%. Increasing replacement percentage with WWA further reduced expansion.

These results are in accordance with Wang and Baxter who studied ASR expansion of mortar with high alkali cement, reactive aggregate and three types of co-fired (herbaceous with coal) fly ash, concluding that these additions mitigated ASR compared to OPC as binder [3][8].

Dns results show, as expected, considering GWA did not show pozzolanic properties, that diffusivity does not decrease with cement replacement by GWA. Wang et al. [19] used a coarser wood fly ash in concrete obtaining less strength from 1 month to 1 year and marginally higher chloride permeability than the pure cement control concrete at 56 days. GWA results seem to confirm findings by these authors. However, WWA as a pozzolanic material proved to enhance chloride resistance with increased percentage replacement.

4 CONCLUSIONS

GWA and WWA used as cement replacement in mortar indicated marginal or no loss in terms of strength, expected reduction in carbonation resistance and improved ASR resistance with increased replacement. Chloride resistance reduced for non pozzolanic GWA but increased for pozzolanic WWA.

For GWA Therefore durability of mortar may have not been improved because GWA proved to be crystalline and non-pozzolanic, unlike other types of wood waste (fly) ash. However GWA a nonglossy fine material may be used as filler in cement-based materials with marginal durability loss but improved strength at later ages.

WWA seems to be promising to use as pozzolanic partial replacement material for cement, with no strength loss and leading to enhanced durability.

Wood waste ash is a complex waste material varying with the combustion process, species of wood and further processing and therefore demands prior assessment of effect in mortar and concrete for use as cement replacement for sustainability in construction.
Table 3 - Obtained results in researches programmes.

<table>
<thead>
<tr>
<th>Age (days)</th>
<th>CTL1</th>
<th>GWA05</th>
<th>GWA10</th>
<th>Enhancement GWA05 versus CTL1</th>
<th>Enhancement GWA10 versus CTL1</th>
<th>age (days)</th>
<th>CTL2</th>
<th>WWA10</th>
<th>WWA20</th>
<th>Enhancement WWA10 versus CTL2</th>
<th>Enhancement WA20 versus CTL2</th>
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<tr>
<td>Flexural strength, Mpa</td>
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<tr>
<td>7</td>
<td>10.1</td>
<td>9.3</td>
<td>8.9</td>
<td>-9%</td>
<td>-13%</td>
<td>7</td>
<td>6.8</td>
<td>6.9</td>
<td>6.4</td>
<td>1%</td>
<td>-5%</td>
</tr>
<tr>
<td>28</td>
<td>11.1</td>
<td>10.4</td>
<td>10.1</td>
<td>-7%</td>
<td>-10%</td>
<td>28</td>
<td>7.3</td>
<td>7.0</td>
<td>7.6</td>
<td>-5%</td>
<td>5%</td>
</tr>
<tr>
<td>90</td>
<td>11.1</td>
<td>11.1</td>
<td>10.4</td>
<td>0%</td>
<td>-7%</td>
<td>90</td>
<td>8.1</td>
<td>7.6</td>
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<td>Compressive strength, Mpa</td>
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<tr>
<td>7</td>
<td>42.9</td>
<td>39.1</td>
<td>36.5</td>
<td>-9%</td>
<td>-18%</td>
<td>7</td>
<td>39.7</td>
<td>42.2</td>
<td>40.2</td>
<td>6%</td>
<td>1%</td>
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<tr>
<td>28</td>
<td>49.5</td>
<td>47.5</td>
<td>44.3</td>
<td>-4%</td>
<td>-11%</td>
<td>28</td>
<td>53.5</td>
<td>52.4</td>
<td>53.5</td>
<td>-2%</td>
<td>0%</td>
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<tr>
<td>90</td>
<td>52.3</td>
<td>56.8</td>
<td>50.7</td>
<td>8%</td>
<td>-2%</td>
<td>90</td>
<td>59.6</td>
<td>61.0</td>
<td>60.3</td>
<td>2%</td>
<td>1%</td>
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<tr>
<td>Dns, x10^-12 m²/s</td>
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<tr>
<td>78</td>
<td>15.5</td>
<td>17.9</td>
<td>16.9</td>
<td>-15%</td>
<td>-10%</td>
<td>90</td>
<td>13.9</td>
<td>12.9</td>
<td>9.9</td>
<td>8%</td>
<td>29%</td>
</tr>
<tr>
<td>ASR expansion (%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>0.1910</td>
<td>0.1495</td>
<td>10%</td>
<td></td>
<td></td>
<td>14</td>
<td>0.1993</td>
<td>0.16</td>
<td>0.07</td>
<td>18%</td>
<td>66%</td>
</tr>
<tr>
<td>Carbonation, mm</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>28</td>
<td>2.62</td>
<td>3.33</td>
<td>4.6</td>
<td>27%</td>
<td>76%</td>
</tr>
</tbody>
</table>
5 REFERENCES

PROBABILISTIC MODELING DURABILITY OF WOODEN WINDOW FRAMES

IJ.J. van Straalen (1), L.M. Abspoel-Bukman (1), W.M.G. Courage (1) and J. Mennink (1)

(1) TNO, Delft, The Netherlands – ijsbrand.vanstraalen@tno.nl; linda.abspoel@tno.nl; wim.courage@tno.nl; jeroen.mennink@tno.nl

Abstract

Nowadays, maintenance of wooden window frames strongly depends on the practical experience of the parties involved. To get a better understanding of the required maintenance period and maintenance actions, a model has been developed to predict durability. This model makes it possible to optimize maintenance in relation to the required quality, taking into account the current value of degradation and factors causing degradation, which reflect the building variables and its surrounding.

Since degradation processes have a strong stochastic nature, a probabilistic approach is applied in the model developed. This approach takes the stochastic nature of all relevant parameters into account and combines physicochemical relationships, test results, expert opinions and inspection results.

The outcome of the model is a prediction of the degradation process in time, taking into account uncertainty. The model is incorporated in a web-tool with a focus on practicality (suggesting maintenance actions, compare effects of maintenance). In this paper the principles of this probabilistic model are described.

1 INTRODUCTION

Maintenance of wooden window frames nowadays strongly depends on the practical experience of the parties involved. To get a better understanding of the required maintenance interval and maintenance actions, a project has been performed by TNO within the period 2009-2012. The aim of the project was to develop a practically applicable web-tool which quantifies the degradation behaviour of wooden window frames and enables the user to optimize maintenance costs.

A brief overview of durability and service life prediction methods over the past decades has been given in [1]. This overview shows the importance of the contribution of the CIB W080 working commission, related RILEM technical committees working in this area and the implementation of this topic within various countries around the world. It also stated that progress in this area of expertise, concerning durability, might be expected within the next
For the development of a practically applicable tool, the definition of the performance of wooden window frames during the exploitation phase of a building is required. To be able to use these functional requirements in the communication and agreement between building owner and constructor or maintenance company, it is necessary to quantify these requirements. To do this, degradation mechanisms have been identified with respect to each of the five functional requirements. Each functional requirement is described by several degradation mechanisms.

2 DESCRIPTION OF DEGRADATION OF WOODEN WINDOW FRAMES

The basis of the model is formed by the degradation mechanisms describing the five functional requirements. In total, 32 degradation mechanisms have been identified as given in table 1. This is based on more than 2 decades of experience within the field of practical consultancy related to maintenance of wooden window frames in the Netherlands.

For each degradation mechanism, the levels of performance are defined by symptoms. The definition of the symptoms is based on a classification system taking into account five levels of degradation from A (no degradation) to E (highest defined level of degradation), resulting in an inspection library describing all symptoms for the 32 mechanisms relevant to the degradation process. An example of the developed classification system is given in figure 1.

The speed of degradation of each of the 32 mechanisms of degradation depends on the value of the factors causing degradation. In total 27 factors have been identified on basis of expert knowledge. The overview given in table 2 shows that these factors of influence can be divided into the following three groups: design of the building and its surrounding, window frames and its details, and maintenance of existing buildings. These groups refer to the phases in the design and exploitation of a building. The choices made in each of these phases define the value of the factors causing degradation, which mean the speed of degradation can be modified and optimized. To be able to quantify the value of these factors, an additional classification system has been developed based on five levels of impact, ranking from 1 (low influence) to 5 (high influence), as for example illustrated in figure 2.

3 DEVELOPMENT OF DEGRADATION MODEL

The previous chapter has shown that the performance levels of the degradation mechanisms can be quantified by symptoms, classified by five levels, and the speed of degradation of these mechanisms (shift to a lower performance level) is dependent on the value of the factors causing degradation, also classified by five levels.
Table 1: Overview of all relevant mechanisms of degradation of wooden window frames

<table>
<thead>
<tr>
<th></th>
<th>Mechanism</th>
<th></th>
<th>Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Water tightness – connection to building</td>
<td>17</td>
<td>Shrinking or swelling</td>
</tr>
<tr>
<td>2</td>
<td>Water tightness – connection between window and window frame</td>
<td>18</td>
<td>Mechanical damage</td>
</tr>
<tr>
<td>3</td>
<td>Water tightness – connection between window or window frame and glass</td>
<td>19</td>
<td>Weathering of sharp edges</td>
</tr>
<tr>
<td>4</td>
<td>Sealant joint in connection to glass</td>
<td>20</td>
<td>Perimeter clearance</td>
</tr>
<tr>
<td>5</td>
<td>Sealant joint in other connections</td>
<td>21</td>
<td>Leaching of dust content</td>
</tr>
<tr>
<td>6</td>
<td>Air tightness – connection to building</td>
<td>22</td>
<td>Degradation of glass</td>
</tr>
<tr>
<td>7</td>
<td>Air tightness – connection between window and window frame</td>
<td>23</td>
<td>Reduction in ease of operation</td>
</tr>
<tr>
<td>8</td>
<td>Air tightness – connection between window or window frame to glass</td>
<td>24</td>
<td>Corrosion of fasteners</td>
</tr>
<tr>
<td>9</td>
<td>Degradation of timber – connection to building or face side</td>
<td>25</td>
<td>Corrosion of hinges and locks</td>
</tr>
<tr>
<td>10</td>
<td>Degradation of timber – on connections to or between windows</td>
<td>26</td>
<td>Reduction of glossiness</td>
</tr>
<tr>
<td>11</td>
<td>Degradation of timber – induced through glass</td>
<td>27</td>
<td>Degradation of color</td>
</tr>
<tr>
<td>12</td>
<td>Laminated parts become visible</td>
<td>28</td>
<td>Chalking</td>
</tr>
<tr>
<td>13</td>
<td>Finger joints become visible</td>
<td>29</td>
<td>Pollution</td>
</tr>
<tr>
<td>14</td>
<td>Cracks, knots and previous repairs become visible</td>
<td>30</td>
<td>Blistering</td>
</tr>
<tr>
<td>15</td>
<td>Connections open up</td>
<td>31</td>
<td>Cracking</td>
</tr>
<tr>
<td>16</td>
<td>Crooking or twisting</td>
<td>32</td>
<td>Loss of adhesion</td>
</tr>
</tbody>
</table>

Symptom 14 - Cracks, knots and previous repairs become visible

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
<td><img src="image5.png" alt="Image" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>ranking</th>
<th>symptoms</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>No cracks, knots and previous repair are visible; moisture content &lt; 20%</td>
</tr>
<tr>
<td>B</td>
<td>Laminated parts become visible; 20% &lt; moisture content &lt; 25%</td>
</tr>
<tr>
<td>C</td>
<td>Weathering and cracks become visible; moisture content &gt; 25% &amp; moisture penetration</td>
</tr>
<tr>
<td>D</td>
<td>Rotting of timber on the outside of the glass; max 30 cm³</td>
</tr>
<tr>
<td>E</td>
<td>Serious rotting of timber both in and outside of the glass; &gt; 30 cm³</td>
</tr>
</tbody>
</table>

Figure 1: Example of classification of five levels of degradation
### Table 2: Overview of all factors causing degradation of wooden window frames

<table>
<thead>
<tr>
<th>Design of the building and its surrounding</th>
<th>Window frames and its details</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Location and height of project</td>
<td>15 Species of timber</td>
</tr>
<tr>
<td>2 Orientation of façade</td>
<td>16 Lamination and finger joints</td>
</tr>
<tr>
<td>3 Inclination of façade</td>
<td>17 Paint system</td>
</tr>
<tr>
<td>4 Overhang of the roof</td>
<td>18 Amount of timber outside the glass</td>
</tr>
<tr>
<td>5 Method of installation</td>
<td>19 Size of the timber components</td>
</tr>
<tr>
<td>6 Depth of window frame in relation to rest of façade</td>
<td>20 Distance between sill and ground</td>
</tr>
<tr>
<td>7 Size of the window frame</td>
<td>21 Glass placed from inside or outside, sealant joint or bandage</td>
</tr>
<tr>
<td>8 Size windows and direction of opening</td>
<td>22 Joints</td>
</tr>
<tr>
<td>9 Separation between air and water tightness</td>
<td>23 Coverings</td>
</tr>
<tr>
<td>10 Window frame in contact with insulation of façade</td>
<td>24 Angle of components</td>
</tr>
<tr>
<td>11 Connections and couplers</td>
<td>25 Radius of components</td>
</tr>
<tr>
<td>12 Color and finishing of window frame</td>
<td>Maintenance of existing buildings</td>
</tr>
<tr>
<td>13 Reachability</td>
<td>26 Level of last maintenance</td>
</tr>
<tr>
<td>14 Maintenance program of window frame</td>
<td>27 Time since last maintenance</td>
</tr>
</tbody>
</table>

### Factor 5 – Method of installation

<table>
<thead>
<tr>
<th>ranking</th>
<th>Method of installation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Window frame placed at later time in building process</td>
</tr>
<tr>
<td>2</td>
<td>Window frames form façade</td>
</tr>
<tr>
<td>3</td>
<td>Window frame placed in timber frame structure, no masonry façade</td>
</tr>
<tr>
<td>4</td>
<td>Window frame placed in timber frame structure, masonry façade</td>
</tr>
<tr>
<td>5</td>
<td>Window frame placed in masonry façade</td>
</tr>
</tbody>
</table>

![Figure 2: Example of classification of five levels of impact of the factor](image-url)
To model the degradation mechanism in time, the relation between symptoms, factors causing degradation and time needs to be established. To develop such a model, theoretical models (physio-chemical, biological, or empirical based), expert knowledge/opinions, and/or inspection data of various buildings can be used. Currently there are no theoretical models available for any of the 32 identified degradation mechanisms. For this reason it has been decided to rely only on expert knowledge/opinions and inspection data of various buildings to develop the model.

The first step taken in the development of the model is the description of the lifespan of each of the degradation mechanisms. Since the nature of the degradation process is highly stochastic and both expert knowledge/opinions and inspection data of various buildings are effected by uncertainties, a generic description of the lifespan is applied which is already formulated within a probabilistic context. The lifespan $L$ represents the total period of time needed to degrade from a symptom value of A (no degradation) to a value of E (highest defined level of degradation) for each degradation mechanism:

$$L = A_0 + \sum_{i=1}^{n} A_i(x_i - x_{i,0}) + uS$$

(1)

Where $x_i$ is the value of the influence factor $i$, $n$ is the total number of factors causing degradation considered, $x_{i,0}$ the default value of the factor causing degradation $i$, $A_0$ is the coefficient of the model in case $x_i$ is equal to $x_{i,0}$ (default lifespan), $A_i$ is the coefficient of the model describing the lifespan related to the factor causing degradation $i$, $S$ is the standard deviation of the lifespan $L$, which cannot be represented by other variables, and $u$ is the standard normal distributed variable. Within the probabilistic description of the lifespan $L$, it is in addition assumed that both $x_i$ and $x_{i,0}$ are known as input parameter and for this reason deterministic, while, $A_0$, $A_i$ and $S$ are based on expert opinions and data of other buildings and for this reason stochastic.

Initially, the values of both $A_0$, $A_i$ and $S$ are determined by questioning experts with knowledge from daily practice. The procedure of this approach is described in [3]. Among other questions, the experts are asked to indicate the lifespan of a wooden window frame under specific circumstances, which are described by a given set of factors causing degradation. Also, the experts are asked to indicate the difference in lifespan if the value of only one of these factors changes. It turned out that the difference in lifespan is minimal if only one factor changes value. This made it not possible to distinguish the effect of the 27 different factors in this way. It was decided to combine the factors causing degradation into four groups (given numbers of factors refer to table 2):

- Weathering (1 to 4, 6, 12, 18, 19, 20, 23 and 24)
- Risk (5, 7 to 14, 16 to 19 and 21 to 25)
- Material (12, 15, 16, 17 and 21)
- Maintenance (13, 14, 26 and 27)

These groups make it possible for the experts to indicate both the lifespan of a wooden window frame given the value of these factors for each of the four groups, as well as indicating the difference in lifespan when the value of a factor of one of the groups changes. For this reason, $x_i$ in equation 1 does not make use of the separate factors, but the grouped factors. The value of the grouped factors is determined by the values of the individual factors, using a weighting procedure. This weighting procedure is also based on expert opinion. It is
noted here that the grouping performed here is similar to the grouping used in factor methods proposed in literature [2].

The second step taken in the development of the model is to define the relation between symptom levels \( (P) \), lifespan \( (L) \) and time \( (t) \). The following relation is used:

\[
P = P_{\text{crit}} \left( \frac{t-a}{L} \right)^c
\]

where \( P_{\text{crit}} \) is the highest value of degradation which can be reached (level E), and \( a \) and \( c \) are constants depending upon the type of degradation mechanism. The values of both \( a \) and \( c \) also have to be determined on basis of expert knowledge/opinion and or data, under the assumption that their values are deterministic and that the stochastic nature of degradation can be represented by the stochastic parameters \( A_0, A_i \) and \( S \) (in equation 1). Depending on the value of \( c \), the speed of degradation can either increase in time, decrease in time or be constant in time.

4 PROPOSED PROBABILISTIC PROCEDURE

With use of equations (1) and (2) the degradation of each one of the 32 symptoms can be calculated. Since the parameters \( A_0, A_i \) and \( S \) are assumed to be stochastic, the calculation procedure will differ from the procedure for a deterministic set of equations, which can be solved easily.

In case the degradation models are only based on expert knowledge/opinions, it is possible to determine the distribution functions (represented by its type, and parameters like mean value and standard deviation) of the stochastic parameters \( A_0, A_i \) and \( S \) by performing a statistical analysis on the answers given by the experts.

Within the practically applicable web-based tool of the prediction model developed, the choice has been made to also collect the inspection results of all buildings for which maintenance is predicted through use of the tool. These results contain the symptom values of all 32 symptoms on the specific date that the inspection took place and the value of the 27 factors causing degradation. Since inspections will be done on a regular basis for a large number of buildings, the database will grow over time. To update the prediction model represented by equations 1 and 2, the Bayesian statistic is followed. With this method the distributions of the stochastic parameters \( A_0, A_i \) and \( S \) can be refined as part of the calculations necessary to predict the degradation (performance level of the symptoms and matching lifespan) for a given specific set of factors. The a-priori distributions of the model represented by equations 1 and 2, are taken from the expert knowledge/opinion and with use of a so-called Bayesian analyses [4] all the data available in the database is used to determine the a-posterior distributions of the stochastic parameters \( A_0 \) and \( A_i \). The integration of the Bayesian equations is performed by the less time consuming Markov-Chain Monte Carlo method [4]. The result of this analyses is an update of equations 1 and 2 that can be used for prediction.

Besides updating the equations for prediction from expert knowledge/opinions to knowledge from actual inspection results, the collected data will also be used to validate the model. Validation of the model will be performed when data becomes available.
5 PRACTICAL APPLICATION FOR WOODEN WINDOW FRAMES

A web-based tool has been developed, based on the probabilistic model described in the preceding chapters. The tool is practically orientated, takes uncertainties into account, presents degradation curves for all considered degradation mechanisms and makes it possible to get an overview of degradations related to the five functional requirements: aesthetics, protection, operation, air tightness, and water tightness. In addition suggestions regarding maintenance actions are proposed and effects of maintenance actions can be compared.

The use of the proposed probabilistic model in practice is illustrated through an example of an apartment building with a southwest/northeast orientation. The northeast façade contains the gallery with the front doors of the apartments, as shown by the left picture in figure 3, while the southwest façade contains the windows of the living rooms of the apartments and the balconies, as shown by the right picture in figure 3. The northeast façade has window frames next to the front doors, which are covered from direct sunlight and which are located at the shadow side of the building. For the southwest façade, the window frames of the living rooms do not have any covering and are directly exposed to the sunlight. A difference in maintenance period might be expected, which can be calculated with the tool.

![Figure 3: Examples of overhang of roof at northeast façade of the apartment building in this example (left) and southwest façade of the apartment building (right)](image)

In case of the functional requirement aesthetics one of the degradation mechanisms governing this requirement is ‘reduction of glossiness’. Based on the current value of the symptoms and factors causing degradation the prediction of degradation is calculated. This is represented by the blue, red and green bended lines in figure 4. A minimum required performance level with a symptom value C is assumed, depicted by the horizontal blue lines in figure 4. Looking at the average prediction, a difference in time between maintenance periods of the two façades is equal to 15 months (59 months for northeast façade minus 44 months for southwest façade).

In addition to the predictions for all relevant degradation mechanisms and functional requirements, the tool gives also advise on the maintenance (in the example of reduction of glossiness this will be cleaning or re-painting) and on preventive measures by changing the factors causing degradation (in this example this could be installing cover plates over horizontal parts most exposed to direct sunlight in the southwest façade).
6 CONCLUSIONS AND RECOMMENDATIONS

- The optimization of maintenance of wooden window frames can be supported by the web based tool based on the prediction model described in this paper.
- Based on the predicted life spans for relevant degradation mechanisms and functional requirements, advices on maintenance and preventive measures can be underpinned.
- The developed prediction model can also be used for other building products like roofs, façade panels and installations.

REFERENCES

DURABILITY OF JOINTED REINFORCED CONCRETE PAVEMENTS UNDER SEVER EXPOSURE CONDITIONS: A CASE STUDY IN IRAN

Poura Arabali(1) and Mohammad Shekarchi(1)

(1) Construction Materials Institute, School of Civil Engineering, Faculty of Engineering, University of Tehran, Tehran – poura_arabali@alumni.ut.ac.ir, shekarch@ut.ac.ir

Abstract
Durability and service life of concrete pavements are endangered by several factors. These infrastructures may be subjected to deteriorative events such as freezing and thawing cycles, cracking due to shrinkage, curling, temperature gradient, and moisture gradient. In this paper, concrete mix design and behavior of a Jointed Reinforced Concrete Pavement has been investigated which is located in a region under severe exposure conditions. These conditions are due to the freezing and thawing cycles in the region, and application of deicing chemicals on the pavements which is prevalent in cold weather conditions in Iran. This pavement contains dowel bars in transverse joints. Low water to cementitious materials ratio, high amounts of air entraining agents, and silica fume have been used in concrete mix designs. 54 samples were made for testing and evaluating the mix designs in an experimental program consisted of freezing and thawing cycles, shrinkage, flexural strength, and compressive strength tests. According to ASTM C672, experimental results showed that almost no sign of scaling was observed in samples with high air contents with or without silica fume. A concrete mixture has been proposed, which meets the durability requirements of concrete pavements in the region.

1 INTRODUCTION
Compared to asphalt pavements, reinforced concrete pavements can offer a more sustainable solution due to lower maintenance [1, 2] and other environmental benefits, such as reductions in lifetime energy consumption and volume of aggregates used in construction [1]. However, some of the pavements exhibit significant level of distress before ending of the design life, needing extensive repair, rehabilitation, and maintenance. These activities cause severe inconvenience to the travelling public and cost millions of dollars over life of a pavement [3].

There are many factors that can lead to pavement distress. For instance, large numbers of cyclic loads as induced by traffic can reduce the performance of concrete pavements by
propagating cracks, deteriorating the elastic properties, increasing the fatigue fracture toughness [1, 4, 5], and leading to the brittle failure of the material. Fatigue deterioration of plain concrete has also been attributed to cyclic creep of the compression zone of concrete. [1]

Concrete mechanical properties alone cannot accurately predict pavement performance, since excellent long-term performance of concrete pavements is associated with both concrete strength and durability properties. The durability of concrete pavements can be defined as the ability to resist chemical and physical attack, primarily against environmental exposure. Particularly under harsh environmental conditions, constructing a highly durable concrete is important [6].

In cold regions, pavement structures are subjected to freezing and frost heave in winter, and thaw settlement and weakening during spring. Consequently, the mechanical properties of pavement structures can be greatly affected by seasonal changes in temperature and soil moisture. During winter, the stiffness of unbound layers generally increases because of ice bonding of soil particles in the base and subbase, and ice lens formation in the subgrade. In contrast, during spring thaw, the pavement structure may become saturated from thawing ice, and the bearing capacity can be substantially decreased. Under such conditions, substantial settlements can occur, if the structure is exposed to heavy freight vehicles. [7]

During the freezing season, intermittent freeze–thaw cycles can also affect structural adequacy [7]. The results indicated that longitudinal fracture and broken slabs were the major types of distresses of concrete pavements in permafrost regions [8].

Moreover, the use of deicing salts during winter roadway maintenance in most cold climate regions is growing, and pavement therefore needs to be able to simultaneously withstand repeated freeze–thaw cycles and the application of salt solutions [9]. The use of deicing chemicals leads to some adverse effects on the structure, function and environment [10,11]: damage to concrete, pavements and bridge decks (e.g., surface scaling and corrosion of reinforcement), rust in conduit pipes, and destruction of the soil ecological environment [10]. The presence of salts can also lower the freezing temperature of the pore solution, deepening the freeze-thaw cycle effected zone in the concrete [6]. The presence of salt can significantly increase the severity of freeze-thaw damage [6].

In one study [6], pavement durability is measured through concrete permeability and chloride penetration resistance. A low permeability reduces the ingress and movement of fluids, water, salts, and other pollutants. Reduced permeability and chloride penetration resistance are important in both reinforced and unreinforced pavements. According to this study, concrete permeability and chloride penetration resistance of ordinary Portland cement concrete pavements are closely related to compressive strength. Higher compressive strength results in lower permeability. [6]

It is commonly believed that mineral admixtures can significantly improve permeation-related durability of concrete, and air entrainment can enhance frost resistance, although it also causes a reduction in concrete strength [12]. However, it remains rather controversial in literature whether air entrainment is required for frost-resistant high performance concrete, and what the difference is between the effects of mineral admixtures and air entrainment on frost resistance [12]. According to an experimental investigation, it was demonstrated that frost resistance might be independent on strength of concrete; and it is revealed that, as to concrete at a 0.32 water/cement ratio, air entrainment should be a main approach to enhance frost resistance, although the pozzolans could be used to increase long-term strength of concrete [12]. However, according to another investigation [13], regarding the effect of
pozzolans on frost resistance, it was reported that pozzolans could make microstructure of concrete more compact and improve the frost resistance.

On the other hand, recent investigations of selected deteriorated concrete pavements in Minnesota have suggested that even some adequately air-entrained concrete pavements have prematurely deteriorated due to freeze-thaw. In these cases, it was found that the air content in the concrete had gradually been in-filled with secondary products such as ettringite due to repeated action of freezing-thawing, thus making the concrete susceptible to freeze-thaw attack. According to this study, higher level of entrained air content than that is conventionally used is necessary for a 60 year design life. [3]

Another source of distress in concrete pavements may be cracking due to the tensile stresses created by shrinkage, temperature and moisture differentials, and by the effects of curling and warping. To effectively control this cracking, it is important to have the joints properly spaced. For all undoweled slabs, the shrinkage of the concrete mixture should be minimized as much as possible through adequate curing. [14]

In this paper, mix design and material characterization of a jointed reinforced concrete pavement for construction in a residential region of Iran, near Tehran, is studied. Durability is a very significant factor in the pavement design in this region, since it is subjected to freeze-thaw cycles and application of deicing salts during winter. The application of high amount of air entertainment in combination with supplementary cementitious materials utilization is controversial for concrete pavement construction, especially in Iran, since there are few studies regarding concrete pavement durability in Iran. Therefore, the factors mentioned have been studied in concrete mix design for constructing a durable concrete pavement. Laboratory testing have been conducted and represented.

2 EXPERIMENTAL PROGRAM

54 specimens were made for laboratory testing. The experimental program investigated shrinkage, flexural strength, compressive strength, and behavior of concrete under freezing-thawing cycles. Concrete mixture proportions are shown in Table 1.

The results of Rangaraju’s study [3] confirmed that high performance concrete, produced with slag and high air content (7 to 10%), can sustain high number of freeze-thaw cycles without any deterioration. The accompanied loss in strength was overcome by limiting water-cementitious material ratio (w/cm) to a maximum value (0.40). Preliminary testing also showed that even at entrained air contents up to 10%, desired levels of strength at the time of opening to traffic were achieved. [3]

The conventional percentage of air entrained content for nominal maximum aggregate size of 25.4 mm and severe exposure conditions is approximately 6% [14,15]. However, in this study higher amount of air entraining agent was used, so that the air content of P1 and P2 mixes of fresh concrete were about 10.5%. This can lead to a loss in concrete strength. Thus, w/cm of the mixtures was limited to 0.36 to make up the loss. Polycarboxylate chemical additive was used as superplasticizer to provide workability of the mixture. Silica Fume (SF) was used in one of the mixes to evaluate its effect on the durability and strength characteristics of the concrete pavement.
Table 1: Concrete mix proportions for laboratory testing

<table>
<thead>
<tr>
<th>Mix</th>
<th>Cement (kg/m³)</th>
<th>SF (kg/m³)</th>
<th>Fine Aggregate (kg/m³)</th>
<th>Coarse Aggregate (kg/m³)</th>
<th>Water (kg/m³)</th>
<th>Superplasticizer (kg/m³)</th>
<th>Air entraining agent (kg/m³)</th>
<th>w/cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>375</td>
<td>0</td>
<td>1150</td>
<td>500</td>
<td>135</td>
<td>1.13</td>
<td>1.13</td>
<td>0.36</td>
</tr>
<tr>
<td>P2</td>
<td>350</td>
<td>25</td>
<td>1158</td>
<td>500</td>
<td>135</td>
<td>1.13</td>
<td>1.13</td>
<td>0.36</td>
</tr>
</tbody>
</table>

2.1 Flexural Strength

Modulus of Rupture (MOR) is often used in concrete pavement thickness design as a representative of concrete flexural strength. It is calculated as the tensile stress in the extreme fiber of a plain concrete beam specimen under third-point loading that produces rupture according to ASTM C78 [16]. The results from this procedure are used to represent the flexural strength of a concrete slab [14].

Concrete prisms were made for measuring flexural strength of concrete mixes. For each mixture and each age, three prisms were tested, and the average MOR of concrete specimens made of P1 and P2 mixtures at each age are represented in Table 2. The flexural tests were carried out under center-point loading according to ASTM C293 [17]. Then, the relationship between MOR values obtained from center-point loading and third-point loading, suggested by American Concrete Institute (ACI) [14], has been used:

\[
\text{MOR}_{3\text{pt}} = 0.9 \times \text{MOR}_{\text{center−pt}}
\]  

Table 2: Average Modulus of Rupture of specimens at different ages

<table>
<thead>
<tr>
<th>Mix</th>
<th>MOR (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3 days</td>
</tr>
<tr>
<td>P1</td>
<td>2.61</td>
</tr>
<tr>
<td>P2</td>
<td>2.79</td>
</tr>
</tbody>
</table>

Results show that although high air content was produced in concrete, specimens gained satisfactory flexural strength. According to ACI [14], under average conditions, concrete that has a MOR of about 4.8 MPa at 28 days is economical. Prisms with SF displacement gained larger amount of MOR, as predicted, and compensated the loss of strength more than samples without pozzolans.

2.2 Compressive Strength

While design of concrete pavements is generally based on the tensile strength of concrete, it may be useful to use compressive strength testing in field for quality control acceptance purposes and in the laboratory for mixture design purposes [14]. Compressive tests were carried out on cubic specimens. For each mix and each age, three prisms were tested, and the average strength and density of concrete specimens made of P1 and P2 mixtures at each age are represented in Table 3.
Table 3: Average compressive strength and density of the specimens

<table>
<thead>
<tr>
<th>Mix</th>
<th>Compressive strength (MPa)</th>
<th>Density (Kg/m(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3 days</td>
<td>7 days</td>
</tr>
<tr>
<td>P1</td>
<td>12.3</td>
<td>17.4</td>
</tr>
<tr>
<td>P2</td>
<td>13.4</td>
<td>25.5</td>
</tr>
</tbody>
</table>

The results show that despite the relatively low w/cm ratio, the high amount of air content caused a reduction in both compressive strength and density of hardened concrete, even in samples with SF displacement. However, samples with SF compensated the loss of compressive strength more than the others, because SF improves the interfacial transition zone, resulting in porosity reduction of this zone.

Due to the direct relationship between compressive strength and modulus of elasticity (E) which plays an important role in pavement thickness design [18], this module decreases with high air content. Therefore, pavement thickness should be increased, so that the final design can fulfill both durability and mechanical requirements.

2.3 Shrinkage

While drying, Concrete may shrink approximately 1.5 mm for every three meters of pavement length. This shrinkage may produce a tensile stress greater than the early tensile strength of concrete, leading to cracking [14]. Due to the large ratio of surface to volume in concrete pavements, their durability is potentially endangered by the shrinkage cracking. Spacing of the initial drying shrinkage cracks depends on concrete properties, variations in subgrade friction, curing, and climatic conditions [14].

In this study, for each concrete mix design, three prisms with dimension of 75×75×285 mm, and three prisms with the size of 100×100×500 mm were made, and their elongation was measured according to ASTM C157 [19] at different times during 90 days. Their average shrinkage is represented in Figure 1. The specimens were kept in a room with constant temperature and humidity (Figure 1). The temperature is maintained at 25±1°C and the relative humidity is constantly 50±2%.

The results show that there is a slight difference between shrinkage of samples with or without SF in the first 90 days. In comparison with other mixtures in other studies [20] which have approximately the same w/cm ratio, cement consumption, and/or SF displacement, these samples with high air content experienced higher shrinkage values. Additionally, the values of the E for the different types of concrete are reported to relate to the drying shrinkage values [21]. Therefore, by increasing the air content, and so reducing the modulus of elasticity, the pavement is more susceptible to the drying shrinkage cracking.

In the field, attempts were made to minimize the shrinkage of this concrete pavement through adequate and continuous curing for seven days. Furthermore, spacing of longitudinal and transverse contraction joints was limited to eight meters for preventing with the size of shrinkage cracking in reinforced slabs.
2.4 Freezing and Thawing Cycles

Mixtures used in this study have a low w/cm, adequate cement, entrained air, plus adequate curing and a period of drying, according to ACI 325 12R [14], so that the pavement should be able to resist cycles of freeze-thaw and the action of deicing salts. For evaluating this resistance, six specimens with dimensions of 100×200×250 mm were made for each mix design according to ASTM C672 [22]. This test method covers the determination of the resistance to scaling of a horizontal concrete surface, subjected to freeze-thaw cycles in the presence of deicing chemicals [22]. After completion of moist curing, the flat surface of the specimen was covered with almost 6 mm of a solution of calcium chloride and water, having a concentration of 40 g/lit. Then, they were exposed to 50 freeze-thaw cycles during 50 days. The specimen surfaces after 50 cycles have been shown in Figure 2.

At the end of 50 cycles, the specimens of P1 mixture showed very slight scaling with maximum depth of 3 mm, and no coarse aggregate was visible. The specimens of P2 mixture showed almost no sign of scaling. Thus, according to the ASTM C672 rating [22], which is between 0 and 5, the p1 mixture could gain 1, and P2 mixture gained 0, a better rating. Specimens with high air content have outstanding freeze-thaw resistance according to this standard method. SF could improve the concrete durability by reducing the concrete permeability and porosity of the interfacial transition zone.
3 DISCUSSION

The results of the experimental program showed that high air content could improve freeze-thaw resistance of concrete pavement. It also did not have remarkable negative effect on concrete MOR. However, it adversely influenced the compressive strength and concrete shrinkage. Shrinkage cracking is hazardous to pavement performance and concrete durability, and makes concrete pavement more vulnerable to deteriorative chemicals. In addition, for compensating the reduction of $E$, imposed by compressive strength loss, the pavement thickness should be increased. Therefore, it imposes high cost on the project. Moreover, although combination of SF with high air-entraining agents improved characteristics of this type of concrete, it could not solve the problem completely. Since SF could only redeem the material behavior slightly, its application in the field was not economical. Moreover, since concrete was pouring in hot weather of summer in the field, the initial rate of pozzolanic reactions and so drying rate would be increased. Thus, application of SF could increase the plastic shrinkage cracking potential during the first 24 hours.

By considering all of these factors, the mix design P1 was selected for construction in the field. However, the air-entraining agent was decreased, so that air content for field construction was limited to almost 7%. The mix proportion used in the field, and average compressive strength at 28 days ($f_{c'}$), tested for quality control, are represented in Table 4. The specimens under freezing-thawing cycles showed slight scaling, and according to the ASTM C672 rating [22], the mixture could gain the rating 2.

The pavement performance has been evaluated after several real freezing and thawing cycles. No remarkable sign of cracking or distress has been observed. The surface of the pavement has been shown in Figure 3.

Table 4: Concrete mix proportions and compressive strength

<table>
<thead>
<tr>
<th>Mix</th>
<th>Cement (kg/m³)</th>
<th>Fine Aggregate (kg/m³)</th>
<th>Coarse Aggregate (kg/m³)</th>
<th>Water (kg/m³)</th>
<th>Super-plasticizer (kg/m³)</th>
<th>Air entraining agent (g/m³)</th>
<th>w/cm</th>
<th>Air content (%)</th>
<th>$f_{c'}$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P3</td>
<td>375</td>
<td>1150</td>
<td>500</td>
<td>135</td>
<td>1.13</td>
<td>180</td>
<td>0.36</td>
<td>7</td>
<td>33.9</td>
</tr>
</tbody>
</table>

Figure 3: Surface of the constructed pavement

4 CONCLUSION

Experimental study showed that air content as high as 10.5% can greatly improve freeze-thaw resistance of concrete. However, it can adversely affect other aspects of the pavement durability such as shrinkage cracking potential. Moreover, it imposes extra costs by requiring
a rise in the pavement thickness. SF only redeems the behavior and durability of high air-entrained concrete slightly, and its application for concrete pavement durability is not economical. Therefore, by lowering the air content to 7%, and without using SF, a mixture was achieved that showed a satisfactory performance in freeze-thaw cycles test, and also after construction, so that the pavement could tolerate freeze-thaw cycles and deicing chemicals in the region. This mixture can be used for constructing durable concrete pavements with materials existing in this region.

REFERENCES
RESPONSE MONITORING OF NATIONAL HERITAGE FIVE STORY WOODEN PAGODA BY THE USE OF IMAGE PROCESSING.

T. Mikoshiba (1), Y. Niitsu (2), T. Hanazato (3) and C. Minowa (4)

(1) National Reseach Institute for Earth Science and Disaster Prevention, Tsukuba, Japan, miko@bosai.go.jp
(2) Tokyo Denki University, Chiba New Town, Japan, yniitsu@cck.dendai.ac.jp
(3) Mie University, Tsu, Japan, hanzato@arch.mie-u.ac.jp
(4) Tomoe Research & Development, Tokyo, Japan, ccmino@aol.com

Abstract
The Five storied pagoda is built of wood and is known as a high earthquake proof structure. The pagoda is composed of the frame and the center column. We began the response monitoring of national heritage five storied pagoda which is located in Ichikawa City close to Tokyo and constructed in 1622; Hokekyo-ji Pagoda. In the monitoring, image processing system is applied. Using the marker positions, the each story displacements to first story are calculated. The relative displacements were compared with two times acceleration integral displacement using filtering. In Great East Japan Earthquake, the displacements about 75mm and small residual were obtained.

1 INTRODUCTION

In Japan, as shown in Figure 1, there are five storied pagodas of more than 22, which were built before 1850. Although there are many reports of its characteristics under the earthquake and strong winds, there are a few reports on them under actual strong loads of the seismic and wind. Many micro tremor measurements of the real pagoda were conducted [1,2].

Figure 1. Five story pagodas constructed before 1850 in Japan (after Fujita[2])
Monitoring is carried out on the Nakayama Hokekyo-ji Pagoda, located at Ichikawa-shi, close to Tokyo. Image processing techniques was applied. We presents its response of 2011 East Japan Great earthquake, also under the strong wind load by using vibration sensors and image processing. The softening of rigidity according to the increase of response deforamton.

2 HOKEKYOU-JI TEMPLE FIVE STORY WOODEN PAGODA

The pictures of Hokekyo-ji Pagoda and its location are shown in Figure 1 and 2. It is a five storied pagoda and built of wood with Japanese old style connections. It is a 30.76m-high pagoda. It was dedicated in 1622 and renovated several times, in 1743, 1864 and 1912. The pagoda is composed of a frame and a center column. On the top of the center column, a metal ornament called a Sourin is attached. The structure has a square and symmetrical plan. The front of the pagoda is face to the southwest. The difference between pagoda axis and compass axis is 40degree. Figure.3 shows the cross section of the pagoda. The frame is composed of 5 blocks (stories) and the blocks are piled together. Around the center column(about ø450mm), four columns of quadrate section(about 200mm) are fixed on the composite girders. The center column is hung with the link system made up of 4 units of steel plate with two steel rings at two points; top position and the third story of the frame. The hanging column is estimated to be adopted in repairing work of 1912 or 1743. More investigation is required. The bottom of the center column is not fixed and it is structurally restricted to move freely by the iron dowel. The center column is structurally independent from the surrounding frame. Most of timbers were estimated to be replaced in partially demounted repairing work between 1900 and 1912. Judging from reconstruction of Hall between 1987 and 1997, the timber material species used for the pagoda were estimated Keyaki (in Japanese) for columns, pine for rafters, etc. In Great East Japan Earthquake, there was no damage in the Pagoda.
3 MONITORING SYSTEM

At first, vibration sensors were installed in Hokekyou-ji Five Story Wooden Pagoda. In a year, the image processing system was installed.

3.1 Vibration Sensor System
Vibration sensors and digital recorders were used. Horizontal and vertical accelerations are measured at several points on the frame and centre column. These are shown in Figure 3. Frame responses are recorded continually in sampling frequency of 100Hz. At center column, responses of more than 100Hz occurred as shown in Figure 5. Sampling frequency of 1000Hz is selected for center column.

3.2 Image Processing System
Image procession monitoring system consists of CCD camera, LED markers and data acquisition computer. Figure 6 shows outline of image procession monitoring system. The positions of the markers are captured by a CCD camera placed at first story beam. Each marker position (pixels) in camera image, shown in Figure 7, is calculated instantly in data acquisition personal computer on site. The marker position data are stored at hard disk in sampling frequency 10Hz, every hour. It takes one second to store one hour data in hard disk. One year data capacity is about 70giga byte.

3.3 Image Processing
Fundamentally, the marker in camera image is not a point light source. The light source is blurred in the camera image, and the luminance distribution of the pixels can be obtained from the pixels which exceeded the threshold intensity and then were recognized as an image of marker. The luminance gravity center are calculated and marker position is decided.

Figure 6 Image procession monitoring system.

Figure 7 Captured multiple markers image
4 LONG TERM CONTINUAL RECORDING OF IMAGE PROCESSING DATA

The image processing provided the long term horizontal deformation. Continual observation initiated at 0am from June 1st 2009. The records: relative displacements between fifth and first story beams are given in Figure 8. The seasonal and cyclic data fluctuation was found in the time history records. Humidity data about half year are shown in Figure 8. East Japan Great Earthquake shows the maximum data during nearly two years, and the relative displacements shifted about 10mm. However the shift values returned back to previous position. The continual observation after East Japan Great Earthquake stopped by electric power outage on March 16.

![Figure 8. Deformation between Fifth and First Story Beams from June 2009 to March 2011](image)

5 MONITORED RESPONSES OF GREAT EAST JAPAN EARTHQUAKE

Seismic Intensity around Ichikawa City, where Hokekyouji Temple locates, was IV in Great East Japan Earthquake (March 11th 2011). Maximum acceleration values on ground surface were respectively 0.279G, 0.135G, 0.167G in X,Y,Z directions. One time Integral velocity value was about 30cm/s. Biggest peak natural periods are about 0.7second in X direction, 3.5second in Y direction. Longest natural period was about 20second.

5.1 Sensor Records

Most of records recorded successfully. However many records were saturated. The records, which were taken at fifth and third story beams, went over the saturated levels in many times. The records of top and fourth roof girders would be reliable. Therefore, the maximum response acceleration values were estimated about 0.8G and 0.6G in X, Y directions. One time integral through 0.2Hz HPF were conducted to estimate velocity time histories, as shown in Figure 9 and 10. The wave shapes of integral velocity of saturated records were different from appropriate ones. The maximum velocity values of top roof girder were about 40cm/s.
All records of center column went over the saturated levels. The data recorded in 1000Hz. In the integrals, saturation effects were considered to be small. Therefore, the time histories of one time integral velocity through 0.2Hz HPF indicated the reasonable ones. Time histories of one time integral velocity are shown in Figure 11, 12.

5.2 Relative Displacement Observation Using Image Processing

The earthquake occurred at 2:47pm, and the sunlight glared with the LED markers. The automatic marker selection program wandered, and generated shock noises, as shown in Figure 13. Two markers were installed at each roof girder. If two marker positions differed very much, reasonable marker position was selected. In the case of no suitable positions, linear interpolation was based on the data before and after.

Relative displacements between fifth marker and CCD camera positions in X direction presented the maximum value about 75m, and residual relative displacement of 10mm remained, as shown in Figure 14. This relative displacement on March 16 after five days was gone. The fifth relative displacement in Y direction moved about 70mm. The residual relative displacements in hall direction were very small in comparison with X direction components in Figure 15.
6 MODEL ANALYSIS

Primitive model analysis was carried out. In the observed response of the Great East Japan Earthquake, the relative displacement between the fifth and first roof positions remained only 10mm residual. So, elastic analysis could be used for the discussion. The analysis methods already presented in WCTE paper (Minowa et al. 2010) of the assumed mode method were used. Major parameters in the analysis were the same as the analysis of the WCTE paper. Calculated natural frequencies are 0.56Hz (1.78s) of first mode, 1.27Hz of second mode, 1.60Hz of third mode. In microtremor measurement:

6.1 Responses to Great East Japan Earthquake

Five story pagoda responses to the 2011 East Japan Earthquake of Hokekyou-ji for each direction, X and Y direction are calculated. Equation without wind force inputs is used for the calculation. The inputs were the ground time histories, which were recorded at 14:47 on March 11th 2011. The input waves were through low pass filter 20Hz. The model and constants are the same as that of previous modal analysis. Calculation step interval time in the case is 0.0001sec. Figure 16 is the calculation results.

Figure 14. Relative displacements in X

Figure 15. Relative displacements in Y

CALCULATED EARTHQUAKE RESPONSES AT FIFTH STORY FROM GROUND

Figure 16. Calculated relative displacement time histories to the Great East Japan Earthquake.
6.2 Wind Responses

Strong wind responses were recorded in the morning on March 21, 2009. Observed maximum wind at Hokekyou-ji site was more than 30 m/s from south direction. The input is the wind speed time history, which was recorded at 13 m height from 4:40am to 5:20. Time history response calculations were conducted. Specific parameters for wind response analysis were introduced. Wind pressure area of the pagoda was assumed for the analysis, wind pressure coefficient was taken as $C_w=1.2$. The side wall effects were not considered. Figure 17 shows observed and calculated responses of 5th story in approach and hall directions. The model has only a simple elastic characteristic, therefore the drift deformation could not be described. However, the drift deformation in observation was vanished soon after strong wind, in a few hours.

7 DISCUSSIONS

Natural period extensions after increase of response displacements were recognized in observation, as shown in Figure 18. Natural frequency extended from 0.8Hz in small amplitudes to 0.5Hz in large amplitude of 5cm approximately. Also, damping constants were increased from 1-2% in small amplitude to about 10% in large amplitude.

Indicating in Figure 1, the national heritage five story wooden pagodas have fundamental natural frequencies between 1Hz and 0.5Hz in micro tremor amplitudes, approximately. Oldest one in seven century has the natural period of about 1 second. Judging from Figure 1, the deterioration or aging could not be identified.
Concerning species, zelkova and cypress were frequently used for Japanese traditional architectures, such as five story pagodas. Relations between strength and aging were estimated as Figure 19. And Young modulus would possess the linear relation with strength. Therefore, aging effects of wood Young modulus could follow the strength-aging relation of Figure 19. Hokekyou-ji five story pagoda, which was repainted in 1980 and 2007, was dismantled and repaired between 1900 and 1912, just 100 years ago. Indicating in Figure 19, cypress of 100 years after logging would bring the maximum strength (Young modulus), zelkova of 100 years after logging would be the 90% strength. According to the consideration, no changes of response characteristics, which are based on material aging, will be identified.

8 CONCLUSIONS

Image processing gave relative displacement data successfully. Concerning acceleration observation, some data saturated partially. Every observed acceleration data of center column saturated. However, one time integral velocities provided reasonable time histories, because of high frequency sampling 1000Hz. Image Processing gave the maximum deformation of 75mm, and residual relative displacement 10mm. The residual ones vanished in as few days. The dominant frequency elongation was observed. The rigidity of the pagoda was estimated to decrease at 40% of initial one in large amplitude. Elastic response analyses to the earthquake and strong wind were carried out. Damping in the earthquake is estimated to be more than 5%. High damping would be estimated at strong wind.

The Hokekyoji Wooden Pagoda could be concluded as a good maintained structure. No material degradation was found in visual observations, earthquake and wind response data analysis. Wooden structures could be sustainable.

REFERENCES

Outdoor exposure test of photocatalyst-coated materials in various test locations within Asia

Hiroshi Toyoda(1), Taketoshi Murakami(2), Taizo Sano(3) and Koji Takeuchi(4)

(1) Chief Research Engineer, R&D Department Taiyo Kogyo Corporation, Japan
   -TH000902@mb.taiyokogyo.co.jp
(2) Kanagawa Academy of Science and Technology, Japan -pg-murakami@newkast.or.jp
(3) Research Institute for Environmental Management Technology, Advanced Industrial
    Science and Technology (AIST), Japan - sano-t@aist.go.jp
(4) Research Institute for Environmental Management Technology, Advanced Industrial
    Science and Technology (AIST), Japan - takeuchi-koji@aist.go.jp

Abstract
Photocatalytic coatings have been widely used for building materials in order to keep their surfaces clean or to remove the environmental contaminants. Test methods for self-cleaning have been often published [1-4], but its performance has not been confirmed in different environmental conditions.

Then as a collaborative work of the Committee of Asian Standardization for Photocatalytic Materials and Products (CASP), that was established in 2007 to develop standard test methods, we have carried out the outdoor exposure test of both titanium dioxide (TiO$_2$)-coated and non-coated materials for nine months at twelve test locations in Asian countries. Japan, as the organizer, prepared and supplied the test pieces for the outdoor exposure test. The color difference between exposed materials and initial materials was measured to evaluate self-cleaning performance.

The TiO$_2$-coated materials showed excellent self-cleaning performance throughout the Asian countries. The original appearance of the materials can be maintained for a long time by application of a photocatalytic material, TiO$_2$. This result indicates that the photocatalysis aides in reducing the cost for cleaning as well as extending the life of the materials.

1 INTRODUCTION

A photocatalyst is a material that functions as a catalyst when exposed to light. There are various materials that show photocatalytic capability, and TiO$_2$ is said to be the most effective. TiO$_2$ usually absorbs ultraviolet light and is highly reflective white powder that is widely used as an ingredient of white pigment, white plastic and white paper. It is also utilized for cosmetic products since it shields against ultraviolet light. Furthermore, it was
found that TiO$_2$ shows a hydrophilic property as well as an oxidative decomposition ability under the irradiation of ultraviolet light$^{[5,6]}$. Then the photocatalyst is used in the products of various fields depending upon the effect, as shown in Figure 1$^{[7]}$. Among these areas of activity in TiO$_2$, the products that give self-cleaning performance are steadily increasing in the world.

![Figure 1: Application of TiO$_2$.](image)

In this report, we will evaluate the self-cleaning performance of TiO$_2$-coated materials by the outdoor exposure test at twelve locations in Asian countries and the effect of self-cleaning performance will be discussed.

2 EXPERIMENTAL

2.1 Specimen

Polyurethane (PU)-coated steel plate and polyvinyl chloride (PVC) tent fabric were chosen as the base materials. The size was 8cm x 30 cm. Firstly, we undercoated the base materials with a protective/adhesive layer to suppress the degradation of base materials caused by the photo-redox reaction with TiO$_2$. And then, the TiO$_2$ solution was applied onto the protective/adhesive layer. Figure 2 shows the cross section of the TiO$_2$-coated material.

![Figure 2: Cross section of the TiO$_2$-coated material](image)

We used five kinds of specimens that consist of both TiO$_2$-coated and non-coated materials for the outdoor exposure test.
Table 1 shows the details of the specimens and photocatalytic characteristics such as decomposing ability and hydrophilic property. Photocatalysis Industry Association Japan (PIAJ) certifies the limited contact angle for hydrophilic property and the decomposition activity index for decomposition ability to dirt[7]. Sample A does neither satisfy the hydrophilicity, nor the decomposition performance. Sample T satisfies the decomposition performance but does not satisfy the hydrophilicity. Sample B satisfies both the performances. Then, we compared self-cleaning performance for three kinds of TiO$_2$-coated materials.

### Table 1: Characteristics of specimens.

<table>
<thead>
<tr>
<th>Base material</th>
<th>Test method</th>
<th>PIAJ criteria</th>
<th>Polyurethane(PU) coated steel plates</th>
<th>Polyvinyl chloride (PVC) tent fabrics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface treatment</td>
<td></td>
<td></td>
<td>Non(PU)</td>
<td>TiO$_2$</td>
</tr>
<tr>
<td>Sample name</td>
<td></td>
<td></td>
<td>N</td>
<td>A</td>
</tr>
<tr>
<td>Limited contact angle (degree)</td>
<td>JIS R 1703-1</td>
<td>below 30</td>
<td>-</td>
<td>23.5</td>
</tr>
<tr>
<td>Decomposition activity index (R)</td>
<td>JIS R 1703-2</td>
<td>above 5</td>
<td>-</td>
<td>4.7</td>
</tr>
</tbody>
</table>

#### 2.2 Outdoor exposure test

The outdoor exposure test was carried out at twelve test locations in Asian countries. The specimens were attached to south- (north- only in Indonesia) facing racks with aluminum plate bars in the inclination of 45 (30, 23) degrees. Table 2 shows the outdoor exposure test sites. Figure 3 shows the scene of outdoor exposure test at a certain test place.

### Table 2: Outdoor exposure test sites.

<table>
<thead>
<tr>
<th>Country City</th>
<th>China Beijing</th>
<th>India Hyderabad</th>
<th>Indonesia Bandung</th>
<th>Korea Yongin</th>
<th>Malaysia Selangor</th>
<th>Philippine Taguig</th>
<th>Singapore Singapore</th>
<th>Taiwan, Hsinchu</th>
<th>Thailand Pathumthani</th>
<th>Vietnam Hanoi</th>
<th>Japan Kanagawa</th>
<th>Japan Tokyo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mark of country</td>
<td>C</td>
<td>ID</td>
<td>IN</td>
<td>K</td>
<td>M</td>
<td>P</td>
<td>S</td>
<td>TW</td>
<td>TH</td>
<td>V</td>
<td>KA</td>
<td>TK</td>
</tr>
<tr>
<td>Latitude</td>
<td>39.90</td>
<td>19</td>
<td>-6.91</td>
<td>37.24</td>
<td>3.50</td>
<td>14.51</td>
<td>1.35</td>
<td>24.80</td>
<td>14.00</td>
<td>21.03</td>
<td>35.3</td>
<td>35.6</td>
</tr>
<tr>
<td>Test angle (degree)</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
<td>45</td>
</tr>
</tbody>
</table>
Specimens were set up and placed in line as shown in Figure 4. Five kinds of specimens were sent back to the organizer after the prescribed outdoor exposure periods.

2.3 Evaluation

The color difference (delta E) between exposed specimens and unexposed specimens was evaluated by the color meter (KONICA MINOLTA :CR-10) based on ISO 7724-1 and ISO 7724-2. Measurements were carried out at three different points in one specimen and the average value was calculated.

3 RESULTS AND DISCUSSION

Outdoor exposed specimens after 9 months at Malaysia are shown in Figure 5.

TiO$_2$-coated materials (specimen A, B and T) keep the initial appearance, however, non TiO$_2$-coated materials (specimen N and P) change to dark color due to attaching dirt. The color difference (delta E) between exposed specimens and unexposed specimens was
evaluated by the color meter (KONICA MINOLTA :CR-10) based on ISO 7724-1 and ISO 7724-2. Measurements were carried out at three different points in one specimen and the average value was calculated.

### 3.1 Non TiO$_2$-coated materials

Figure 6 show the results of delta E for the outdoor exposed specimens at each location. Figure 7 show the comparison of delta E’s at all the test locations for each specimen. It is clear that delta E’s of sample N and P which belong to the non-TiO$_2$ one increase with the increase of exposure time for most of the test sites. This means that dirt attaches onto the surface of the samples. Delta E’s of samples in Singapore and Malaysia were the highest among all test locations, and the one in Kanagawa (Japan) was the lowest.

The PVC base of sample P contains a plasticizer. It is especially liable to adhere dirt matters because the plasticizer migrates to the surface and adsorbs dirt matters while they are exposed to heat, sunlight, and rain. Hence, delta E of sample P increased much higher than that of sample N.

As for the test angle, delta E’s for lower test angles of 23 degrees (Taiwan) and 30 degrees (Thailand) are slightly higher compared to those for 45 degrees.

### 3.2 TiO$_2$-coated materials

Delta E’s of TiO$_2$-coated materials (sample A, B and T) hardly change during nine-months, except for China. The exposure condition of China was exceptional. The test rack was shaded under the roof, then the photocatalytic activity slightly arose because of lack of sunlight and rain.

According to the judging criterion$^8$, delta E less than 5 is judged that dirt is not observed. Delta E of TiO$_2$-coated materials showed less than 5 even after nine months. Therefore, all the TiO$_2$-coated materials exhibit excellent self-cleaning performance in spite of the base materials and test locations.

Self-cleaning performance of three kind of TiO$_2$-coated materials (sample A, B, and T) was almost at the same level despite of the different photocatalytic properties.
Figure 6: Plot of color difference against exposure period for various samples at various locations.

Figure 7: Plot of color difference against exposure period for each sample at all locations.
4 CONCLUSIONS

4-1 Non TiO$_2$-coated materials
- More dirt adheres in Malaysia and less in Kanagawa.
- The main factor for dirt adhesion is site environments.
- Delta E’s for lower test angles are larger compared to that for 45 degrees.

4-2 TiO$_2$-coated materials
- TiO$_2$-coated materials show excellent self-cleaning performance at all the test locations.
- 45 degrees is an appropriate angle to evaluate the self cleaning properties.

5 ACKNOWLEDGEMENTS

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6 REFERENCES

REHABILITATION OF A WASTEWATER TREATMENT PLANT

Mário Pina Barreto (1), Isabel Torres (2)
(1) Águas do Mondego, Coimbra – pinabar@dec.uc.pt
(2) Department of Civil Engineering, University of Coimbra, Coimbra – itorres@dec.uc.pt

Abstract

Biogas is produced at various stages in the organs of wastewater treatment plants (WWTP), particularly those who are in contact with mud: primary and secondary sedimentation tanks, with emphasis on the biodigestors, principal organ of sludge treatment. The chemical composition of biogas is mainly CH4 (≈ 68%), CO2 (≈ 29%), H2S (≈0.02%) e pH (≈6/7).

During the process of biological treatment of wastewater treatment plant is naturally produced biogas whose composition includes the presence of sulphur dioxide, H2S. This component is a highly corrosive gas and it is the major responsible for appearance of pathologies in various elements of these WWTP. It attacks mainly metallic elements such as reinforcement, piping and other equipment of these installations.

Consequently, this gas in the absence of appropriate preventive measures may cause an increased number of maintenance and rehabilitation interventions in infrastructure, with increased costs in the respective repairs.

There are numerous situations of sanitation infrastructures where there is the presence of pathologies associated with biogas, which will have necessarily to suffer rehabilitation works with some wingspan.

This paper will present a case study of structural rehabilitation of organs of a WWTP (in biodigestors), including the diagnosis of the observed pathologies, its origin and the rehabilitation solutions adopted.

This case study ends with a description of the work performed and the materials that were carefully studied for technical solutions employed.

Keywords: biogas, sulphur dioxide, rehabilitation, WWTP.

1 INTRODUCTION

Biogas production over the bodies of domestic WWTP, particularly in those who are in contact with sludge and mostly in digesters is a reality. Hydrogen sulfide (H2S) is the component that causes greater problems in the maintenance of WWTP organs and pipes due to its highly corrosive power, being responsible for many rehabilitation interventions with its inherent costs.

In this paper we present the case study of the rehabilitation of two digesters carried out at the WWTP of Choupal in Coimbra, Portugal.
This is one of the oldest WWTP projects of a considerable dimension carried out by the Portuguese State. Its project was designed in the early 70s before the existence of any specific legislation for this purpose. Inaugurated in 1992, the treatment solution of Choupal WWTP is based on a treatment by trickling filters system.

At the time these works were accompanied by the first author.

It is the largest WWTP with a conventional treatment system by trickling filter in Portugal. This infrastructure is dimensioned to treat an average daily flow rate of about 40,000 m$^3$, from 220 thousand people.

2 PRESENTATION OF THE CASE STUDY

The WWTP Choupal presents the treatment of the affluent in a sequence of organs comprising three phases of treatment:

The liquid phase includes the following organs: preliminary waste water treatment (three rows of thick and thin bar screens, two grit chambers and two degreasing chambers, two primary clarifiers, four trickling filters and two secondary clarifiers.

The solid phase is composed by a gravitic thickener, two heated anaerobic digesters, a centrifugal, seven drying beds and a sludge park.

The gas phase (biogas production), has two mobile summit gasometers, a heating sludge system and a turbine powered by biogas for electricity production.

In this WWTP there are two similar circular digesters, each one having a 20 m inside diameter, seated on a conical base, topped with conical roofs. The vertical walls of digesters have 9.50 m in height, with 1.00 m below ground level. Whole the structure is in reinforced concrete, and the vertical walls are horizontally and vertically pre stressed.

In order to avoid friction losses each of the circular pre-stressed cables was divided into 4 sections, with the amendments made in concrete massifs salient from the shaft in which the extremities of the cable intersect staying the anchor heads located at their tops.

The amendments are made alternately in 8 massifs with 0.35m thickness (beyond the thickness of wall) and 2.40 m wide with the height of the wall.

Roofing is seated on the walls with a neoprene joint and with the intermediate and peripheral spaces filled with black agglomerated cork. Strangely, this joint that should be flexible has their movements restricted by pairs of steel bars that connect the walls to the conical cover. The connection of the walls to the base is rigid.

![Figure 1: View of digesters: before and after rehabilitation](image)

2.1 Observed pathologies: source and diagnosis

Since there was a high concentration of H$_2$S, it was found that the digesters had an inadequate internal coating, presenting serious structural problems that needed urgent
recovery as the reinforcement demonstrated some state of corrosion and it was visible a great
deterioration of the concrete and threatening danger of collapse of the structure.

In the joint between the wall and the cover there were signs of passage of gases and liquids
preventing thereby the maintenance of pressure in the tanks for the production of biogas.

The structural concerns were related to the existing state of generalized cracking in vertical
walls, the traces of iron oxide which indicated reinforcement corrosion and the
malfunctioning of the upper joint

![Figure 2: Malfunctioning of joint and fissure in the sealing concrete of the anchor heads](image)

Beyond a cracking with an undefined orientation eventually due to shrinkage it was
noticed with more evidence a vertically oriented cracking reflecting the effects of the
perimetral traction.

Cracks were also observed in the linking to concrete placed in a 2nd phase to cover the
heads of the anchorage pre-stress cables.

In-situ checks were done starting by removing the concrete (Figure 3) around an anchor
head, checking its state of conservation (because corrosion phenomena could have caused loss
of fixation and reduction of pre-stress) and confirming that the number and diameter of the
existing steel bars corresponded to the projected ones.

![Figure 3 – Removing of the concrete for the analysis of the anchor head](image)

It was concluded that there was no reinforcement in the concrete sealing of the anchor
heads which should have led to the appearance of cracks between this layer and the massive
of reinforced concrete.

A "scanner" was used to do a check of the reinforcement in reinforced concrete. This
method, while not allowing detecting accurately the diameters, allowed confirming a
reasonable correspondence between the project and the work.

Some simple tests of the strength of concrete were also performed using a esclerometer. In
these analyzes nothing unusual was detected (we obtained a mean value of 42 MPa, which
corresponding characteristic value was not very different from the class of the concrete projected, which was 35 MPa).

To try to better identify the cause of the problems a verification of the project was also done. It was concluded that the actions of the interior hydrostatic impulse used for determining the perimeter traction in these organs of the WWTP were correct (using the method of calculation of circular tanks authored by Montoya, Messeger and Moran). It was decided to proceed to a detailed analysis of the project drawings and it was found that the positions of the reinforcement of the walls was not right, they were changed between the inner and outer faces.

2.2 Employed corrective measures

It should be noted that all the works took place with WWTP in operation and this fact has complicated the development of the works and also the "exploration and operation" of the WWTP.

A prestressed structure based on outer peripheral ribs which would create compressive stresses contrary to those which should have led to vertical cracking was used.

The prestress was obtained by the application of screw bars that promoted the grip between different sections in a way to introduce a compression in the vertical walls of the digesters to avoid the appearance of cracking (figure 4, 5, 6 and 7). This reinforcement led to the closure of some cracks.

Despite the difference of efforts along the height, it was decided, for reasons of constructive facility, employing a single type of steel bar with different spacings. For that steel profiles UNP 100 class Fe 430 were used in order to allow the direct application of coating panels.

The tightening moment of the steel bars was applied in 3 stages and controlled by dynamometric wrench.

The placing and spacing of the rods had to attend to the location of several existing pipelines in the digesters. The internal sealing was also completely renovated and a liquid membrane of polyurea has been applied on the whole surface. The joint between the walls and the roof was also waterproofed with a hypalon membrane.

The works have been completed about 3 years ago being in full operation in the treatment system of the WWTP having been proven the success of the rehabilitation solution performed.

Figure 4: Metallic structure reinforcement –Steel bar
Em nenhum nível será aplicado aperto de varões com desfasamento superior a uma fase, em relação aos níveis contíguos.

Se os varões roscados forem cortados após galvanização, os seus topos deverão ter tratamento anti-corrosivo, a aprovar.

Momento a aplicar: 1ª fase 173 N.m
2ª fase 346 N.m
3ª fase 519 N.m

cerca de 400 (x 2)
classe 8.8 galv.
Varão roscado M24
Porcas c/ freio ou porca + contra-porca

NOTAS:

Eventuais calços em chapa para passagem de tirantes de aço para compensar irregularidades no betão de aço para compensar irregularidades no betão de aço para compensar irregularidades no betão

دا

Figure 5: Metallic structure reinforcement - Elevational view

Figure 6: Metallic structure reinforcement – Section

Figure 7: Metallic structure reinforcement – Constructional details
2.3 Main interventions and materials used

- Removal of the sludge from the digesters including the removal of the existing petrified zone in the bottom using percussion with water jet compressor and hammer.
- Removal of the pipelines and equipment that interfere with the construction of the metallic structure and its mounting at the end.
- Exterior cleaning of the walls of the digesters including runoffs. Rehabilitation of the deteriorated surfaces with a controlled retraction and fiber-reinforced mortar based on cement- binders, micro-silicate, special additives and selected aggregates.
- Filling of the zone of the anchor head with concrete (C30/37) including the application of additional steel bars A 400 (400 MPa characteristic strength).
- Removal of the deteriorated coating inside deposits, water-blasting and repair of the concrete degraded areas with a controlled retraction mortar and fiber-reinforced based on cement- binders, micro-silicate, special additives and selected aggregates.
- Rehabilitation of corroded steel bars by cleaning with high pressure water jet with sand and cement-based anti-corrosion painting. Repointing of cracks with a retraction controlled and fiber-reinforced mortar based on cement binders, micro-silicate, special additives.
- Steel structure for reinforcement of the structure consisting of steel sheets and steel profiles of Fe 430 (S275) class, including anticorrosion treatment connections and fixings to the reinforced concrete structure grounding and application of prestressing.
- In installation of the metal frame screws and threaded galvanized rod of the class 8.8 were used and the following technique was used:
  - The tightening of the rods was done with a torque wrench in 3 phases covering up all the deposit in each phase before moving on to the next one;
  - The moment applied in the torque wrench in the different phases was: 1\textsuperscript{st} stage 173 Nm, 2\textsuperscript{nd} phase 346 Nm and 3\textsuperscript{rd} phase 519 Nm.
- Repair and replacement of the existing expansion joint between the walls and roof so as to be guaranteed the possibility of movement with the gluing of a membrane of "hepalon" with 15 cm of width, 2 mm of thickness elongation capacity exceeding 400% and a tensile strength than higher 3 N/mm\(^2\)

The amendment of the joint was made by thermo-welding, with an overlap of about 5 cm.
• Application of a waterproofing coating and a protection chemically resistant to domestic effluent and to sulfur dioxide from the digester interior surfaces, so as to give them impermeability to liquids and gases with modified polyurea liquid membrane ability to bridge cracks up to 0.5 mm polyurea liquid membrane.

A cleaning, stripping and repair with waterproofing mortar of epoxy cement nature with 2 mm thickness an adhesion tension superior to 4.4N/mm² and a thermal expansion coefficient about 16.9 * 10⁻⁶ / °C was done previously.

3 CONCLUSIONS

The works were completed more than three years ago and the digesters are in full operation integrated in the operation process of the WWTP. Until now no more structural or non-structural pathologies have been noticed what confirms the success of the rehabilitation solutions carried out.

4 REFERENCES

Abstract

The deterioration of the investigated Austrian sewage system is attributed to several complex processes, which are referred to as bacteriogenically induced sulfuric acid attack. Anaerobic bacteria, present within the sewage system had consumed the organic matter, thereby reducing sulfate to hydrogen sulfide (H$_2$S). Subsequently, degassing of H$_2$S followed by its oxidation by aerobic bacteria Acidithiobacilli within the concrete ultimately led to the formation of sulfuric acid. This sulfuric acid reacted with the cement paste to form gypsum and bassanite. These minerals finally caused the severe damage of the concrete in the manholes.

Various crucial parameters for detecting alteration features were determined in the field and laboratory, including (i) measurements of temperature, pH, alkalinity, chemical compositions of the solutions, (ii) the characterization of the chemical and mineralogical composition of solids, and (iii), the determination of gaseous H$_2$S, CH$_4$ and CO$_2$ concentrations within the sewer pipe atmosphere. Stable isotope data were used to decipher individual reaction mechanisms.

1 INTRODUCTION

Corrosion of concrete based sewer systems due to the emission of hydrogen sulfide from wastewater is a huge economical factor and worldwide occurring issue. The so-called biogenic sulfuric acid attack can reduce the lifespan of concrete structures dramatically, from expected 100 down to 30-50 years, in extreme cases even down to 10 years [1, 2]. The required remediation is challenging and demands high costs. For instance, remediation charges of Los Angeles County exceeded €400 million in 1996 [3].
At the same time the reaction mechanisms and paths, which cause the deterioration of concrete are still under debate. In this study, we provide a deeper understanding about the driving forces of bacteriogenically induced concrete corrosion using a multi-proxy approach. For this purpose an Austrian sewer system which is urgently due for restoration, after a lifespan of only 8 years, was extensively investigated.

2 SITE DESCRIPTION

The investigated sewer system is handling the wastewater of around 13 000 persons. In 2004, two power mains were installed, in order to achieve proper wastewater transport. Ever since community complains about odor rose and subsequently deterioration of the concrete within parts of the gravity sewer started. Figure 1 displays a schematic sketch of the sewer system and Figure 2 shows one of the heavily corroded manholes.

3 MATERIALS AND METHODS

Solid and liquid samples from different manholes throughout the whole system shown in Figure 1 were collected for further mineralogical and geochemical analyses. Gaseous samples were measured in order to determine the concentration and the stable isotopic signature of sulfur. Additionally, long term gas observations were conducted within several manholes to value total H$_2$S volumes.

3.1 Liquids

Liquids were sampled throughout the whole sewer system for chemical analyses. The drinking water from the surrounding region was also analyzed in order to gain reference data. On site measurements of pH, electric conductivity, O$_2$ concentration and redox potential were carried out. In the lab, the solutions were filtered using 0.45 µm membranes prior to alkalinity analyses by potentiometric titration with 0.005 M HCl. The concentration of dissolved components was measured by a Dionex ICS-3000 ion chromatograph (IC) and a PerkinElmer Optima 8300 DV inductively coupled plasma optical emission spectrometer (ICP-OES).

3.2 Solids

Deteriorated concrete was sampled from different sites and shoveled into plastic bags. The samples were dried at 40°C, and subsequently grounded for mineralogical analysis using a PANalytical X’Pert PRO diffractometer (XRD). Mineral phase identification was carried out with the PANalytical X’Pert HighScore software (version 2.2e). A hydraulic press was used to extract the pore fluids of the concrete for the above chemical analyses [4].

3.3 Gas Phase

Concentrations of gaseous H$_2$S, CH$_4$ and CO$_2$ within the sewer pipe atmosphere were measured using a Draeger 3000 gas monitor. Several long term gas measurements were installed in order to determine variations in the total H$_2$S contents.

3.4 Isotope Analyses

Gas samples of the sewer atmosphere were taken using a gas sampling tubes. The isotopic signature of carbon was directly measured. The stable sulfur isotope ratios ($^{34}$S/$^{32}$S) were analyzed by combustion of BaSO$_4$- or ZnS- using a Thermo Finnigan 253 mass spectrometer [5]. The $\delta^{34}$S values, gained from BaSO$_4$ represent samples extracted from the wastewater and...
concrete. The $\delta^{34}$S values obtained from ZnS reflect the signature of gaseous hydrogen sulfide ($\text{H}_2\text{S}$), which was captured by a 5% zinc acetate solution and consequently precipitated as ZnS. Results are given in $\delta^{34}$S-notations in ‰ relative to the Vienna-Canyon Diablo Troilite (V-CDT) standard. Figure 3 shows one of the gas traps in which H$_2$S was precipitated as ZnS.

4 RESULTS AND DISCUSSION

The analyses of deteriorated concrete samples displayed mainly quartz and feldspar (albite and anorthite), which represent residuals of the siliceous aggregates of the concrete. Beside the latter phases, gypsum (CaSO$_4$·2H$_2$O) and bassanite (CaSO$_4$·0.5H$_2$O) were found in huge quantities. Thus, an almost complete transformation of the pristine cement matrix occurred, a process that is most likely caused by intense sulfuric acid attack. Figure 4 shows a typical composition of the deteriorated concrete within sewer manholes.

Redox potentials from -69 down to -310 mV were measured within the first storage basin and gravity sewer, respectively. This is indicative of strictly reducing environments related to intense bacterial activity within the power main. The resultant low oxygen levels of about 0.3 mg/l in wastewater from the storage basin favored colonization of sulfate-reducing bacteria strains within the biofilm of the power main. Bacteria of the genus Desulfovibrus and Desulfobulbus that occur in such environments are known to oxidize organic matter rapidly, using sulfate as electron acceptor in the absence of oxygen or nitrogen [1, 6]. The significant decrease in sulfate concentration with flow direction, from 250 to 24 mg/l verified the reduction of sulfate to sulfide speciation by bacterial activity. Simultaneously, the H$_2$S level rose up to 200 ppm in the atmosphere of the manholes. The H$_2$S was liberated from the wastewater into the manholes atmosphere due to changes in surrounding pressure, flow turbulences and temperature [2, 7]. Gaseous H$_2$S is considered to be rapidly absorbed into the surface of concrete where it oxidizes, either by biotic or abiotic control, over a chain of complex reactions [8]. During early stages of the biogenic sulfuric acid attack, acid/base reactions of CO$_2$ and H$_2$S with the concrete resulted in the decrease of the pH from ~13.5 to ~9.5 [3]. Under such pH conditions progressive colonization of the concrete’s surface by bacteria strains like Acidithiobacillus occurred. With persistent decreasing pH new strains of bacteria developed and colonized the surface of the manholes [9]. In this study, the expressed pore fluid from the corroded concrete yielded a pH value below 1, generating an extremely aggressive environment. The bacteria extracted from the deteriorated concrete were identified as Acidithiobacillus Thiooxidans and Acidithiobacillus Ferrooxidans. Those bacteria are known to be active at pH <1 if sulfate concentrations exceed 74 g/l [10]. In our samples the sulfate concentrations were up to 104 g/l. Figure 5 shows the activity of different bacteria families as a function of pH.

Biogenically induced corrosion of concrete has been verified by the $\delta^{34}$S analyses of BaSO$_4$ and ZnS, which clearly show an isotopic fractionation between the SO$_4$ of wastewater and pore fluids of about 10 ‰. The latter isotopic signature was transferred to the newly formed gypsum. While we obtained $\delta^{34}$S values between 6.0 and 8.5 ‰ for the wastewater, both the pore fluids and gypsum displayed $\delta^{34}$S values around -3.5 ‰. In addition, a trend towards an enrichment of the heavy stable sulfur isotopes in the wastewater was recognized with flow direction, emphasizing ongoing bacterial activity.
5 CONCLUSIONS

The application of a multi proxy approach, including chemical, mineralogical, biological and isotopic analyses, is a very useful tool to investigate complex reactions induced by biogenic sulfuric acid attack. The deterioration of concrete in an Austrian sewer system was clearly attributed to bacteriogenically induced sulfuric acid attack and associated formation and degassing of H₂S. The observed biogenically induced isotopic fractionation of 10 ‰ δ³⁴S indicates intense colonization of different families of sulfate reducing and sulfur oxidizing bacteria. The pH values below 1 created an extremely aggressive (micro)environment within pore structures of the concrete which enhanced the decomposition of the cement paste and its alteration into gypsum. Rapid concrete corrosion is unavoidable under such conditions. The development of new strategies for sustainable remediation of the sewer system is in progress.

REFERENCES

Table 1: Characteristic data of fluids from the storage basins (WW-SB1), the gravity sewer (WW_GS1/2) and expressed pore fluids of the deteriorated concrete (PF).

<table>
<thead>
<tr>
<th></th>
<th>pH</th>
<th>EC  [mS/cm]</th>
<th>Redox [mV]</th>
<th>O₂ [mg/l]</th>
<th>H₂S_(air) [ppm]</th>
<th>SO₄²⁻ [mg/l]</th>
<th>δ³⁴S [%o]</th>
</tr>
</thead>
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<tr>
<td>WW-SB1</td>
<td>8</td>
<td>1.351</td>
<td>-69</td>
<td>0.25</td>
<td>bdl.</td>
<td>64</td>
<td>5.9</td>
</tr>
<tr>
<td>WW-GS1</td>
<td>7.4</td>
<td>1.298</td>
<td>-157</td>
<td>1.72</td>
<td>up to 44</td>
<td>23</td>
<td>7.7</td>
</tr>
<tr>
<td>WW-GS2</td>
<td>7.9</td>
<td>1.303</td>
<td>-188</td>
<td>0.68</td>
<td>up to 35</td>
<td>45</td>
<td>8.4</td>
</tr>
<tr>
<td>PF</td>
<td>0.8</td>
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<td>n.a.</td>
<td>n.a.</td>
<td>n.a.</td>
<td>104210</td>
<td>-3.4</td>
</tr>
</tbody>
</table>

Figure 1: Schematic view of the sewer system with the two storage basins, the two power mains and the gravity sewer sections. Orange colors indicate intense corrosion, while grey colors represent low degradation degrees.
Figure 2: Deteriorated concrete within one of the manholes.

Figure 3: Gas trap in a corroded manhole. This trap consists of two “Woulf’sche bottles” that contain 5% Zn-acetate solutions and an air pump. Here, $\text{H}_2\text{S}$ reacts with the Zn-acetate to form solid ZnS.
Figure 4: XRD pattern of deteriorated concrete from sewer manholes. Main component is gypsum, with aggregate residuals of quartz and anorthite.

Figure 5: Distribution of bacteria families and their activities as a function of pH (modified after [9]).
DURABILITY OF CEMENT-REDUCED ECO-FRIENDLY CONCRETES AGAINST SULFATE ATTACK

Hainer S. (1), Rezvani M. (1), Baldermann A. (2), Proske T. (1), Mittermayr F. (3)

(1) Institut für Massivbau, Technische Universität Darmstadt, Darmstadt, Germany. hainer@massivbau.tu-darmstadt.de
(2) Institute of Applied Geosciences, Graz University of Technology, Graz, Austria.
(3) Institute of Technology and Testing of Building Materials, Graz University of Technology, Graz, Austria. f.mittermayr@tugraz.at

Abstract

The durability of two newly developed cement-reduced eco-friendly concretes with high limestone powder content and low water-powder ratios was recently successfully tested against carbonation and slight freeze-thaw attack by Proske et al. [1]. Mittermayr et al. [2] demonstrated the resistance of these eco-concretes against sulfate attack which will be highlighted in this study.

Mortar specimens with the same paste composition than that of the eco-friendly concretes were stored in a sodium sulfate solution and Ca(OH)2 reference solution for 200 days at 8°C, together with reference mortar samples that reflect conventional concrete compositions. Several analytical techniques were applied after immersion in order to assess mechanical, mineralogical, and microstructural alteration features related to sulfate attack. This includes expansion rate and residual compressive strength measurements, combined with X-ray diffraction and electron microprobe analyses.

Gypsum, bassanite and ettringite obviously caused the main damage as their occurrences are linearly correlated to the expansions rates of the mortar specimens. In all test specimens no thaumasite was found in spite of potentially favorable conditions. This may be related to the evolution of the experimental solutions and the competing formation of calcite and vaterite. Both eco-friendly mixtures exhibited a better sulfate resistance than their respective reference samples and are therefore suggested to be applicable sulfate-loaded environments.

1 INTRODUCTION

1.1 Principles of eco-friendly concretes

Recent cement production accounts for approximately 5 % of the global anthropogenic CO2 emissions and thus is a driving force for global warming and ocean acidification [3]. The majority of the produced cement is currently consumed by the concrete industry. Hence, the short- to medium-term decrease of the cement clinker content in concrete mixtures is suggested to be essential for the reduction of CO2 emissions connected to the concrete industry. Such alternative mixtures are referred to as eco-friendly concretes. Eco-friendly concretes are typically developed based on a promising concept which includes (i) the application of highly reactive cements, (ii) the reduction of the water volume and introduction of highly efficient superplasticizers as well as (iii) addition of inert additives and supplementary cementitious materials (SCM) such as limestone powder, fly ash, and ground granulated blast furnace slag (GGBFS) instead of cement clinker in the mixture [1]. Based on this approach a reduction of more than 30 % cement clinker content in comparison to conventional concrete can be achieved, while maintaining the required mechanical properties and durability of the concrete [1]. The positive effect of SCMs on concrete properties has been studied intensively in the last decades and their application in the concrete industry increased exceptionally [4]. However, the availability of e.g. reactive fly ash and GGBFS is limited in many countries and often cost-intensive [5, 6].
The efficient application of reactive SCMs with high contribution to concrete performance is therefore required. Currently, limestone powder additives have received particular attention complying with both ecological and economic benefits and mechanical requirements [7, 8].

In previous studies eco-friendly concretes with a reduced cement content and low water-powder ratio were developed successfully and their resistance against carbonation and slight freeze-thaw attack was demonstrated [1, 8, 9]. It has been proved that these eco-friendly concretes exhibit similar mechanical properties than the conventional concretes as well as an acceptable durability against freeze-thaw attack and water penetration [8, 9]. Compared to the conventional concretes the global warming potential of eco-friendly concretes was reduced by more than 30% [1]. However, the durability of these concretes against sulfate attack is still to question and therefore the aim of the present study.

In order to evaluate the resistance of the two newly developed cement-reduced eco-friendly concretes with high limestone powder contents against sulfate attack, laboratory experiments were performed by immersion of respective mortar specimens in a sodium sulfate solution (30 g/l Na₂SO₄) at 8 °C.

2 MIX DESIGN, EXPERIMENTAL METHODS AND PROCEDURE

2.1 Materials and mix design approach

The sulfate resistance of the cement-reduced eco-friendly concretes and conventional concretes was laboratory-proved by using mortar mixes with an equivalent paste composition [1, 8, 9]. In total, four cement-reduced mortars containing limestone powder and fly ash as well as three conventional reference mortars were tested. The mix design of the mortars is shown in Table 1. In order to investigate the effect of the cement type on sulfate resistance, three normative cements, namely (I) Portland cement (CEM I 32.5 R), (II) Portland-slag cement (CEM II/A-S 52.5 N), and (III) blast furnace slag cement (CEM III/B 42.5 N) were used. The applied limestone powder had a calcium carbonate content of > 98 wt. %, with an average grain size diameter of ~15 µm and a Blaine value of 330 m²/kg. The fly ash had a similar grain size, but a slightly lower Blaine value of 300 m²/kg. In all mixtures the CEN standard reference sand (EN 196-1) with a maximum grain size of 2.0 mm was used. Mixtures Ref I, Ref II and Ref III, made of the respective cement types (I), (II) and (III) represent conventional reference mortars with a water/cement-ratio (w/c) of 0.60. This is the maximum value according to the German standard (DIN EN 206-1/DIN 1045-2) for exposure class XA1 with sulfate concentration in groundwater of 200 < SO₄²⁻ ≤ 600 mg/l. The cement content of these reference mortars was in accordance with EN 196-1. Mixtures Ref II* and Ref III* reflect semi-reference mortars with a water/powder ratio (w/p) of 0.60 and correspondingly higher w/c values of 1.07 and 0.76. The mix design of the cement-reduced eco-friendly mortars is characterized by high limestone powder contents and a low w/p-ratio of approximately 0.35 (Eco II and Eco III). The percentage of cement reduction in these eco-mortars was up to 35% compared to the equivalent concretes [1].

<p>| Table 1: Mix design for the mortar samples, from Mittermayr et al. [2]. |</p>
<table>
<thead>
<tr>
<th>Mix ID.</th>
<th>Unit</th>
<th>Ref I</th>
<th>Ref II</th>
<th>Ref III</th>
<th>Ref II*</th>
<th>Eco II</th>
<th>Ref III*</th>
<th>Eco III / Eco III-28d</th>
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<tbody>
<tr>
<td>Water/cement (w/c)</td>
<td>[-]</td>
<td>0.6</td>
<td>0.6</td>
<td>0.6</td>
<td>1.07</td>
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<td>0.76</td>
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<td>Water/powder (w/p)</td>
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<td>0.6</td>
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<td>0.6</td>
<td>0.36</td>
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<td>Cement type</td>
<td>[-]</td>
<td>CEM I 32.5 R</td>
<td>CEM II/A-S 52.5 N</td>
<td>CEM III/B 42.5 N</td>
<td>CEM II/A-S 52.5 N</td>
<td>CEM III/B 42.5 N</td>
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<td></td>
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<tr>
<td>Clinker [g]</td>
<td></td>
<td>450</td>
<td>405</td>
<td>339</td>
<td>262</td>
<td>293</td>
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<td>32</td>
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<td>486</td>
<td>290</td>
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<td>Limestone powder [g]</td>
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<td>256</td>
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<td>Water [g]</td>
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<td>233</td>
<td>270</td>
<td>241</td>
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<td>PCE Superplasticizer [g]</td>
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<td>-</td>
<td>-</td>
<td>2.9</td>
<td>-</td>
<td>1.6</td>
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<td>Standard sand 0-2 mm, according to DIN EN 196-1</td>
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</table>

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2.2 Casting, curing, and test procedure

Mortar specimens - prisms (40×40×160 mm³) and thin prisms (10×40×160 mm³) - were fabricated according to EN 196-1. The mortar samples were demolded after 48 ± 2 hours and subsequently stored in a saturated Ca(OH)₂ solution for 14 days at 20 °C. On the basis of the SVA-testing procedure for sulfate resistance used in Germany [10], the specimens were pre-cured 14 days at 20 °C in saturated Ca(OH)₂ solution and then divided into two aliquots and subsequently stored in 30 g/l Na₂SO₄ and saturated Ca(OH)₂ reference solution, respectively, at 8 °C for 200 days. Distilled water was used for the preparation of the experimental solution which was renewed every four weeks. During the immersion period, the volume ratio of the sodium sulfate solution to the sample was 6:1. The specimens were placed on plastic spacers about 2.0 cm above the bottom of the tank. In order to study the effect of hydration, the pre-curing time of three specimens of Eco III type was extended to 28 days (named Eco III-28d), followed by immersion in Na₂SO₄ and Ca(OH)₂ solutions at 8 °C as described above.

The strain development of the thin prisms was measured in two weeks intervals by means of a strain gauge with an accuracy of 0.001 mm/m. The compressive strength of the mortar prisms was determined after 56 days of storage, in accordance with EN 196-1. Visual inspection and subsequent mineralogical and microstructural investigations were conducted on the mortar prisms that were immersed for 200 days.

3 RESULTS AND DISCUSSION

3.1 Visual inspection

Typical appearances of mortar samples stored in 30 g/l Na₂SO₄ solution for 200 days are pictured in Figure 1. In order to evaluate the visual deterioration level, the samples were divided into four categories reflecting no damage, minor damage, major damage and destroyed. The Ref II and Ref II* mortar mixes suffered severe damage due to intense sulfate attack and were completely destroyed, while Eco II and Ref I mixes developed a major degree of damage. Ref III and Ref III* showed only some, but rather less pervasive visual cracks on the surface and were therefore classified into the minor damage class. Eco III and Eco III 28d offered no visual damage, suggesting the highest sulfate resistance.

3.2 Compressive strength

The compressive strength values of samples immersed in a reference Ca(OH)₂ solution and sodium sulfate solution for 56 days at 8 °C are presented in Figure 2. After 14 days of pre-storage, the CEM II/A-S-based reference mortar (Ref II) exhibited a compressive strength of 39 N/ mm² which is significantly higher than that of the CEM I and CEM III-based reference mortars (Ref I and Ref III) with approximately 30-33 N/mm².
Considering constant water contents, the reduction of the cement content resulted in decreasing compressive strength values. For instance, Ref II* and Ref III* mixes are characterized by lower compressive strength values of 28-30 N/mm² compared to Ref II and Ref III. The low w/p-ratio of Eco III resulted in an increase in compressive strength up to 40 N/mm² compared to Ref III, whereas Eco II and the corresponding mix Ref II offered the opposite behavior. Notably, prolonged curing of 28 days (Eco III-28d) strongly increased the compressive strength up to 49 N/mm².

After 56 days of immersion in saturated Ca(OH)₂ solution a significant increase in compressive strength was recognized for all mortar mixes (Figure 2). The eco-friendly mixes (Eco II, Eco III and Eco III-28d) exceeded the compressive strength value of their respective reference mortars. However, after 56 days of storage in Na₂SO₄ solution the compressive strength of the mortar mixes decreased up to 7 %, except for Ref I, Ref II and Ref II*, where the compressive strength was slightly improved (Figure 2). However, after 200 days of sulfate exposure the latter mortars developed the highest visual damage degrees (Figure 1) as well as the largest expansion rates.

The increase of strength between 14 days pre-storage and 56 days storage in Ca(OH)₂ solution can be explained by the progressing formation of hydrated phases in the cementitious matrix. This process is distinctive for mortars with latent hydraulic and puzzolane additives like blast furnace slag and fly ash, respectively. After 56 days of sulfate exposure, slight improvements in compressive strength were recognized in some mortars, i.e. Ref I, Ref II and Ref II* (see Figure 2), which reflects the microstructural densification of these mortars due to the development of expansive products such as gypsum and bassanite [11]. Our results reveal that specimens that showed increasing compressive strength values after sulfate exposure exhibited a higher expansion rate after 56 and 91 days, suggesting that initial improvements in strength went along with negative effects of excessive expansion of the alteration products. Consequently, the results of the strength measurements after Ca(OH)₂ exposure can be used for the characterization of the mechanical mortar properties. However, the strength evolution during sulfate testing did not provide reliable information about the sulfate resistance.

![Figure 2: Compressive strength of mortars after pre-storage in Ca(OH)₂ and after 56 days exposure in Na₂SO₄ solution, modified after Mittermayr et al. [2].](image)
3.3 Strain

The strain values were determined by subtracting the strain of mortars stored in saturated Ca(OH)$_2$ solution (as control specimens) from those immersed in 30 g/l Na$_2$SO$_4$ solution. The mortar samples designed with CEM I 32.5 R and CEM II/A-S 52.5 N showed much higher expansions than the mortars made of CEM III/B 42.5 N. Accordingly, Ref I exhibited the largest expansion of 1.5 mm/m, followed by Ref II (1.0 mm/m) and Ref III (0.55 mm/m). Ref II* displayed a drastic elongation of 4.78 mm/m. Surprisingly, Ref III* (0.31 mm/m) exhibited a lower strain than Ref III despite the higher w/c-ratio. The behavior of Eco II was similar to that of Ref II, as seen in the comparable elongation of about 1.0 mm/m. The lowest strain development was recognized in the Eco III and Eco III-28d mortars, as indicated by the low strain values of 0.1 and 0.05 mm/m, respectively. It is notable that all of the blast furnace slag cement-based mortars fulfilled the requirements of the SVA-test limit for sulfate resistant cement (< 0.5 mm/m), notwithstanding their higher w/c-ratio.

Until 56 days of sulfate exposure, all mortar mixes had expansion rates below the guideline limit [10] of 0.5 mm/m (see Figure 3). After 56 days a significant expansion was evident for Ref I, Ref II, Ref II* and Eco II typed mortars. On the other hand, the Ref III, Ref III*, Eco III, and Eco III-28d mixes showed no significant expansion until 91 days of exposure. These observations clearly indicate that Portland- and Portland slag cement based mortars are more susceptible to sulfate attack than blast furnace cement-based mortars. Reactive hydration products such as portlandite and calcium aluminate hydrate are considered to be less abundant in slag and fly ash based cementitious materials and thus their widely absence lower the risk of formation of expansive phases during sulfate attack like gypsum and ettringite [12].

![Figure 3: Expansion rates of Ref I, Eco II and III during 90 days, modified after Mittermayr et al. [2].](image)

3.4 Mineralogical composition and microstructural evaluation

Comparison of the results obtained from XRD analyses and strain development revealed a direct correlation between the expansion of the mortars and their corresponding total gains of sulfate mineral phases including gypsum, bassanite and ettringite. Severe damage and plenty of decomposition features such as intense cracking and leaching were evident in some mortars, as seen in the back-scattered electron (BSE) images shown in Figure 4. Since the interface transition zone (ITZ) contains the highest portlandite contents
and a higher porosity than the rest of the cement paste [13], gypsum was mainly developed in the ITZ between aggregates and matrix.

Figure 4: Back-scattered electron images of Ref I and Eco III obtained after 200 days of exposure in 30 g/l Na$_2$SO$_4$ solution, modified after Mittermayr et al. [2].

### 3.5 Exposure classification and application

The Portland- and Portland slag cement based mixtures Ref I, Ref II and Ref II* exhibited a generally low resistance against sulfate attack. Thus, the application of these mixtures in highly sulfate-loaded environments is not recommended. However, the application in low sulfate environments (XA1) with $200 \leq \text{SO}_4^{2-} \leq 600$ mg/l is acceptable, according to DIN 1045-2/EN 206-1. The cement-reduced Eco II mortar mix offered a slightly better sulfate resistance than the corresponding mortar Ref II. According to the equivalent performance concept for concrete properties, a mixture such as that of Eco II should be acceptable for environments under slight sulfate attack (XA1), notwithstanding the normative minimum w/c-value of 0.60.

In spite of a slightly higher w/c-ratio than required by the SVA-guideline [10] (w/c = 0.50) Ref III*, Eco III and Eco III-28d have developed less than 0.5 mm/m expansion and thus passed the test. Consequently, concretes made of these mortars are suggested to be applicable against moderate sulfate attack (XA2) for $600 \leq \text{SO}_4^{2-} \leq 3000$ mg/l, according to DIN 1045-2/EN 206-1. Since the measured expansion rates of Eco III and Eco III-28d were very low, concretes with such a paste composition are probably suitable for concrete structure exposed to severe sulfate attack (XA3) with $3000 \leq \text{SO}_4^{2-} \leq 6000$ mg/l.

### 4 CONCLUSION

Based on our findings the following conclusions can be drawn for the reference mixes and cement-reduced eco-friendly mortars (Eco II and Eco III):

- A good correlation between expansion rate and visual damage degrees was observed for all specimens, whereas assessing the damage levels by means of residual compressive strength measurements was not beneficial.
- The gain of sulfate phases was directly correlated with the expansion rate for samples that contained gypsum and bassanite. The appearance of both ettringite and gypsum resulted in a much higher expansion.
- Despite of potentially ideal conditions, no thaumasite was found in the mortar samples after 200 days of sulfate exposure. This is believed to be related mainly to the evolution of the experimental solutions.
- The cement-reduced mortars Eco II and Eco III showed less expansion after 91 days of exposure in 30 g/l sodium sulfate solution compared to their respective reference mortars. However, the expansion rates of blast furnace cement (CEM III/B)-based mortar mixes were significantly lower than Portland cement (CEM I) and Portland slag cement (CEM II/A-S)-based mortars.
Eco-friendly concretes with low water and cement clinker content but high amounts of limestone powder are appropriate for application in environments with low sulfate concentration (XA1 according to DIN 1045-2/EN 206-1). The application of Eco III in moderate sulfate-loaded environments (XA2) is possible and a sufficient durability, even under severe conditions (XA3), is achievable with prolonged pre-curing.

REFERENCES

PHYSICAL AND CHEMICAL PROPERTIES OF CEMENTITIOUS MATERIALS UNDERGOING ACCELERATED DECALCIFICATION

Arezou Babaahmadi (1), Luping Tang (2) and Zareen Abbas (3)

(1) Division of Building Technology (Building Materials), Chalmers University of Technology, Gothenburg, Sweden, arezou.babaahmadi@chalmers.se

(2) Division of Building Technology (Building Materials), Chalmers University of Technology, Gothenburg, Sweden, tang.luping@chalmers

(3) Department of Chemistry, University of Gothenburg, Kemivägen 4, Gothenburg, Sweden, Zareen@chem.gu.se

Abstract
A major degradation scenario within safety assessment analysis of nuclear waste repositories is decalcification of cementitious materials due to long-term contact with groundwater. Understanding the changes in Physical and Chemical properties of cementitious barriers is of high importance. In order to decrease the uncertainty in the assessments due to extrapolating short term data sets, acceleration methods enhancing the decalcification process can be used. In this study with application of an accelerating electro-chemical migration method, the calcium leaching effect on cementitious specimens is studied. To account for the changes in chemical and mineralogical properties after leaching, paste specimens of size 75×50Ø mm and to study the ageing effect on physical properties, concrete specimens of size 50×1000 mm were treated with migration method. LA-ICP-MS, XRD and SEM were employed to perform chemical and mineralogical assessments and permeability and water adsorption tests methods were carried out in order to study the physical properties of the leached samples. The results indicate that total leaching of Portlandite content is expected in leached samples. Moreover, up to 15 times of higher gas permeability coefficient and 4 times higher water adsorption rate are detected in aged samples.

Keywords: Nuclear Waste Management, Calcium leaching, Acceleration method, Pore structure

1 INTRODUCTION
Within constructions of nuclear waste repositories considerable amounts of concrete as an economic construction material with structural strength and high pH buffering capacity, is
used. In Sweden the Swedish nuclear fuel and waste management company (SKB) is required
to demonstrate stability of the safety barriers for a service life of up to 100000 years. In this
time perspective, the major deterioration factor is the chemical interaction between the
groundwater and the concrete structures [1, 2]. The water, having low calcium content will
cause dissolution of the main hydration products of cementitious material, calcium hydroxide
– Portlandite, noted as \( \text{CH} = \text{Ca(OH)}_2 \), and calcium silicate hydrates, noted as CSH.
In the environment that prevails in the repository the leaching process will be extremely
slow but still inevitable. For this reason, in order to make predictions of the long term
chemical and mechanical characteristics of the concrete structures either extrapolated data
from short term experiments at natural conditions, with the risk of also extrapolating the
errors, or data from experiments where the calcium leaching has been accelerated by means of
chemical agents can be used.
There exist two categories of acceleration methods in the literature. One is application of
electrical field [3-6], supported by the fact that the charged substances can be transported with
electrical field, and another is immersion of cementitious samples in strong chemical
solutions instead of deionized water [7-10]. However, the size of the specimen that is used in
order to reach the quasi-steady state leaching is small and may be unsuitable for mechanical
and other physical tests, such as permeability and diffusivity. Therefore, more effective
ageing test methods for specimens with larger sizes are desirable.
This paper presents an electro-chemical migration method to accelerate leaching of
calcium from solid cementitious materials. Basically, the cementitious specimen as a porous
media is placed in an electrochemical cell. The electrical potential or current and the
concentrations of anolyte and catholyte solutions are optimized to reach the highest possible
leaching rate without introducing unexpected damage in the specimen due to temperature rise,
osmotic pressure or chemical attack. Paste specimens are treated to account for the chemical
and mineralogical properties of the sample after ageing as well as the comparability of the
ageing function with natural ageing. The assessments are performed by means of LA-ICP-
MS, XRD and SEM instrumental analysis. Moreover the method is applied on concrete
specimens of the size 50×100Ø mm and the effect of leaching on physical properties like
permeability or water adsorption is studied.

2 MATERIAL AND METHODS

2.1 Test Specimens
To facilitate chemical and mineralogical analysis cement paste without aggregate was
studied. The paste specimens used in this study were cast from a mixture of Swedish
structural Portland cement for civil engineering (CEM I 42.5N BV/SR/LA) and deionized
water at a water-cement ratio of 0.5. The chemical composition of the cement is listed in
Table 1. Fresh cement paste was cast in acrylic cylinders with an internal diameter of 50 mm
and a length of 250 mm. The cylinder’s ends were sealed with silicone rubber stops. In order
to produce specimens with a homogeneous composition and structure, the cylinders
containing fresh paste were rotated longitudinally at a rate of 12-14 rpm for the first 18-24
hours of hydration, after which the rubber stops were removed and the ends of the cylinders
were sealed with plastic tape. The specimens were stored for over 6 months in a moist plastic
box and then cut to cylinders with the size of Ø50×75 mm for use as specimens in the
experiments. In order to prevent carbonation, saturated lime water was used at the bottom of
the plastic box. The initial calcium and silica contents in the cement paste specimens are presented in Table 2, calculated considering C3S2H3 to be the composition of CSH.

Table 1. Chemical characteristics of Swedish CEM I 42.5N BV/SR/LA

<table>
<thead>
<tr>
<th>Chemical formulation</th>
<th>CaO</th>
<th>SiO2</th>
<th>Al2O3</th>
<th>Fe2O3</th>
<th>MgO</th>
<th>Na2O</th>
<th>K2O</th>
<th>SO3</th>
<th>Cl</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percentage</td>
<td>64</td>
<td>22.2</td>
<td>3.6</td>
<td>4.4</td>
<td>0.94</td>
<td>0.07</td>
<td>0.72</td>
<td>2.2</td>
<td>0.01</td>
</tr>
</tbody>
</table>

Table 2. Initial calcium and silica contents in a cement paste specimen (Considering C3S2H3 as the composition of CSH)

<table>
<thead>
<tr>
<th>Component</th>
<th>mole/gr paste</th>
<th>mole/specimen*</th>
<th>Ca/Si (in mole)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSH</td>
<td>0.0044</td>
<td>1.02</td>
<td>1.5</td>
</tr>
<tr>
<td>CH</td>
<td>0.003</td>
<td>0.68</td>
<td>1</td>
</tr>
<tr>
<td>Other hydrates</td>
<td>0.0018</td>
<td>0.41</td>
<td>0.6</td>
</tr>
<tr>
<td>Total</td>
<td>0.0092</td>
<td>2.11</td>
<td>3.1</td>
</tr>
<tr>
<td>Silica content</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CSH</td>
<td>0.003</td>
<td>0.68</td>
<td>-</td>
</tr>
</tbody>
</table>

In order to investigate the physical and mechanical properties: permeability and water adsorption, concrete specimens were casted in cylinders with the size of Ø 100×200 mm with two different water cement ratios (according to the properties of the concrete used in SFR) [1, 2], Table 3. The specimens were cured in saturated lime water for more than 3 month. The observations from the slump test prior to casting was 25 mm slump for the concrete with W/C=0.48 and 35 mm for W/C=0.62.

Table 3. Properties of concrete used in SFR in Forsmark

<table>
<thead>
<tr>
<th>Properties</th>
<th>Silo 1)</th>
<th>BMA 2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement type</td>
<td>Swedish structural cement</td>
<td>Swedish structural cement</td>
</tr>
<tr>
<td>W/C</td>
<td>0.48</td>
<td>0.62</td>
</tr>
<tr>
<td>Cement content kg/m³</td>
<td>350</td>
<td>300</td>
</tr>
<tr>
<td>Aggregate volume fraction³</td>
<td>0.7</td>
<td>0.7</td>
</tr>
</tbody>
</table>

2) Estimated based on the previous Swedish concrete class K30.
3) Estimated based on general mix design of concrete mix proportion, which is in agreement with Höglund, 2001 [2] for the concrete in silo.
2.2 Experimental Set-Up for Electro-Chemical Migration Method

The experimental set-up of the electrochemical migration method is inspired by the rapid chloride migration test developed by Tang, Figure 1 [4]. In this method, the specimen was placed between two electrolyte solutions providing a porous medium for ion migration. In case of using paste specimen a specimen of size Ø50×75 and concrete specimens of size Ø100×50 were used. The curved surface of the specimen was sealed using an asphalt tape which was extruded up to 2-3 times longer than specimen’s height providing an empty volume of about 200 ml as the anolyte container. A plastic box with the capacity of 30 liters was used as the catholyte container. The anode was made of a titanium mesh which was equipped with a plastic support preventing direct contact with the specimen. The cathode was made of stainless steel which was mounted on a plastic support in a similar way as described in NT BUILD 492 [5]. The cells were connected to an external potential supplier with adjustable current and potential. In order to avoid a temperature-induced mechanical destruction of the specimen due to the Joule effect the current applied to the specimen was controlled. The applied electrical field was adjusted to a constant current of 250 mA as concluded in a series of trial experiments.

As pointed out in Introduction, previous experiments for accelerated leaching of calcium from cementitious materials have relied on the application of concentrated ammonium nitrate solution increasing the dissolution rate of calcium hydrates [7]. According to Equation (1), Carde et al. [6] and Heukamp et al. [7], the application of ammonium nitrate solution enhances the dissolution of calcium hydrates due to production of Ca(NO$_3$)$_2$ which has a very high solubility.

\[
Ca(OH)_2 + 2NH_4NO_3 \rightleftharpoons Ca^{2+} + 2OH^- + 2NH_3 + 2NO_3^- \tag{1}
\]

Also in this study, ammonium nitrate solution was used as catholyte solution in order to dissolve the calcium hydroxides. However, in order to prevent the specimen from undesired aggressive decomposition of CSH at such a high concentration NH$_4$NO$_3$, a concentration of 0.3 M was chosen.
Lithium hydroxide solution was selected as anolyte solution. This was motivated by the fact that the Li$^+$ ions do not exist in the pore solution. Moreover, Li$^+$ ions with a crystallographic radius of 0.07 nm, have a high surface charge density and therefore they are strongly hydrated in water and acquire a large size [8]. The thick water layer around Li$^+$ in a solution will reduce the tendency for diffusion or migration and consequently will also reduce any competing potential in migration with calcium ions. Furthermore, with application of a hydroxide salt of lithium, the produced H$^+$ ions at the anode were neutralized and localized acidic characteristics in the anolyte solution with corrosive effects on the specimen were prevented. In this study the pH of the catholyte solution was maintained >13.

In order to maintain the pH level in the catholyte and anolyte solutions as well as to compensate for the consumed ions (OH$^-$ ions in the anolyte solution and H$^+$ ions in the catholyte solution), the solutions were frequently recharged. The quantities of the salts required for recharging were calculated according to the Faraday’s rule of electrolysis as given in Equation (2).

$$I \cdot t = F \cdot z \cdot \left( \frac{m}{M} \right)$$

where, $I$: Current (A), $t$: time (seconds), $F$: Faraday number = 96485 C/mol, $M$: Molecular mass of substance (g/mol), $m$: mass of substance (g) and $z$: the valance number of ions.

With the application of a current of 0.25 A, approximately 20 grams of ammonium nitrate and 6 grams of lithium hydroxide per specimen were needed (in powder form) to recharge the catholyte and anolyte solutions, respectively, for every 24 hours.

2.3 Instrumental Analysis

Line scans quantifying the longitudinal changes in Ca/Si ratios of solid paste samples were performed by Laser Ablation-Inductive Coupled Plasma-Mass Spectrometry (LA-ICP-MS). Laser Ablation analysis was performed using a New Wave NWR213 laser ablation system coupled to an Agilent 7500a quadrupole ICP-MS (upgraded with shield torch and a second rotary vacuum pump).

Characterization of the crystalline phases in paste samples was performed with X-Ray Diffraction (XRD) analysis. A Siemens D5000 (CuKα = 1.5418 Å) X-Ray diffractometer, equipped with Gobel mirror was used. The measurements were carried out by using 0.050° per step and at a time step of 2 s. The powder sample was prepared by crushing and grinding the solid sample in a mortar while immersed in ethanol and vacuum drying after grinding. For each analysis 0.5 grams of the powder placed on a thin-walled glass sample holder was used. The results were calibrated using 0.05% mass of Si powder as standard.

2.4 Gas Permeability and Water adsorption tests

The gas permeability and water adsorption tests were performed according to state of the art report of Rilem technical committee 189-NEC [9]. The concrete specimens treated for 5 weeks with electro chemical migration test as well as untreated concrete specimens were analysed to account for the changes in physical properties after ageing.
3 RESULTS AND DISCUSSIONS

3.1 Chemical and Mineralogical analysis

According to LA-ICP-MS analysis, as illustrated in Figure 2, the Ca:Si ratio of the aged sample is plotted as a function of axial and radial position in the specimen. As it can be seen Ca:Si profile has a considerably homogenized distribution throughout the specimen. Moreover, to demonstrate the hypothesis regarding preferential leaching of Portlandite when using migration method, XRD analyses were performed, as presented in Figure 3. It is clearly shown that the Portlandite peaks are dramatically reduced as compared to the pristine material.

As reported by Adondet and Buil [10] total dissolution of Portlandite content followed by progressive decalcification of CSH gel is expected in natural leaching process. Considering the presented results comparable leaching processes between electrochemical migration and natural leaching can be envisioned.

Figure 2. LA-ICP-MS results

Figure 3. XRD analysis results
3.2 Physical properties

Figure 4, illustrates the results according to gas permeability test. As presented, it can be seen that the coefficient of permeability is almost 15 times higher for the calcium leached specimens. Moreover, the results from water adsorption test are presented in Figure 5; showing approximately 3-4 times of higher water adsorption rate for the calcium leached specimens compared to pristine materials. All this indicates considerable changes in pore structure after ageing of cementitious materials.

Figure 4. Gas permeability test results

Figure 5. Water adsorption test results
4 CONCLUSIONS

A newly developed migration method was presented to enhance the calcium leaching of proper sized cementitious samples to be used in further physical and mechanical tests. The chemical and mineralogical properties of aged paste specimens envision total dissolution of Portlandite content and progressive decalcification of the CSH gel. The pore structure of calcium leached samples was concluded to be relatively more porous compared to pristine materials exhibiting 15 times higher gas permeability coefficient and up to 4 times higher water adsorption rate.

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REFERENCES

EVOlution of the cement mortar microstructure caused by salt crystallization and water freezing investigated by multicycle mercury intrusion porosimetry

Witold Grymin (1), Marcin Koniorczyk (1), Piotr Konca (1), Alicia Marciniak (1) and Dariusz Gawin (1)

(1) Department of Building Physics and Building Materials, Łódź University of Technology, Łódź – witold.grymin@p.lodz.pl, marcin.koniorczyk@p.lodz.pl, piotr.konca@p.lodz.pl, alicja.marciniak@p.lodz.pl, dariusz.gawin@p.lodz.pl

Abstract

The paper presents application of the Multi-cycle Mercury Intrusion Porosimetry for analyzing the evolution of the cement mortar microstructure due to the sodium sulphate crystallization and cyclic water freezing. The influence of the above mentioned degradation processes was investigated for several types of mortar, recipes of which differed with the content of an air-entraining admixture (AEA) and w/c ratio. Cumulative pore volume was measured for two cycles of intrusion-extrusion in order to calculate the contribution of ink-bottle type pores. The most severe damage caused by the sodium sulphate crystallization was observed for the mortar without any AEA addition – the pores of diameter 0.2-60μm increased their volume up to 250%. In the samples containing 0.1% and 0.2% of AEA the pores of diameter 0.6-30μm increased their volume up to 155%. After the freeze-thaw cycles, smaller damage of the material microstructure was observed also for the mortars containing AEA. Both the salt crystallization and frost-induced damage have an adverse effect on the mortar microstructure, which can be restrained by the addition of AEA.

1 INTRODUCTION

A complex pore microstructure is characteristic for the cement-based materials. The total porosity consists of the closed pores, which do not contribute significantly to the permeability of the material, and open pores. The pore network consists of larger voids, which are interconnected in a disordered way through a system of smaller pores. The size of the pores varies from nanometres (gel pores) to millimetres (air voids) [1]. This microstructure can be altered, inter alia, by development of the expansive phases, such as salt and ice crystals or the product of the alkali-aggregate reaction, which induce additional stresses in the solid skeleton.
Since the parameters characterizing the inner structure of the cement-based materials, such as porosity or permeability, determine not only their strength, but also risk of degradation of concrete due to aggressive environment, great attention was focused on their studying. One of the research methods which gives the most complete picture of the inner structure of the materials is the Multi-cycle Mercury Intrusion Porosimetry (MMIP). In the presented research study the MMIP has been used to determine influence of the salt crystallization and the ice formation on the pore size distribution and contribution of ink-bottle type pores in mortars.

2 MULTI-CYCLE MERCURY INTRUSION POROSIMETRY

MIP is a method used to characterize distribution of the pore size in the cement-based materials. It is assumed that the material consists of cylindrical pores, which are directly connected to the source of mercury. A sample is placed in a measuring chamber filled with mercury, which is a non wetting fluid. As pressure is applied on the mercury, it intrudes into the pore system. The higher the pressure is, the smaller pores can be filled with the mercury. The diameter of the pores, which are filled at a given pressure, can be calculated according to the modified Young-Laplace equation, which is commonly known as Washburn equation [2]:

\[ d = -\frac{4\gamma \cos \theta}{P}, \]

where \( d \) is cylindrical pore diameter, \( \gamma \) is the surface tension of mercury, \( \theta \) is the advancing contact angle of mercury, and \( P \) is the applied pressure of mercury.

It has to be emphasized that this method is characterized by some limitations and simplifications, which have to be taken into account during analyzing the results:

- the pores are irregular, not of cylindrical shape, what can entail in obtaining inaccurate values of the pore diameters [3, 4]. However, this inaccuracy refers to all the samples tested with this method, so a qualitative comparison between samples seems justifiable.
- the result depends on the size of the specimen [5]. In the presented study all the samples were of the same dimensions in order to exclude influence of this effect on the differences between the samples.
- majority of pores in the material are connected with the surface through a system of interconnected pores of varying size and shape. In order to fill the larger pores, which are not directly connected to the surface of the sample, with mercury, pressure corresponding to the radius of the smallest connecting pore has to be applied, what can cause not only overestimation of smaller pores and underestimation of larger pores in the specimen, but mercury entrapment in the ink-bottle type pores during extrusions as well [3, 6].

The abovementioned effect was used to determine the contribution of ink-bottle type pores, \( C_{\text{ink-bottle}} \), considering the difference between the pore volume filled during the first, \( V_{\text{por}}^{1\text{int}} \), and the second, \( V_{\text{por}}^{2\text{int}} \), cycle of intrusion, according to the equation:

\[ C_{\text{ink-bottle}}(r \geq r_0) = 1 - \frac{V_{\text{por}}^{2\text{int}}(r_0)}{V_{\text{por}}^{1\text{int}}(r_0)}. \]
3 MATERIALS AND METHOD

The cement mortars’ recipe is given in Table 1. The deterioration of their microstructure was investigated for three mortars made of CEM I 32.5R in case of the sodium sulphate crystallization (S-01, S-02, S-03) and for four mortars made of CEM I 32.5R in case of the frost-induced damage (F-01, F-02, F-05 and F-06). Content of AEA was equal to 0% (series S-01, F-01, F-02), 0.1% (series S-02, F-05) and 0.2% (series S-02, F-06). The w/c ratio was established at the level of 0.40 (series: F-02, F-05, F-06), 0.45 (series S-01, S-02, S-03) or 0.50 (series F-01) in order to estimate influence of the w/c ratio on the evolution of the pore microstructure.

Table 1: Mixture composition of the tested cement mortars (amount used to obtain 3 beams 4x4x16 cm).

<table>
<thead>
<tr>
<th>Series</th>
<th>Cement [g]</th>
<th>Sand [g]</th>
<th>Water [g]</th>
<th>AEA [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-01</td>
<td>450</td>
<td>1350</td>
<td>203</td>
<td>0.00</td>
</tr>
<tr>
<td>S-02</td>
<td>450</td>
<td>1350</td>
<td>203</td>
<td>0.45</td>
</tr>
<tr>
<td>S-03</td>
<td>450</td>
<td>1350</td>
<td>203</td>
<td>0.90</td>
</tr>
<tr>
<td>F-01</td>
<td>450</td>
<td>1350</td>
<td>225</td>
<td>0.00</td>
</tr>
<tr>
<td>F-02</td>
<td>450</td>
<td>1350</td>
<td>180</td>
<td>0.00</td>
</tr>
<tr>
<td>F-05</td>
<td>450</td>
<td>1350</td>
<td>180</td>
<td>0.45</td>
</tr>
<tr>
<td>F-06</td>
<td>450</td>
<td>1350</td>
<td>180</td>
<td>0.90</td>
</tr>
</tbody>
</table>

Specimens 4x4x16 cm were cast, stored in the water for 28 days and dried in the oven until constant mass was reached.

Afterwards, the samples described as F-01, F-02, F-05 and F-06 were subjected to repeated freeze-thaw cycles. The MIP was performed after 0, 25, 50 and 100 cycles.

Meanwhile, the specimens described as S-01, S-02 and S-03 were stored in the closed vessel with relative humidity equal to 5÷10%RH and temperature equal to 30±1°C. Then the samples were conditioned in sodium sulphate water solution. The concentration of the solution was equal to 0.25kg/kg. The samples were sealed, placed in the climatic chamber and subjected to 10 heating and cooling cycles (4 hours of cooling from 30°C to 10°C, constant temperature 10°C for 4 hours, 4 hours of warming from 10°C to 30°C, constant temperature 30°C for 4 hours), causing cyclic salt crystallization. After the cycles the specimens were put in the demineralised water at the temperature 30°C for 2 weeks, during which water was changed on a daily basis, in order to remove the salt crystals from the pore system. The MMIP was performed both for specimens before and after conditioning in sodium sulphate.

From each sample three cores, what is a recommended form of samples for the MMIP [7], of diameter equal to 8mm and height equal to 15mm were drilled, dried to a constant mass and investigated by means of multi-cycle MIP method using AutoPore IV device. The presented results of cumulative pore volume and contribution of ink-bottle type pores are the average of the results for three samples tested for each case.
4 RESULTS

4.1 Influence of frost-induced damage on the microstructure

Some main physical properties of the tested materials are presented in Table 2. Comparing the cumulative pore volume (Fig. 1) for series F-01 (w/c=0.5, AEA=0%) and series F-02 (w/c=0.4, AEA=0%) one can notice that a higher w/c ratio causes a significant increase of porosity in the material. Furthermore, the distribution of pore size alters more substantially in case of higher w/c ratio, especially for the pores of diameter greater than 10μm, what indicates more severe frost-induced damage in this pore size range. Comparison of results for series F-02 (w/c=0.4, AEA=0%), F-05 (w/c=0.4, AEA=0.1%) and F-06 (w/c=0.4, AEA=0.2%) reveals a considerable increase of porosity for the last one. The samples prepared without addition of AEA (series F-01, F-02) were destroyed before reaching 100 cycles of freezing and thawing. Increase of AEA content from 0.1% to 0.2% reduces in a great extent evolution of the pore microstructure caused by the frost-induced damage. In case of series F-06 only a
slight increase of smaller pores and reduction of larger pores can be observed. Comparing the value of the threshold diameter, below which there is a rapid mercury intrusion and above which there is a little intrusion, one can notice, that this value is larger for samples with higher w/c ratio, what is in agreement with other research studies concerning this problem, i.e. [8].

![Graphs showing the contribution of ink-bottle type pores for different series and cycles.](image)

Figure 2: Contribution of ink-bottle type pores obtained in MIP for “F-“ series before and after freeze-thaw cycles.

The contribution of the ink-bottle type pores are presented in Fig. 2. The most significant changes arising as a result of the frost-induced damage can be observed for the specimens containing AEA (series F-05 and F-06). The results for series F-05 indicate that contribution of the ink-bottle type pores increased from 55% to 70%. The tensile stress induced by the ice formation can cause not only cracks and change of the diameter of the existing pores, but also opening closed pores to the intrusion of mercury and variation of the diameters of the capillary pores leading to the larger pores, what changes relative volume of the ink-bottle type pores.
Table 2: Main material properties of the investigated mortars for “F-0x“ series after 0, 25, 50 and 100 freeze/thaw cycles.

<table>
<thead>
<tr>
<th>Material parameter</th>
<th>F-01</th>
<th>F-05</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0c.</td>
<td>25c.</td>
</tr>
<tr>
<td>Total pore area [m²/g]</td>
<td>5.94</td>
<td>8.75</td>
</tr>
<tr>
<td>Bulk density [g/ml]</td>
<td>2.03</td>
<td>2.04</td>
</tr>
<tr>
<td>Apparent density [g/ml]</td>
<td>2.51</td>
<td>2.57</td>
</tr>
<tr>
<td>Porosity [%]</td>
<td>19.04</td>
<td>20.70</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Material parameter</th>
<th>F-02</th>
<th>F-06</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0c.</td>
<td>25c.</td>
</tr>
<tr>
<td>Total pore area [m²/g]</td>
<td>5.67</td>
<td>6.01</td>
</tr>
<tr>
<td>Bulk density [g/ml]</td>
<td>2.10</td>
<td>2.20</td>
</tr>
<tr>
<td>Apparent density [g/ml]</td>
<td>2.51</td>
<td>2.56</td>
</tr>
<tr>
<td>Porosity [%]</td>
<td>16.44</td>
<td>14.11</td>
</tr>
</tbody>
</table>

4.2 Influence of sodium sulphate crystallization on the microstructure

The main parameters of the tested materials are summarized in Table 3. The results of the cumulative pore volume and the contribution of the ink-bottle type pores are presented in Fig. 3. As it can be noticed, sodium sulphate crystallization induces significant changes in the microstructure of the mortars. In the course of salt crystallization larger pores increase their volume, especially the ones of diameter 500nm up to 20μm. Addition of the AEA reduces considerably damage caused by the salt crystallization. A value of the threshold diameter, for samples after salt crystallization, shifts towards smaller pores for samples containing AEA. This might indicate reduction of damage caused by the addition of the AEA. The higher the AEA content is, the higher is the contribution of the ink-bottle type pores. One can notice that for some samples (series S-02, S-03) porosity after salt crystallization decreased. It can be caused by closure of some pores, which were previously open to mercury intrusion.

Table 3: Main material properties of the investigated mortars for “S-0x“ series before and after salt crystallization.

<table>
<thead>
<tr>
<th>Material parameter</th>
<th>S-01</th>
<th>S-02</th>
<th>S-03</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total pore area [m²/g]</td>
<td>8.98</td>
<td>8.93</td>
<td>7.43</td>
</tr>
<tr>
<td>Bulk density [g/ml]</td>
<td>2.20</td>
<td>2.10</td>
<td>2.22</td>
</tr>
<tr>
<td>Apparent density [g/ml]</td>
<td>2.53</td>
<td>2.43</td>
<td>2.61</td>
</tr>
<tr>
<td>Porosity [%]</td>
<td>13.10</td>
<td>13.80</td>
<td>14.74</td>
</tr>
</tbody>
</table>
Figure 3: Comparison of the average cumulative pore volume and contribution of ink-bottle type pores obtained by means of MIP for the “S-” series before and after sodium sulphate crystallization.
5 CONCLUSIONS

Application of the MMIP technique for analysis of the microstructure evolution caused by development of the expansive phases in mortars was presented. The MMIP test allows to obtain some data about the pore size distribution in the material, even though the pore size is underestimated. Different types of cement mortar, varying in respect of AEA content and w/c ratio, were analyzed. The higher content of AEA is, the higher is contribution of ink-bottle type pores. Both the frost-induced damage and damage caused by salt crystallization can be restrained by the use of AEA admixtures. The degradation processes influenced heavily the material strength and transport properties, as indicated the test results presented elsewhere [9].

ACKNOWLEDGEMENTS

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REFERENCES

DURABILITY PERFORMANCE OF CONCRETE WITH GLASS POWDER AS SUPPLEMENTARY CEMENTITIOUS MATERIAL

Hongjian Du and Kiang Hwee Tan

Department of Civil and Environmental Engineering, National University of Singapore, Singapore – ceedh@nus.edu.sg; tankh@nus.edu.sg

Abstract

This paper reports the experimental results on durability properties of concrete in which cement was partially replaced by recycled waste glass powder. The glass powder was finely ground to a similar particle size distribution as cement, and added at contents of 0, 15, 30, 45 and 60% by weight of the binder content. Water penetration depth, rapid chloride migration and chloride diffusion tests were carried out at 28 days to determine the durability performance. The amorphous silica in the glass could dissolve in alkaline solution and react with lime in the pore solution to form calcium silicate hydrate. This pozzolanic reaction could densify the microstructure of cement paste, particularly at the interface of aggregate particles. Therefore, the mechanical and durability properties are expected to be better provided that adequate pozzolanic reaction has reached to some extent. The test results indicate that at 28 days the pozzolanic reaction is so obvious that mixes with 15, 30 and 45% glass powder exhibited higher compressive strength than the reference mix without glass powder. The resistance of concrete to water and chloride penetration continuously increases with higher glass powder content. At 60% replacement level, the water penetration depth was reduced by 80% while the chloride migration and diffusion coefficient were reduced by 90% and 53%, respectively. Based on this study, concrete with recycled glass powder as supplementary cementitious material is sustainable because of the reduced cement content as well as the enhanced durability performance.

1 INTRODUCTION

Incorporating recycled waste glass into building materials has attracted interests due to environmental protection and solid waste management issues, as well as better material performances. Extensive research has been carried out to study the feasibility of using recycled glass as sand substitution in cement composites previously and the results obtained were encouraging as no significant mechanical degradation was found even at high replacement levels up to 100% [1-4]. At the same time, mortar or concrete containing glass sand exhibits higher resistance to chloride penetration because of the pozzolanic reaction of
finely ground glass powders [2,4]. The primary reaction of pozzolanic materials is the attack on the amorphous silica framework by OH\(^{-}\) ions, followed by the reaction with Ca\(^{2+}\) in the pore solution of cement paste and last the formation of additional calcium silicate hydrates (C-S-H). C-S-H is the most important cement hydration product since it can bind different phases in the composites and provide strength. Therefore, the pozzolanic reaction is beneficial to concrete with respect to mechanical and durability properties owning to the additionally formed C-S-H. In addition, no deleterious alkali-silica reaction would be observed provided that the glass powder is sufficiently fine, for instance 0.3 mm [5].

Since cement is the most energy and cost intensive component in concrete, it is a more sustainable and economic step to use recycled glass as cement replacement in concrete. Some pioneer researchers have conducted experiments to examine the pozzolanic activity of fine glass powders and consistently found that the finer particle size can produce higher pozzolanic activity [6,7]. However, previous research mainly focuses on the influence of pozzolanic reaction on the mechanical behaviors. Study of transport properties of harmful ions in such concretes was limited [8,9]. Schwarz et al. [8] reported that 10% glass powder is the optimum replacement level and concluded that glass powder has the potential to improve the durability based on this sole glass powder content. Carsana et al. [10] found that the glass powder can give better improvement in the mechanical and durability performances of mortar than natural pozzolans and similar effect as fly ash at 30% cement replacement. However, the effect of glass powder content on the pozzolanic reaction and durability has not been discussed. Therefore, this study will investigate the durability of concrete with glass powder as a supplementary cementitious material, at various cement replacement contents.

2 MATERIALS AND TEST METHODS

2.1 Materials

Chemical compositions of ordinary Portland cement (OPC) used in this study are listed in Table 1. The waste glass was crushed from soda-lime glass bottles, that is, beer bottles, after they were washed with tap water. The bottles were first reduced into smaller sizes by a jaw crusheer and further ground by a ball miller for 1 hour. The particle size distribution is displayed in Fig. 1, from which the mean particle size for OPC and glass powder is found to be close to each other, about 10 and 8 µm, respectively. Fig. 2 shows the scanning electron microscope (SEM) image for the glass powder, at a magnification of 2000. The specific gravities of OPC and glass powder are 3.15 and 2.65, respectively. Glass powder has a negligible water absorption capacity of 0.07%. Coarse aggregate with a maximum particle size of 10 mm was used. The fineness of sand used in this study is 2.65.

Table 1: Chemical compositions of OPC and GP (%)
2.2 Mix Proportion and Test methods

In this study, the glass powder was used as OPC substitution with contents of 15, 30, 45 and 60% of the total binder by weight. A reference concrete mix with water-cement ratio (w/c) of 0.485 was selected. The mix proportion is summarized in Table 2. For each mix, seven φ100×200 mm cylinders were cast among which three specimens were for compressive strength, two specimens were water penetration depth, and one each for chloride migration coefficient and chloride diffusion tests. All specimens were compacted on a vibration table and covered with a plastic sheet. After demolding on the next day, the specimens were cured in water until the age of 28 days. Compressive strength, water penetration depth, chloride migration coefficient and chloride diffusion was carried out according to ASTM C39, BS EN 12390-8, NT Build 443 and 492, respectively.

![Figure 1: Particle size distributions of OPC and GP](image1)

![Figure 2: SEM images of glass powder at different magnifications](image2)

3 TEST RESULTS AND DISCUSSION

3.1 Compressive strength

The effect of glass powder content on the 28-day compressive strength is shown in Fig. 3. It is clear that compressive strength increases with glass powder substitution amount until
45% owning to the pozzolanic reaction. During the pozzolanic reaction, calcium hydroxide (CH) reacts with glass powder and forms additional calcium silica hydrates: CH+S+H→C-S-H. Hence, the microstructure of the cement paste is refined, particularly for the interfacial transition zone (ITZ) between aggregate particles and cement paste. The ITZ is normally the weakest phase in concrete composites, compared to the bulk cement paste and aggregates. As a result of wall-effect, the water content at ITZ is higher and hence the microstructure is more porous compared with the bulk paste. The CH content is higher while the C-S-H amount is lesser. Under mechanical loading, crack usually initiates from and gradually propagates along ITZ, until the concrete composite fails.

For concrete with cement partially replaced by glass powder, CH at ITZ can be turned to C-S-H, leading to a denser microstructure and higher strength. Once ITZ can be enhanced, the strength of concrete is expected to be higher than plain OPC concrete. Concrete with 60% glass powder has a slightly lower strength. It is assumed that at this high OPC replacement ratio, there is insufficient CH for the pozzolanic reaction of glass powder.

Table 2: Mix proportions of concrete with different glass powder contents

<table>
<thead>
<tr>
<th>Mix No.</th>
<th>Content, kg/m³</th>
<th>Water</th>
<th>Cement</th>
<th>Glass Powder</th>
<th>Coarse aggregate</th>
<th>Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPC</td>
<td></td>
<td>185</td>
<td>380</td>
<td>0</td>
<td>825</td>
<td>960</td>
</tr>
<tr>
<td>15GP</td>
<td></td>
<td>185</td>
<td>323</td>
<td>57</td>
<td>825</td>
<td>955</td>
</tr>
<tr>
<td>30GP</td>
<td></td>
<td>185</td>
<td>266</td>
<td>114</td>
<td>825</td>
<td>947</td>
</tr>
<tr>
<td>45GP</td>
<td></td>
<td>185</td>
<td>209</td>
<td>171</td>
<td>825</td>
<td>940</td>
</tr>
<tr>
<td>60GP</td>
<td></td>
<td>185</td>
<td>152</td>
<td>228</td>
<td>825</td>
<td>933</td>
</tr>
</tbody>
</table>

Figure 3: Compressive strength of concrete with glass powder at 28 days

3.2 Water penetration depth

At 28 days, two cylinders were coated with epoxy at the top and side surfaces and subjected to a water pressure of about 0.75 MPa at the bottom surface for 7 days. After that,
the cylinders were axially splitted and the water penetration front was measured and reported as the average depth. The variation of water penetration depth into concrete with glass powder content is shown in Fig. 4. The depth of water penetration continuously decreases with higher glass powder content, up to 60%. Transport properties of normal strength concrete are governed by its component in the order of ITZ, cement paste and aggregates. In concrete with glass powder, pozzolanic reaction enhances both of the ITZ and the bulk paste, leading to a higher resistance to water penetration. Different with compressive strength, the concrete mix with 60% glass powder has the lowest water penetration depth, about 20% of that of the plain concrete. The strength is controlled by the porosity of the composites while transport properties are controlled by the pore size distribution as well. The refinement on the pore size distribution by glass powder pozzolanic reaction can further contribute to the improved durability.

![Figure 4: Water penetration depth into concrete with glass powder at 28 days](image1)

![Figure 5: Chloride migration coefficient of concrete with glass powder at 28 days](image2)
3.3 Chloride migration coefficient

Chloride migration coefficient \(D_{\text{nm}}\) was calculated from the following formula as per NT Build 492:

\[
D_{\text{nm}} = \frac{0.0239(273 + T) L}{(U - 2)t} \left( x_d - 0.0238 \sqrt{\frac{(273 + T) L x_d}{U - 2}} \right)
\]

where \(U\) is the applied voltage of 20 V (in this study); \(t\) is the testing duration of 24 hours; \(T\) is the average temperature before and after the test (30 °C); \(L\) is the specimen length (50 mm); and \(x_d\) is the chloride ingression depth as determined by spraying 0.1 M silver nitrate solution. The effect of glass powder content on the chloride migration coefficient is displayed in Fig. 5. The migration coefficient is remarkably reduced by the cement substitution with glass powder, especially with 30% content. The result agrees well with the water permeability, indicating that pozzolanic reaction has a huge improvement on the durability of concrete. Attributed to the better ITZ and cement paste, the pathways for chloride ingress are blocked. Compared to OPC, glass powder has a higher fraction of fine particles (as shown in Fig. 1), and hence a denser microstructure and higher impermeability.

3.4 Chloride diffusion coefficient

To determine the chloride diffusivity, one \(\phi 100 \times 100\) mm cylindrical specimen was immersed in a 185g/L sodium chloride solution for 28 days. All the surfaces were coated with epoxy except one end face, which was exposed to chloride solution to guarantee one direction chloride diffusion into the concrete. After the immersion, the chloride content was determined for every 10 mm concrete slices from the exposed surface. The results are shown in Fig. 6 for concrete with different glass powder content. The apparent diffusion coefficient can be obtained by best-fitting the chloride content profiles by using the following Crank’s solution to Fick’s second law:

\[
C(x,t) = C_0 \left[ 1 - \text{erf} \left( \frac{x}{2\sqrt{Dt}} \right) \right]
\]

where \(C(x,t)\) is the chloride content at depth \(x\) and immersion time \(t\); \(C_0\) is the chloride content at the surface; \(\text{erf}\) is the error function; and \(D\) is the apparent diffusion coefficient. The best-fit curves and their corresponding diffusion coefficients for each concrete mix are also shown. It is apparent that the diffusivity drops significantly with glass powder content in the concrete. This is due to the enhanced ITZ and paste matrix, and is in a good agreement with the results on water permeability and chloride migration coefficient.

3.5 XRD patterns and microstructures

XRD pattern for paste with 0, 30% and 60% glass powder substitution is shown in Fig. 7. It is clearly seen that the CH amount is reduced with glass powder content and almost diminishes for 60% replacement. This provides an evidence that the pozzolanic reaction has consumed CH partially for 30% and almost complete for 60%. The optimum glass powder substitution ratio is between 30 and 60%. The secondary C-S-H can produce a more homogenous cement paste, particularly at ITZ, as shown in Fig. 8. It is easy to find CH (plate crystals) and ettringite (in needle shape) in the paste at the OPC concrete, and large pores existing between different hydration phases. In contrast, a dense and more homogenous structure is found for concrete with 30% glass powder. The pozzolanic reaction greatly turned
crystal CH into amorphous C-S-H. Large pores are much less noticeable. This change in the chemical compositions and pore size distribution is the origin for the better mechanical and durability performances for concrete with glass powder as OPC substitution.

Figure 6: Chloride profiles and regression curves for concrete with glass powder at 28 days

Figure 7: XRD patterns of cement paste with glass powders

Figure 8: SEM images of concrete with OPC (left) and 30% glass powder (right)
4 CONCLUSIONS

This study experimentally ascertained the beneficial influence of using glass powder as supplementary cementitious materials in concrete, with respect to mechanical and durability properties. From the results obtained, it is concluded that: (a) glass powder as cement substitution can increase the 28-day concrete strength with the replacement ratio up to 45%, due to the pozzolanic reaction; (b) the bulk cement paste and ITZ are enhanced in microstructures and strength by the pozzolanic reaction; (c) calcium hydroxide can be consumed by glass powder and the formed calcium-silicate-hydrates are more homogenous and less porous; and (d) durability is more greatly improved than compressive strength because of the refinement on the pore size distribution in concrete. Concrete with 60% glass powder content shows the highest resistance to water permeability, chloride migration and diffusion. In closing, it is noted that concrete incorporating recycled glass powder as cement replacement can reduce the carbon footprint as well as provide better mechanical and durability performances.

ACKNOWLEDGEMENTS

The technical assistance of Ms. Li Wei from the Structural Engineering and Material Laboratory, National University of Singapore, in the conduct of XRD tests, is gratefully acknowledged.

REFERENCES

USE OF 2-D NANO-PARTICLE TO ENHANCE THE CONCRETE RESISTANCE TO CHLORIDE INGRESS

Hongjian Du (1), Hongchen Jacey Gao (1,2) and Sze Dai Pang (1)

(1) Department of Civil and Environmental Engineering, National University of Singapore, Singapore – ceedh@nus.edu.sg; ceegaoh@nus.edu.sg; ceepsd@nus.edu.sg
(2) Nautic Group Pte. Ltd., Singapore – jacey.ghc@gmail.com

Abstract

Experiments are carried out in this study to improve the durability of cement composites using 2-D nano-particles, namely graphene nanoplatelet (GNP). GNP was added into concrete at content of 0, 0.5%, 1.0%, 1.5, 2.0 and 2.5% by weight of cement. Compared with the commonly used 0-D and 1-D nano-particle (represented by nano-silica and carbon nanotube, respectively), this 2-D material has the advantage of barrier effect which can increase the tortuosity for the harmful agents to pass through the composites, leading to better durability performance. Chloride migration coefficient and diffusion coefficient were determined for each concrete mix, corresponding to accelerated and non-accelerated chloride penetration test, respectively. Both test results reveal that the resistance to chloride ion ingress decreases with GNP content, with the greatest resistance using 1.5% GNP where a reduction of 37% and 80% in the chloride migration coefficient and diffusion coefficient respectively were measured. Pore size distribution was probed using mercury intrusion porosimetry (MIP) and the result showed that 1.5% GNP addition can produce concrete with the lowest fraction of macropores and smallest average pore size. This supports the observations made for the chloride ingress where the concrete mix with 1.5% GNP showed the highest durability performance. However, clustering of GNP at higher contents compromises its improvement on the concrete durability due to agglomeration, resulting in formation of weak pockets and porous zones. This adverse influence might be overcome by better exfoliation and processing methods that are currently being explored.

1 INTRODUCTION

Durability is one of the most important properties of building materials, especially in severe working conditions like the marine environment where the structures are subjected to harmful agents penetration (including chloride ions and sulfate ions) and vulnerable to seawater splash in the tidal zone (accompanied with cyclic drying-and-wetting). Hence, many structures are suffering from chloride-initiated reinforcement corrosion and showing surface
deterioration [1]. Therefore, it is of great importance to increase the concrete resistance to chloride ingress. Supplementary cementitious materials are commonly used because of its advantages of low cost and modification of microstructure of concrete. Fly ash, slag and silica fume have been proven to be highly efficient in improving the impermeability of concrete, mainly due to the pozzolanic reaction [2]. Recently, there has been an interest in using nano-particles to improve the performances of cement-based composites. Ji [3] found that water penetration depth can be reduced by 45% by adding 3.6% of nano-silica (by weight of cement) in concrete. In such concrete, the interfacial transition zone (ITZ) and binding paste is denser and impermeable. Zhang and Li [4] characterized the pore size distribution and resistance to chloride of concrete with nano-SiO$_2$ and nano-TiO$_2$. The results revealed that the pore can be refined and the impermeability to chloride ingress becomes higher. However, the efficiency was decreased with higher nano-particle content exceeding 1% of the binder.

Apart from the above mentioned nano-particles, another carbon-based nano-particle in building materials is emerging recently, namely graphene nanoplatelet (GNP) [5-9]. GNP is a 2-D platelet with diameter and thickness of about several micrometers and nanometer, respectively. Owning to its low cost and higher mechanical and electrical properties, it can potentially be used in construction materials as a nano-filler. Du and Pang [8] reported that the durability of cement mortar can be significantly enhanced by the addition of GNP. The addition of 2.5% GNP can lead to a significant decrease of 64%, 70% and 31% in water penetration depth, chloride diffusion coefficient and chloride migration coefficient, respectively. The distinct improvement in the durability is attributed to the 2-D morphology and impermeability of the GNP which increase the tortuosity against water and chloride ions. Compared to mortar, the volume fraction of aggregates in concrete is much higher, generally occupying 70 to 80% in volume, which resulted in significantly different bulk properties and interfacial transition zone between aggregates and cement paste. The effect of GNP on the transport properties of concrete remains uncertain and it is investigated in this study via chloride permeation and diffusion into concrete with varying GNP contents. At the same time, the pore refinement and barrier effect provided by the addition of GNP will also be discussed.

2 MATERIALS AND TEST METHODS

2.1 Materials
The type of GNP used in this study was A3775 from Asbury Graphite Mills Inc. Its physical properties and its SEM image are shown in Table 1 and Fig. 1 respectively. Fine and coarse aggregates with maximum size of 4.75 and 19 mm respectively, were used with ASTM Type I Ordinary Portland cement (OPC). Darex Super 20, a naphthalene sulfonate based superplasticizer was used as a dispersant in this study to disperse the GNP particles in water and also to improve the workability of the concrete mixture.

2.2 Specimens and test methods
Prior to casting of concrete mixtures, GNP was ultra-sonicated with the aid of water and Darex Super 20 dispersant. This treatment aims to disperse and maintain the stability of the GNP suspension. After 2 hours of sonication in an iced bath, the suspension was slowly stirred by hand for 1 minute before pouring into the concrete mixture. The mix proportions are shown in Table 2 for concrete with varying GNP contents of 0, 0.5, 1.0, 1.5, 2.0 and 2.5%
by weight of the cement. The amount of the dispersant for each mix is also listed in Table 2, as well as the slump values. All the concrete specimens were consolidated on a vibration table and stayed in the steel molds for 24 hours. After demolding, the specimens were cured in a water tank till 28 days.

Table 1: Physical properties of GNP

<table>
<thead>
<tr>
<th>Density, $\rho$ (g/cm$^3$)</th>
<th>Surface area, $A$ (m$^2$/g)</th>
<th>Diameter, $D$ (µm)</th>
<th>Thickness, $t$ (nm)</th>
<th>Aspect ratio, $\lambda$</th>
<th>Purity, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.26</td>
<td>23.7</td>
<td>8.0</td>
<td>37</td>
<td>215</td>
<td>97.96</td>
</tr>
</tbody>
</table>

Table 2: Mix proportions of concrete (kg/m$^3$) and slump (mm)

<table>
<thead>
<tr>
<th>Mix</th>
<th>Water</th>
<th>Cement</th>
<th>GNP</th>
<th>Sand</th>
<th>Coarse aggregate</th>
<th>Darex Super 20</th>
<th>Slump</th>
</tr>
</thead>
<tbody>
<tr>
<td>NC</td>
<td>185</td>
<td>390</td>
<td>—</td>
<td>890</td>
<td>890</td>
<td>1.0</td>
<td>90</td>
</tr>
<tr>
<td>GC0.5</td>
<td>185</td>
<td>390</td>
<td>1.95</td>
<td>890</td>
<td>890</td>
<td>1.2</td>
<td>100</td>
</tr>
<tr>
<td>GC1.0</td>
<td>185</td>
<td>390</td>
<td>3.90</td>
<td>890</td>
<td>890</td>
<td>2.4</td>
<td>90</td>
</tr>
<tr>
<td>GC1.5</td>
<td>185</td>
<td>390</td>
<td>5.85</td>
<td>890</td>
<td>890</td>
<td>3.6</td>
<td>95</td>
</tr>
<tr>
<td>GC2.0</td>
<td>185</td>
<td>390</td>
<td>7.80</td>
<td>890</td>
<td>890</td>
<td>4.8</td>
<td>100</td>
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<tr>
<td>GC2.5</td>
<td>185</td>
<td>390</td>
<td>9.75</td>
<td>890</td>
<td>890</td>
<td>6.0</td>
<td>85</td>
</tr>
</tbody>
</table>

For each mix, six $\varnothing 100 \times 200$ mm cylinders were prepared for compressive and splitting tensile strengths, as well as static and dynamic elastic modulus following ASTM C39, C496, C215 and C469, respectively. Three $\varnothing 100 \times 50$ mm cylindrical discs were used for rapid chloride migration test as per NT Build 492. Three $\varnothing 100 \times 100$ mm cylinders were immersed in a salt solution (185 g NaCl per liter) for 14, 56 and 90 days. On the immersion age of 14 and 56 days, one cylinder was taken out and axially split and sprayed with 0.1M AgNO$_3$ solution on the fractured surface to determine the chloride ion penetration depth. The
remaining cylinder was used to determine the chloride content profile after 90 days of
immersion at a distance of 5, 15, 25, 35 and 35 mm from the immersed end. Total chloride
content (acid soluble) was determined according to BS 1881-124. The pore structure of the
concrete with GNP was investigated by using Mercury Intrusion Porosimetry (MIP) at 28
days. The concrete samples extracted for the MIP tests were devoid of large aggregates to
isolate the effect of GNP on the cement matrix. Two 75×75×285 mm concrete prisms were
used to determine the permeable void content, according to ASTM C642 at 28 days.

3 RESULTS AND DISCUSSION

3.1 Pore size distribution

The pore structure of concrete with GNP has been characterized by MIP and the curves for
the cumulative pore volume and the differential distribution are shown in Figs. 2(a) and (b),
respectively. The pore diameter corresponding to the peak in the differential distribution curve
represents the critical pore size in the matrix. This critical pore size characterizes the mean
size of pore entryways that allows maximum percolation throughout the pore system and
greatly influences the transport properties of harmful fluids and gases in cement-based
material. With the addition of GNP, the peaks of the differential distribution curves shift left,
indicating a drop in the critical pore size. The effect was significant even with 0.5% of GNP
and the reduction in critical pore diameter is about 20%; further addition of GNP does not
cause significantly different reduction in the critical pore diameter. The effect of GNP on the
critical pore diameter in concrete was consistent with the results for cement mortar [8].

![Figure 2: Pore size investigation with MIP (a) cumulative pore volume curves; and
(b) differential distribution curves](image)

According to Mindess et al. [9], it is the macropores (with diameter from 50 nm to 10000
nm) that can affect the transport properties of cement composites. The fraction of macropores
for each concrete mix is listed in Table 3. Concrete with 1.5% of GNP resulted in the largest
reduction in the fraction of macropores which was 30% less than that for plain concrete; this
amount of GNP is also optimal for the reduction in average and median pore diameters.
The pore structure characteristic has shown that GNP can refine the microstructure of concrete with the most significant change reported for 1.5% of GNP A3775. This refinement effect is attributed to (a) the 2D platelets spanning across larger pores which divide them into finer ones; and (b) nucleation sites for promotion of cement hydration, which can lead to a more homogenous microstructure provided that the GNP are uniformly dispersed. However, at GNP content exceeding 1.5%, the refinement effect diminished and this is due to the reduced dispersion efficiency of the sonication process when the aqueous solution became more viscous at higher concentration of GNP. The GNP agglomerated and formed porous zones inside the cement paste matrix (as shown in Fig. 3) which compromised the pore refinement effect of GNP in concrete. To the authors’ best knowledge, homogenous dispersion of a high percentage of nano-particles is one of the most challenging obstacles that limit the large scale application of nanocomposites.

Table 3: Porosity and pore size distribution of GNP reinforced concrete from MIP results

<table>
<thead>
<tr>
<th>GNP content, %</th>
<th>Total porosity, %</th>
<th>Permeable void content, %</th>
<th>Average pore diameter, nm</th>
<th>Median pore diameter, nm</th>
<th>Critical pore diameter, nm</th>
<th>Fraction of macropores, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>18.4</td>
<td>12.0</td>
<td>17.3</td>
<td>47.8</td>
<td>73.8</td>
<td>45.4</td>
</tr>
<tr>
<td>0.5</td>
<td>16.2</td>
<td>12.1</td>
<td>15.0</td>
<td>36.4</td>
<td>59.9</td>
<td>38.2</td>
</tr>
<tr>
<td>1.0</td>
<td>18.1</td>
<td>11.8</td>
<td>14.6</td>
<td>35.4</td>
<td>59.8</td>
<td>36.2</td>
</tr>
<tr>
<td>1.5</td>
<td>15.8</td>
<td>12.2</td>
<td>13.8</td>
<td>27.2</td>
<td>59.7</td>
<td>31.3</td>
</tr>
<tr>
<td>2.0</td>
<td>20.2</td>
<td>11.9</td>
<td>18.8</td>
<td>43.6</td>
<td>47.7</td>
<td>39.6</td>
</tr>
<tr>
<td>2.5</td>
<td>16.5</td>
<td>11.4</td>
<td>15.6</td>
<td>37.2</td>
<td>51.7</td>
<td>36.0</td>
</tr>
</tbody>
</table>

Figure 3: SEM image: dispersion of GNP (left) and clustering of GNP (right) in concrete

3.2 Mechanical properties

The strength and modulus of concrete with different content of GNP are shown in Figs. 4(a) and (b), respectively. The results show that the nano-scale GNP does not provide any advantage nor has any adverse effect on the mechanical properties that were tested and this can be expected since the porosity of concrete is not much affected by the GNP.
3.3 Rapid chloride migration

The chloride migration coefficients for concrete containing various GNP contents are shown in Fig. 5. It is obvious that the addition of GNP could reduce the chloride migration coefficient. Concrete with 1.5% GNP exhibited the highest reduction of 37% compared to the normal concrete. The lowering of the permeability of the concrete by the addition of GNP could be attributed to the refinement in pore distribution in cement matrix and in the interfacial transition zone. Although the influence of the refined pore structure on the mechanical strength was marginal, it contributed significantly to the improved transport properties. At the same time, the GNP can act as impermeable barriers inside the cement paste matrix, which increase the tortuosity and disrupt the pore connectivity for ingressive ions to penetrate.

When GNP exceeded 1.5%, the nanoplatelets would start to cluster and the sonication will be inadequate in dispersing the GNP in the viscous suspension; the energy transfer in the viscous suspension will be lowered and that will be insufficient to disperse the GNP uniformly. Further attempts should be carried out towards achieving better dispersion of GNP particles at higher content.
3.4 Chloride diffusivity

Fig. 6a shows the chloride ion penetration depth at 14 and 56 days. The penetration depth for all GNP content increased with time from 14 to 56 days. The effect of GNP has the greatest reduction on the chloride diffusivity at 1.5% of GNP, which is consistent with the effect of GNP on chloride migration coefficient. This again can be explained by the pore refinement and obstruction of pathway for the diffusion of chloride ion into concrete; tortuosity accounts for approximately up to 40% of reduction in chloride diffusivity while the remaining reduction can be attributed to pore refinement [9]. Exceeding 1.5% of GNP, the GNP agglomerated which led to an effective aspect ratio estimated to be half of the value given in Table 1 [9]. The loose stacking of the GNP agglomerates created weak and porous zones which are reflected in the mechanical strength and chloride diffusivity.

Figure 6: (a) Chloride ingress depth into concrete with GNP; and (b) Chloride content profiles and best-fitted curves

Table 4: Parameters for the best-fitted curves of chloride content profiles

<table>
<thead>
<tr>
<th>Mix</th>
<th>$D_c$, $\times 10^{-12}$ m$^2$/s</th>
<th>$C_0$, wt. %</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>NC</td>
<td>49.7</td>
<td>0.43</td>
<td>0.981</td>
</tr>
<tr>
<td>GC0.5</td>
<td>38.7</td>
<td>0.74</td>
<td>0.987</td>
</tr>
<tr>
<td>GC1.0</td>
<td>42.6</td>
<td>0.53</td>
<td>0.963</td>
</tr>
<tr>
<td>GC1.5</td>
<td>10.0</td>
<td>0.30</td>
<td>0.985</td>
</tr>
<tr>
<td>GC2.0</td>
<td>21.5</td>
<td>0.51</td>
<td>0.975</td>
</tr>
<tr>
<td>GC2.5</td>
<td>31.4</td>
<td>0.59</td>
<td>0.946</td>
</tr>
</tbody>
</table>

The chloride content profiles for GNP reinforced concrete are shown in Fig. 6b. The diffusion of chloride ions into concrete can be described by Crank’s solution to Fick’s second law [9]. The best-fitted curves and the parameters for each fitted curve are displayed in Fig. 6b and Table 4, respectively. The chloride diffusion coefficient decreases with increasing GNP content of up to 1.5% and increases thereafter. Consistent with the result for rapid chloride migration and chloride diffusivity, a GNP content greater than 1.5% compromises the effectiveness of the GNP in reducing the transport of the chloride ions.
CONCLUSIONS

This study is the first investigation on the ingress of chloride ions into concrete which has been added with GNP, a 2-D nanoplatelet. From the experiments, it is clear that GNP can significantly reduce the permeability and diffusivity of chloride due to its refinement on capillary pores in concrete and its 2-D barrier effect. This improvement in durability is most distinct with 1.5% of GNP; beyond this content, clustering of nano-particles may occur and reduces their effectiveness in improving the durability of the concrete.

ACKNOWLEDGEMENTS

The GNP supplied by Asbury Graphite Mills, USA, is deeply appreciated.

REFERENCES


CHARACTERISTICS OF IN-SERVICE, 30-YEAR-OLD EPDM ROOF MEMBRANE

Thomas W. Hutchinson(1), Ken Trischuk(2), Ralph M. Paroli (2)

(1) Hutchinson Design Group, Ltd., 232 E. Main St., Barrington, Illinois, USA 60010 – hutch@hutchinsondesigngroup.com

(2) National Research Council of Canada, 1200 Montreal Road, Ottawa, ON Canada K1A 0R6

Abstract: The physical and mechanical properties of EPDM roof membranes obtained from three 30-year old, in-service roofs in the USA were investigated. The physical and mechanical properties evaluated (using relevant ASTM Standards) were: overall thickness, tear resistance, tensile set, tensile strength and elongation, as well as water absorption. These properties were studied for as received and after heat conditioning for up to 1500 hours at 116 C (240 F). Results showing how these membranes performed before and after heat conditioning are presented with the intent of defining characteristics for long-term service life of roof membranes.

Keywords: EPDM, Roof Membrane, Service Life, Durability

1 INTRODUCTION

Ethylene-propylene-diene terpolymer (EPDM) has been used in waterproofing and roof applications for over 45 years in North America. Introduced into the roofing market in the 1960’s, EPDM grew, especially after the oil embargo of the 1970’s, to be a roofing membrane choice for architects, specifiers, and contractors for both new construction and roofing replacement projects. EPDM has achieved long-term in situ performance in part to its chemical structure, mostly carbon black, which resists ozone and material composition, as well as degradation due to ultraviolet light, the number one degradation element to roofing materials exposed to the sun (see Photo 1). The carbon black also provides reinforcement, yielding improved physical and mechanical properties.

Long-term performance of roof cover material is dependent upon its resistance to the combined effects of ponding water, UV radiation, ozone, heat, and thermal cycling. Geographical location can exacerbate or reduce the impact of climatic factors. In ballasted systems, the ballast acts to provide protection from the UV rays, and minimizes the effect of climatic influences.

1.1 Past Studies of In Situ EPDM Performance

Durability and long-term service life is key to achieving sustainability, and many EPDM roof systems are entering their forth decade of service. This long-term service life was predicted by several studies.

Beech Study (Beech, 1991): In 1991, John Beech of the U.K. based Building Research Establishment (BRE) studied the aged and un-aged properties of poly[vinyl chloride] (PVC), polyisobutylene (PIB), chlorosulphonated polyethylene (CSM) and EPDM, focusing on flexural fatigue as a precursor to performance. The samples were heat-aged and subject to...
Characteristics of in-Service, 30-Year-Old EPDM Roof Membrane

flexural cycles. While the other membranes failed in less than 140,000 cycles, the EPDM never failed. Additionally, samples were subjected to exterior weather conditions in various climatic conditions: mild and wet, hot and humid, hot and dry, for four years. These samples were then subjected to flexural cycling. EPDM withstood 500,000 cycles and the testing was stopped. Beech concluded that based on flexural cycling fatigue testing, the EPDM had the best chance of being a long-term performer.

Photo 1: This ballasted 1.1 mm 45-mil EPDM roof system has been in service for 32 years

Gish Study (Gish & Lusardi, 1991): At the 1991 International Symposium on Roofing Technology in Montreal, Canada, Gish and Lusardi tested samples of 45 in situ EPDM samples cut from various roof system types ranging from five to 17 years of age. All test results met the ASTM International D4637 Standard Specification for EPDM Sheet Used in Single-ply Roof Membrane. Nearly 90% of the samples exceeded the requirements for new membrane.

Hoff Study (Hoff, 1992) and (Hoff, RCI Interface, 1998): Hoff evaluated the performance of aged EPDM membranes through the examination of manufacturer warranty records. Studying warranty repair costs from 1982 to 1993. It was found that the repair costs decreased 60% from 1982 to 1993, which were attributed to advances in several roofing system component technologies, such as the introduction of new adhesives, seam tapes and the introduction of perimeter anchor strips.

Trial Study (Trial, 2004): Trial, in 2003, undertook a study similar to the Gish study, but with an emphasis on membranes that had increased in situ periods: 16 to 23 years. The samples were taken from nine states in the USA and from both ballasted and fully adhered systems. The samples were taken from projects that represented two EPDM manufacturers. The samples were sent for testing. The samples were tested for tensile strength, elongation, tear resistance and weathering. The physical properties of all the samples exceeded the ASTM D4637 specification for new and heat aged EPDM. The conclusion was that based on its weathering properties, EPDM was expected to have long-term performance.

A study by SKZ (Süddeutsche Kunststoff Zentrum) in Germany (SKZ (Süddeutsche Kunststoff Zentrum), 2009) studied and tested EPDM and concluded that one could expect
EPDM roof membrane to last 50 years. With the advent of Life Cycle Analysis (LCA) and their emphasis on service life, this is an important question. Additionally, the design and building industry has an increased awareness about sustainable roof systems and the importance of longer lasting materials (see Photo 2).

1.2 Current Study

In 2010, the EPDM Roofing Association (ERA) was determined to answer the question of “How long can an EPDM roof perform”, and embarked on a long-term service life study themselves. Three specific goals were determined:

1. Verify the long-term performance characteristics of EPDM membranes over 30 years.1
2. Scientifically validate the empirical sustainability experiences.
3. Create a foundation for specifier-to-owner discussions concerning long-term service life.

Five roofs, four ballasted and one fully adhered, across the Midwest USA, with in situ service lives approaching or over 30 years were identified, owners contacted, and samples taken. All roofs were fully performing without moisture intrusion.

The samples were sent for testing per ASTM D4637 for:
- Elongation
- Tensile Strength
- Thickness
- Factory Seam Strength (psi)

---

1 At the time of the study, the only in situ membranes that were around for 30 years were 1.1 mm (45-mil) EPDM membranes. Currently 1.5 mm (60-mil) and 2.3 mm (90-mil) are the standard choice. It is assumed that results for the 1.1 mm (45-mil) material can be prorated for the thicker membrane.
The test results revealed that the physical properties of the membrane in the field for 30 years had physical characteristics in aggregate that were within 8% of that required for brand new material, and all exceeded the requirements for aged membrane.

While the test results proved the positive, that in situ 30-year-old EPDM membrane is performing (has good physical characteristics); the question remained, how long can they last?

Thus, the second phase of testing was undertaken. Additional samples were taken and sent to the National Research Council of Canada (NRC). The protocol this time was to take the 30 year samples and heat-age them for 500 hours at 116 °C (240°F). After heat aging the samples for 500 hours, the samples were to be tested for their physical characteristics. Furthermore, the remaining material was to be placed back in the oven and heat aged another 1000 hours or 30 years for an in situ/heat aged sample of 60 years, and then have the physical properties tested again. For comparison sake, new 1.1 mm (45-mil) EPDM membranes from a roofing contractor’s warehouse were sent for testing as new material and with heat aging of 10 and 30-year equivalency.

2. EXPERIMENTAL METHOD

The material was received in rolls and measured approximately 1200 mm x 1400 mm (47” x 55”). Each material was cut into four (4) pieces and placed in an oven at 116 C (240 F) up to 1500 hours as per ASTM D573.

2.1 Mechanical Properties

Thickness, Sheet Overall - The overall sheet thickness of the EPDM roofing membranes was measured using ASTM D412.

Water Absorption data were obtained using ASTM D471 with samples placed at 70 C for 166 hours.

Tensile Properties - Tensile strength / Ultimate Elongation data were obtained using ASTM D412, Die C and ASTM D751, Grab Method. The specimens were tested in a universal testing machine at a constant crosshead speed of 5 ± 0.2 mm/s (12 ± 0.5 in./min). The gauge length of the specimens was 75 mm (3 in.). A minimum of five specimens was tested in both machine (MD) and cross (XD) directions except for some samples that had limited material.

3. RESULTS AND DISCUSSION

Table 1 No heat aging (as received from the field)

<table>
<thead>
<tr>
<th>Sample</th>
<th>Material Direction</th>
<th>Tensile strength (MPa)</th>
<th>Elongation (%)</th>
<th>Overall Thickness (mm)</th>
<th>Water absorption (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>M</td>
<td>13.5</td>
<td>514.3</td>
<td>0.86</td>
<td>2.47</td>
</tr>
<tr>
<td>F</td>
<td>M</td>
<td>9.5</td>
<td>402.8</td>
<td>0.95</td>
<td>2.13</td>
</tr>
<tr>
<td>6</td>
<td>M</td>
<td>15.1</td>
<td>313.7</td>
<td>0.82</td>
<td>3.59</td>
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</tbody>
</table>

2 500 hours is estimated to be equal to approximately 10 years of in field exposure.
Table 2  500 hours of heat aging

<table>
<thead>
<tr>
<th>Material</th>
<th>Material Direction</th>
<th>Tensile strength (MPa)</th>
<th>Elongation (%)</th>
<th>Overall Thickness (mm)</th>
<th>Water absorption (%)</th>
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<tbody>
<tr>
<td>3</td>
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<td>11.1</td>
<td>145.1</td>
<td>1.04</td>
<td>1.81</td>
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<tr>
<td>4</td>
<td>M</td>
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<td>56.9</td>
<td>1.01</td>
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<td>5</td>
<td>M</td>
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<td>9.2</td>
<td>222.4</td>
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Table 3  1500 hours of heat aging

<table>
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<th>Elongation (%)</th>
<th>Overall Thickness (mm)</th>
<th>Water absorption (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>M</td>
<td>10.0</td>
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<td>1.10</td>
<td>1.51</td>
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<tr>
<td>4</td>
<td>M</td>
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<td>48.6</td>
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<td>M</td>
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<td>2.94</td>
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<td>M</td>
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<td>180.5</td>
<td>1.04</td>
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<tr>
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<td>214.6</td>
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</tr>
<tr>
<td>F</td>
<td>M</td>
<td>9.4</td>
<td>225.7</td>
<td>0.99</td>
<td>1.99</td>
</tr>
</tbody>
</table>

3.1  Thickness, sheet overall

The overall sheet thickness of the samples measured by both methods ranged from 0.82 – 1.40 mm. The minimum requirement is of 1.016 mm (0.040 in.) as specified in ASTM D4637/4637M for new EPDM membranes. The overall sheet thickness change after exposure was expected since they are 30 year samples.

3.2  Water Absorption

As shown in Tables 1 to 3, the water absorption was well below the +8/-2% level set in ASTM D4637/4637M This indicated that the samples were resistant to water absorption.

3.3  Tensile test

The mechanical response of the samples under tensile loading was measured and displayed as force-displacement curves. They varied with test direction and exposure.

Samples were tested before and after exposure. The tensile strengths of the samples were 8.2 – 15.5 MPa MD. The majority were above the ASTM specification of 9.0 MPa. The tensile elongations at break of the field samples prior to heat aging are 313 – 514% and 287 – 506 % for MD and XD, which exceeded the ASTM specification of 300% for new material and 200% after heat conditioning of new material. This shows that the material maintained its
flexibility after 30 years in the field. Further heat conditioning led to a reduced elongation in the membranes.

A decrease in elongation associated with an increase in tensile strength can indicate that a change in the mechanical properties of a material has taken place as a direct result of being exposed to the elements. Solar radiation, rain, temperature change or air pollution may all affect a membrane. However, if the material degradation was quite severe, both tensile strength and elongation value should decrease. As can be observed from the data, the values for most of the membranes decreased after heat conditioning of the 30-year old field samples. It is possible that at this point, processing oils, for example were being lost due to the heat-conditioning leading to the changes in tensile properties.

4. CONCLUSIONS

It is obvious from the data that the heat conditioning of 500 hours and 1500 hours affected the mechanical properties of 30-year old membrane samples. However, it must be remembered that these samples were already in the field for 30 years. Sample F showed that even after 1500 hours of heat conditioning at 116°C, the membrane was able to meet the ASTM specification for mechanical properties (8.3 MPa for tensile strength and 200% for elongation).

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4. ASTM D1204-02 Standard Test Method for Linear Dimensional Changes of Nonrigid Thermoplastic Sheeting or Film at Elevated Temperature
EVALUATION OF CLUSTERED DISTRIBUTION OF SUPERABSORBENT POLYMERS AND ITS RELATION TO AUTOGENOUS SHRINKAGE BEHAVIOUR OF INTERNALLY CURED MORTARS

Koichiro Yokota (1) and Shin-ichi Igarashi (2)

(1)Department of Civil and Environmental Engineering, Kanazawa University, Japan, Kanazawa – koichiro.yokota@gmail.com

(2)Institute of Science and Technology, Department of Civil and Environmental Engineering, Kanazawa University, Japan, Kanazawa – igarashi@se.kanazawa-u.ac.jp

Abstract
Spatial distribution of superabsorbent polymers (SAP) particles was evaluated using point process statistics. Presence of aggregate particles was treated by thinning points of SAP from a basic process. K function of SAP in the thinned point process was compared with that in actual mortars. SAP in the mortars clustered more than in the thinned process. Aggregate had a certain effect to promote clustering of SAP particles. The clustering tendency in the mortars depended on sizes of SAP particles. Differences in autogenous shrinkage behaviors of the mortars can be related to inhomogeneous supply of internal water from the clustered SAP at early ages.

1 INTRODUCTION
Several methods have been proposed to prevent autogenous shrinkage and to reduce the risk of early age cracking, which could have significant effects on the long-term performance in high strength concretes with low water/cement ratios [1]. One of the effective ways is to cure the concrete internally, using prewetted absorptive materials which contain moisture. Generally, lightweight aggregate and superabsorbent polymers (SAP) are considered as promising materials for the internal curing [2].

In order to determine mix proportions of internally cured concretes, equations to estimate the amount of internal curing materials have been used [3,4]. The materials are expected to maintain the entire cross section of concrete supplied with moisture. Thus they should properly disperse to cover the whole cross section within distances of water movement from them.

If spatial distributions are compared between prewetted lightweight aggregate and SAP particles, there is a marked difference in their ways of dispersing. Lightweight aggregate particles can take their locations freely so that they disperse randomly in 3D space. However,
SAP particles are allowed to exist only in cement paste matrix between aggregate particles. In other words, SAP particles are hindered from randomly dispersing. Therefore, SAP may be less efficient for spatial coverage with its internal water although its absorbing capacity is much greater than lightweight aggregate.

In this study, spatial distribution of SAP particles in mortars and cement pastes was evaluated using point process statistics. Distribution of SAP in mortars was also simulated as a thinned point pattern, in which volume fractions of aggregate were used to delete points from basic patterns in the corresponding cement pastes. Differences in clustering of points between the actual and the thinned point patterns were described. Significance of the clustering in the actual mortars was examined by Monte Carlo tests. Effects of the clustered distributions of SAP on autogenous shrinkage of the mortars were also discussed.

2 EXPERIMENTAL

2.1 Materials and mix proportion of cement pastes and mortars

The cement used was an ordinary Portland cement with a Blaine fineness value of 3310 cm$^2$/g. A commercial product of silica fume was used. Its density and specific surface area were 2.20 g/cm$^3$ and 20 m$^2$/g, respectively. The fine aggregate used was siliceous sand of which density and absorption were 2.62 g/cm$^3$ and 0.40%, respectively. Cement paste and mortar specimens with a water-to-binder ratio of 0.28 were produced in accordance with Japanese Industrial Standard (JIS) R 5201. The sand to binder ratio of the mortar was 1.72. Replacement ratio of silica fume was 9.4% by mass of cement. A polycarboxylic acid type superplasticizer was used. Two types of SAP (A and B) were used. SAP-A is produced by aqueous polymerization while SAP-B is obtained by inverse suspension polymerization. As shown in Fig. 1, SAP-A is irregular in shape and contains greater particles. SAP-B is almost a monosized spherical powder. Their absorption capacities are 10.0 g/g and 13.3 g/g of dry mass, respectively. The SAPs were sieved to separate into two sizes. These properties of SAPs are summarized in Table 1. Prism specimens of 40 x 40 x 160 mm were produced in accordance with JIS R 5201. They were demolded at 24 h after casting, and then cured in water at 20°C until 14d.

2.2 Image acquisition

At the prescribed age, slices about 10 mm thick were cut from the prism specimens. Their cross sections were carefully polished with abrasive papers. Colour images of the polished cross sections were acquired with a flatbed scanner. Resolution of the images was 1200 dpi (21.2 µm/pixel). In order to segment cement paste matrix from the colour image, the cross sections were dyed by spraying a solution of phenolphthalein. On the other hand, in order to segment surface voids such as pores and SAP particles, the cross sections were painted with a black ink. Then a fine white powder was put into the voids on the surfaces. The excess powder on the surfaces was removed to make the surface voids white. The black and white cross sections were

![Fig.1 SAP particles at a dry state: (a) SAP-A (b) SAP-B](image)

Table 1 Properties of SAP

<table>
<thead>
<tr>
<th>SAP</th>
<th>Absorption (g/g)</th>
<th>Particle diameter range(µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AL</td>
<td>10.0</td>
<td>300~600</td>
</tr>
<tr>
<td>AS</td>
<td></td>
<td>150~300</td>
</tr>
<tr>
<td>BL</td>
<td>13.3</td>
<td>300~600</td>
</tr>
<tr>
<td>BS</td>
<td></td>
<td>150~300</td>
</tr>
</tbody>
</table>
scanned again to obtain binary images segmented for the voids. The original colour images, the pink colour images for cement paste matrix, and the binary images for the voids were imposed with care for their coordinates and positions (Fig. 2(a)). The imposed image was used for segmenting two phases of aggregate particles and cement paste matrix around them [5].

2.3 Segmentation of SAP particles from the imposed images
Aggregate particles and the surface voids were segmented by the threshold values of brightness and RGB components (Fig. 2(b), (c)). In Fig. 2(c), both of capillary voids and SAP are segmented together. It is difficult to distinguish SAP particles individually in Fig. 2(c). For this reason, it was assumed that the incorporation of SAP did not change capillary pore size distribution in the surrounding cement paste matrix. Particles that could be SAP were statistically determined by the following procedure. At first, mortar specimens without SAP were prepared. Segmentation of capillary pores and other voids was made for the specimens without SAP. The voids and their size distribution were determined by conventional image analysis technique. Then voids corresponding to the volume fractions for each class in sizes were randomly deleted from the binary images of mortars with SAP (Fig. 2(c)).

2.4 Evaluation of spatial structure of SAP
(1) Characterization of an area of cement paste matrix as a random field
A cement paste matrix in a mortar is considered as a complementary set of aggregate particles. If it is assumed that aggregate particles are distributed randomly throughout a mortar section, the cement paste matrix surrounding the aggregate is considered to occupy a space as a random field. In order to characterize the random field of cement paste matrix, covariance function for the field was calculated. When a cement paste matrix is considered as a spatial set Ξ, its covariance function \( C(r) \) is given by eq.(1).

\[
C(r) = \Pr(\mathbf{x}_1 \in \Xi, \mathbf{x}_2 \in \Xi)
\]  

(1)

Where \( \mathbf{x}_i \) (i=1,2) is a position vector of \( \|\mathbf{x}_2 - \mathbf{x}_1\| = r \). To calculate the covariance function, a template with eight radial directions was placed on a binary image, in which a cement paste matrix phase was segmented. Whether both the centre and the endpoint fell in the cement paste matrix phase or not, was determined. This operation was repeated at different positions to obtain 10,000 samples for a distance of r.

(2) K- and L-functions of SAP [6]
SAP particles identified in images were converted to points dispersed in an observation window W. The coordinates of each point \( \{\mathbf{x}_i; i = 1,2, \cdots, n\} \) coincided with...

Fig.2 Processed images of a cross section of mortar:
(a) Pseudo-colour image
(b) Segmentation of aggregate
(c) Segmentation of voids
the gravity centres of the particles. The point intensity $\lambda$ was determined from the images by the following equation.

$$\lambda = \frac{N(W)}{A(W)}$$

(2)

$N(W)$: the total number of points in the window $W$

$A(W)$: the area of $W$

K-function ($K(r)$) was calculated from the point pattern using eq.(3).

$$K(r) = \frac{1}{\lambda^2} \sum_{i=1}^{n} \sum_{j=1}^{n} \frac{1_{0,\gamma_W(|x_j-x_i|)}}{\gamma_W(|x_j-x_i|)}$$

(3)

Where $r$ is distance, and $\gamma_W(r)$ is the isotropized set covariance function of the window $W$. It is given as the mean area of the intersection of $W$ with the $W$ shifted by a random vector of length $r$. The calculated K function was then converted to L-function ($L(r) = \sqrt{K(r)/\pi}$).

(3) Thinning SAP particles in the basic process and K-function for the thinned process

Spatial distribution of SAP in cement paste specimens was used as a basic point process $X_b = \{x_i | i = 1,2, \cdots n\}$. The K-function $K_b$ for $X_b$ was calculated. Distribution of SAP in actual mortar specimens was also considered as a point process $X_p$. If the point process $X_p$ is obtained as a result of thinning operation for $X_b$, then $X_p = X_b \cap \Xi$. The K function of SAP in the thinned process, $K_p$ is then calculated by eq.(4).

$$K_p(r) = \frac{1}{p^2} \int_0^r C(x) dK_b(x)$$

(4)

Where $p$ is the volume fraction of cement paste matrix in mortar.

(4) Simulation of point patterns as a binominal process

In order to test significance of variation in K-function obtained for the actual mortars, fifty random point patterns were simulated as a binominal point process with the same point intensity. The L-function and its 95% confidence interval for the simulated patterns were calculated.

2.5 Length change test

Length change of mortars was measured in accordance with ASTM C1698-09. In a preliminary test, the initial setting time was determined using a cement
paste with the same w/c as the mortar. The initial setting time was used as the origin of length change [7].

2.6 Absorption and particle size distribution of SAP

Water absorption capacity of SAP was evaluated in accordance with JIS K 7223 (i.e. a tea-bag method). Saturated solution of calcium hydroxide was used instead of deionized water to be absorbed in the test.

A small portion of dry SAP powder was dispersed on a piece of black paper. Micrographs of the SAP were taken at the magnification of 50x. Particle size distribution of the SAP was determined by the image analysis.

3 RESULTS AND DISCUSSION

3.1 Clustering of SAP particles due to the presence of aggregate

Covariance functions to characterize random areas of cement paste matrix in mortars are shown in Fig.3. Regardless of types and sizes of SAP, the same functions were obtained for those mortars. The correlation distance in which the function is greater than its asymptotic values is about 1mm for all the mortars. This means that all the cement paste matrices in the mortars had the same statistical structures in terms of spatial randomness.

$K_b$ functions of SAP particles in cement paste specimens are shown in Fig. 4. The $K_b$ functions for different SAPs are compared to one for the Poisson distribution as a null hypothesis. As clearly seen in Fig.4, all the $K_b$ functions are almost the same as that for the Poisson distribution. Therefore, it is reasonable to assume that the SAP particles disperse randomly in the cement paste specimens, regardless of types and sizes of SAP.

Fig. 5 shows comparison of $K$ functions for different SAPs. If actual distribution of SAP in mortar could be a realization of processes thinned from a basic process $X_b$, the $K$ function in the mortar should be close to $K_p$. When $K_p$ is compared with $K_b$, it is found that the thinning for aggregate does not greatly affect distribution of SAP. The initial randomness in cement paste specimens is still maintained in the thinned point patterns. However, SAP particles in the actual mortars were clustered more than the distribution represented by the $K_p$ function. This fact suggests that aggregate particles controlled distribution of SAP more than simply reducing room for SAP. Furthermore, distances in which clustering of SAP was observed depended on the sizes of SAP. When the large SAP of SAP-AL was used, clustering was remarkable in the range greater than 1mm. On the other hand, when the small SAPs of SAP-AS and SAP-BS were used, clustering was observed in the range of short distances. The clustering tendency of SAP-AS and -BS decreased with the increase in distance. At long distances, they followed the Poisson process. As shown in Fig. 3, there were no differences in spatial structure of cement

![Fig.5 Comparison of K-function for points of SAP in thinned and actual processes](image-url)
paste matrix between the mixtures. Therefore, room where SAP can disperse is the same between the mixtures. When SAP particles are small, more particles can exist at a given spacing between aggregate particles. Large SAP particles cannot get closer because of its own sizes of particles. As a result of those geometrical confinement, clustering of small SAP was observed at small distances. However, as for the large SAP-BL, its clustering tendency is different from other particles. Clustering of SAP-BL particles is seen at small distances. Particle size distributions of dry SAPs used in this study are shown in Fig. 6. SAP-A has a wide range of particle size distribution. Most SAP-B particles, however, fall in a narrow range smaller than 0.5mm. SAP-BL is obviously finer than SAP-AL. SAP-B may have more small particles than SAP-A even after sieving dry SAP particles. Therefore, the clustering of SAP-BL can take place at short distances. It is found from these figures that smaller particles are easy to cluster together within the correlation distances of cement paste matrix (Fig. 3).

3.2 Simulation for distribution of SAP in mortar

As shown in Fig. 5, SAP particles in the mortars have a clustering tendency and aggregate particles give further confinement to the distribution of SAP than simply restricting locations of SAP. In order to evaluate significance of clustering in the actual mortars, L-function of the SAP particles in the mortars was compared with that of random point patterns simulated as a binominal point process. L-function and the confidence intervals obtained for the simulated processes are shown in Fig. 7. The interval for large SAP particles (i.e. SAP-AL) is wider than that for small SAP particles (i.e. SAP-AS). When SAP-AL is used, the L-function touches the upper limit in the range between 1.0 and 1.5mm. The L-function for SAP-AS does not lie in the interval at the range between 0.5 and 1.0mm. Therefore, the null hypothesis of Poisson process is rejected for the pattern of SAP-AS. The same tendency as the clustering of SAP-A is also found in SAP-B. The null hypothesis is also rejected for SAP-BS. Therefore, clustering tendencies shown in Fig. 5, especially in the distributions for small SAPs, may not be a realization of random distribution. Cement paste matrix is a complementary set of aggregate, of which spatial location is a random set. However, the complementary area has local room to which small SAP particles are easily confined within the correlation distances. This results in the different clustering distances between SAPs.

Fig.7 L-function of SAP in mortars and confidence intervals of comparable binominal processes
3.3 Effects of sizes of SAP on reduction in autogenous shrinkage

Autogenous shrinkage behaviours of mortars using each SAP are shown in Fig. 8. The large particles of SAP-AL and SAP-BL are more effective in reducing autogenous shrinkage than the small particles of SAP-AS and SAP-BS. The mortars with the large SAPs exhibited greater initial expansion than the mortars with the small SAPs. Periods of the expansion continued longer when the large SAPs were used. Differences in the initial expansion were reflected to subsequent differences in shrinkage at longer ages.

Autogenous shrinkage behaviours of mortars using several types of SAP should be discussed in relation with time when the internal water is released from the SAP. Most of the SAP produced as internal curing materials release the absorbed alkaline water to the surrounding cement matrix at very early ages [8]. This phenomenon occurs irrespective of self-desiccation of the surrounding matrix. It may be an intrinsic property of SAP [8,9]. Fig.9 shows changes in absorptivity of SAPs with time of immersion. All the SAPs exhibited their maximum absorption capacity when they were dipped into saturated calcium hydroxide solution for ten minutes. However, when they were immersed into the solution for a longer time, their absorption decreased quickly. The absorption capacity at one hour is less than a half of the initial capacity for all the SAPs. The smaller SAP, the earlier decrease in absorptivity was observed. Therefore, the small SAP in the actual mortars could also release the internal water at very early ages.

Furthermore, the small SAP had a tendency to cluster together at short distances as mentioned above. Thus moist environment is kept around the cluster of SAP. On the other hand, this early supply of water results in higher degrees of hydration of cement so that dense microstructure is formed in the vicinity of the SAP cluster. This prevents the water from further travelling into a desiccated matrix. As a result, the internal relative humidity of mortar cannot be maintained high because of early exhaustion and restricted movement of the internal water. On the other hand, the large SAP exhibited clustering at long distances where a probability of being cement paste matrix followed the Poisson process. This means the distribution of large SAP is not greatly restricted into small room within the correlation distances of random areas of cement paste matrix. Furthermore, release of the internal water from the large SAP goes on slowly compared to the small SAP (Fig. 9). Thus, the large SAP can cover a wider area with the released water for a long time. This beneficial situation in the initial dispersion could affect the reduction in autogenous shrinkage in the mortars. In other words, large SAP is necessary to realize unbiased moisture distribution due to random dispersion of internal water reservoirs.

Fig. 8 Autogenous shrinkage of mortars

Fig. 9 Changes of absorptivity with time
4 CONCLUSIONS

Spatial dispersion of SAP was evaluated by point process statistics. SAP in mortars was also represented as a point pattern thinned by aggregate. The statistic function for the thinned process was compared with that for real distribution of SAP in actual mortars. Reduction in autogenous shrinkage of the internally cured mortars was related to characteristics of spatial distribution of SAP. Major outputs in this study are as follows;

1) SAP particles dispersed randomly in cement paste specimens. The null hypothesis of Poisson process is valid for the distribution in cement paste.
2) SAP particles in actual mortars exhibited a clustering tendency. Their degree of clustering was greater than that of the thinned point process, which was obtained from the basic point process in cement paste specimens.
3) Distances of clustering depended on sizes of SAP. The smaller SAP clustered at shorter distances than the correlation length of random areas of cement paste matrix in mortars.
4) Clustering of SAP observed in mortars is not realized by the binominal process. The null hypothesis of Poisson process may be rejected for the spatial distribution of SAP in mortars.
5) Properties of absorbing and releasing water depend on sizes of SAP. The release of the internal water from the clustered SAP could result in inhomogeneous moisture distribution in mortars.
6) Biased distribution of SAP due to the clustering could decrease its efficiency as an internal curing material.

5 ACKNOWLEDGEMENT

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6 REFERENCES

HAIL PROTECTED ETFE-ROOF LIGHTS FOR THE NEW ELEPHANT PARK BUILDING OF ZOO ZURICH

P. Flüeler¹, W. Kübler², A. Hohl³
(1) Flüeler Polymer Consulting GmbH, CH 8607 Aathal ZH – info@fluelerpolymer.ch
(2) Walt+Galmarini AG, CH-8008 Zürich – wolfram.kuebler@waltgalmarini.ch
(3) Zoo Zurich AG, CH-8044 Zurich – andreas.hohl@zoo.ch

Abstract
A wooden dome in the new elephant park of Zoo Zurich contains 271 irregularly shaped roof lights varying from 0.7 to 27 m² in size. They cover 2500 m² of the total surface of 7500 m². The site assembled roof lights are covered by 3-layer pneumatically inflated ETFE-cushions. On top, an additional sacrificial ETFE-layer protects the cushions against hail damage.

Since the region of Zurich is hit every 2-3 years by severe hail storms, the building envelope had to meet a hail impact resistance (HIR) class 4 for the entire building envelop according to Swiss Association of Public Building Insurance Companies APBIC.

The hail impact evaluation and final tests using 4 and 5 cm ice projectiles (round and pointed) proofed that the top layer protects the inner cushions against 5 cm projectiles. Pointed projectiles perforate the sacrificial layer but contact with the inner layers is prevented due to the high tear resistance of ETFE. Furthermore, the specific test protocol of membrane structures for the register of hail impact resistant products will be discussed. The opening of the new elephant park is set for June 7, 2014, see www.zoo.ch.

1 INTRODUCTION
In the last 13 years, the greater area of Zurich was subject to 5 major hail storms causing major damage to buildings and vehicles [1]. Collected hail stones had diameters up to 5 cm or greater. The direction of the storms on each of these 4 occasions was west-south-west (2002, 2004, 2011, 2012), the most recent storm in 2013 was from south-south-east (Figure 1). That experience teaches us that hail storms are very frequent and the direction or the hail path may vary. Building envelopes can be impacted on almost all sides.

In early summer 2002, the new Masoala rainforest building of Zoo Zurich with its ETFE-envelope was hit by hail only 2 weeks after its construction [2]. It caused damage worth 4.3 million CHF compensated by the building insurance of the state of Zurich. For further coverage the insurance company required protection against hail. This was achieved by an additional sacrificial layer on top of the three layer cushions. The effectiveness of this
measure was proven only two years later in a new hail storm. In all subsequent storms it proved to be successful enough to protect the ETFE cushions. Until now, small air leaks in the top layer were compensated for by intermittent air pumping.

Figure 1: Directions of hail paths and annual hail damage statistics in CHF of Zurich area years 2002, 2x2004, 2011, 2012 and 2013 (left to right, GVZ 2012, MeteoSchweiz)

2 THE NEW ELEPHANT PARK BUILDING

2.1 Some building properties

The elephant park and its unique building were designed by Markus Schietsch architects, Zurich. Engineering work for the building was carried out by Walt+Galmarini AG, Zurich. The main building (Figure 2) can be described in short terms as follows [3, 4 and 5]:

**Building**
- Size: 5400 m²
- Height: 17 m (internally)
- Form: asymmetric shell
- Span: 85 m
- Roof type: cross laminated timber shell
- Thickness: ca. 95 cm
- Bearing: linear, circumferential
- Circumference: 270 m
- Construction: pre-stressed concrete ring

**Roof lights**
- Arrangement: 271 cut-outs of the shell
- Light size: 2500 m², var. 0.7 to 27 m²
- Light forms: all irregular formed
- Construction: on site, customized
- System: Texlon®, inflated cushions
- Materials: ETFE foils in alum - frame
- Cushions: 3 layers, + hail protection layer
- Waterproofing: single ply reinforced PVC-P membrane, 1.5 mm thick

Figure 2: Main building, wooden dome

Figure 3: Partially covered roof light edges
2.2 **Roof light construction**

Each of the 271 roof lights were assembled piece by piece on the roof because of the irregularity of the roof shape (Figure 4 and 5). The main frames consist of 6 - 9 aluminium profiles which are butt jointed in the corners having a gap of 8 - 20 mm. These profiles act as linear bearings for the ETFE-cushions. On top of the cushion, the sacrificial hail protection layer was mounted and connected to a separate air pressure system. The clamped parts of the cushions were protected on both sides by silicon rubber profiles to prevent water penetration. Leak water is guided and drained by water outlets on lowest levels of the inclined roof lights.

![Figure 4: Cross section at roof light](image)

![Figure 5: Base profile of the roof lights](image)

Special attention was given to the waterproofing of the profile gaps in the corners. During these activities, a temporary roof protected the entire dome in order to achieve optimal work conditions.

2.3 **ETFE-cushion**

The cushions were designed and manufactured by Vector Foiltec from 1.5 m wide extruded ETFE-foils, a polymer alloy of ethylene and tetra-fluoroethylene. Material information can be found in [6]. Foils having thicknesses of 250, 150 and 250 μ (from top to bottom) were jointed by a thermo fusion process to the desired size and shape. The overlay top layer for hail protection was also 250 μ thick. A 4 cm thick spacer profile made of PIR-foam separates the overlay of 4 cm from the ETFE-cushion. The most exposed top edge of the frame is covered by thermally insulated L-shaped aluminium profile (Figure 6 and 7).

![Figure 6: Concept and air inlets](image)

![Figure 7: Insulated cover profile](image)
3 HAIL IMPACT TESTING

3.1 Test protocol

The APBIC test protocols part A, B and 22 were applied with the goal being to evaluate the hail impact resistance (HIR) class [7]. In these protocols all necessary conditions are set. The ice balls have to be launched within 60 s of removal from the storage container. In an initial attempt, the weakest part/zone on a test object needs to be established, with respect to the main functions. For membrane construction, the functional properties of water tightness, light transparency and visual appearance must be tested.

HIR class: HIR class represents the resistance against impact of 1 - 5 cm ice projectiles struck 5 times at the weakest zone of a conditioned material, probe or element.

3.2 Test equipment

For the tests, a pneumatically operated hail launcher was used. The launcher operates vertically and has barrels for projectile (balls and other forms) with diameters from 10-50 mm. The pneumatics allows adjustments in mbar-range and has a time efficient release mechanism. Aiming and distance to target are controlled by two focused lasers in a distance of 300 mm from target. High precise light beams separated by a distance of 150 mm measure accurately the elapsed time and calculate speed of each impact.

3.3 Ice projectiles

As projectiles, ice balls made of clear ice with 40 and 50 mm sizes stored at a temperature of -20° C were used. Their properties differ from real hailstones but provide high reproducibility for the test. In order to simulate irregularly shaped natural hailstones, so called “Teton” projectiles were also launched (Figure 8). They have a similar weight and diameter as the ice balls but have a 5 mm radius tip. This tip exacerbates local stresses of the stricken object. Soft targets like membranes, textiles and others may be more vulnerable to such influences and may perforate and tear.

Figure 8: Teton projectile with 4 cm diameter

3.4 Test object and conditioning

The test object, representing a real application with all boundary and size conditions, is positioned so that the projectiles impact at an angle of 90°. Before release, the target zone of a polymer based material needs to be conditioned by a 3 min. exposure to ice scales. Air pressure inflated structures like ETFE cushions are kept at the prescribed air pressure of the building. Since the roof lights have two separate pressure systems the following air pressure regimes were set:

Overlay to cushion: \( p_{\text{ext}} \leq 203 \pm 15 \text{ Pa} \)
ETFE cushion: \( p_{\text{int}} \leq 230 \pm 15 \text{ Pa} \)
When projectiles produced leaks the overlay had to be repaired and brought up to the required pressure level for the next test.

4 HIR TESTS AND RESULTS

4.1 Prequalification

In the initial phase a soft and a rigid concept were tested and evaluated on dummy roof lights. Test condition and results for two concepts are listed in table 1:

Concept A stiff: Two 6 mm-Polycarbonate plates glued together and separated by 25 mm spacers on the boundaries

Concept B soft: Inflated cushion with 3 ETFE-foils protected by an overlay ETFE-overlay

Table 1: Conditions and results of prequalification tests

<table>
<thead>
<tr>
<th>Roof light concept</th>
<th>A (stiff, even)</th>
<th>B (soft, curved)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Probe</td>
<td>Triangle</td>
<td>Square jointed to triangle</td>
</tr>
<tr>
<td>Shape</td>
<td>Plate</td>
<td>Inflated cushion</td>
</tr>
<tr>
<td>Form</td>
<td>160 x 60 cm$^2$</td>
<td>$\alpha=33^\circ$</td>
</tr>
<tr>
<td>Projectiles</td>
<td>Ice balls 5 cm</td>
<td>Ice balls 4 an 5 cm</td>
</tr>
<tr>
<td>Req. impact energy</td>
<td>4 / 5 cm balls: 11.1 / 27 Joule</td>
<td>4/5 cm balls and Teton: 11.1 / 27 J</td>
</tr>
<tr>
<td>Targets</td>
<td>Corners, tip corner, welding joints, center, edges</td>
<td>Corners, welding joints, center, edges</td>
</tr>
<tr>
<td>Observation</td>
<td>No failures up to 78 Joule, failure at 118 Joule</td>
<td>Balls: indents, no perforation</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Teton: perforation but no tearing</td>
</tr>
<tr>
<td>Weakest zone</td>
<td>Corner</td>
<td>Corners, edges</td>
</tr>
<tr>
<td>Evaluation</td>
<td>HIR class 5</td>
<td>250 $\mu$-overlay protects the cushion against 4 and 5 cm ice projectiles</td>
</tr>
</tbody>
</table>

4.2 Main qualification tests

Despite a high impact energy and reserve against failure, the ownership decided for the soft concept using air pressure to maintain shape. Other reasons like variable size, large thermal induced dimensional changes, vacuum leak, weight etc. as well as price led to this solution. For final acceptance by the building insurance, two real of the 271 actual roof lights had to be tested. Test conditions and results for two different shaped roof lights are presented in table 2.

Table 2: Conditions and results of main tests

<table>
<thead>
<tr>
<th>Roof light (RL)</th>
<th>C (soft)</th>
<th>D (soft)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Probe (light number)</td>
<td>RL no 21A</td>
<td>RL no 20A</td>
</tr>
<tr>
<td>Size</td>
<td>3.9 m$^2$</td>
<td>4.4 m$^2$</td>
</tr>
</tbody>
</table>
Shape

<table>
<thead>
<tr>
<th>Inflated cushion</th>
<th>Inflated cushion</th>
</tr>
</thead>
<tbody>
<tr>
<td>Form</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>Overlay, thickness</td>
<td>ETFE, 250 µ, 4 cm clearance at fixation zone</td>
</tr>
<tr>
<td>Projectiles and required energy</td>
<td>Ice balls and Teton Ø 4 and 5 cm</td>
</tr>
<tr>
<td>11.1 / 27 Joule</td>
<td></td>
</tr>
<tr>
<td>Targets</td>
<td>Corners, welding joints, center, edges</td>
</tr>
<tr>
<td>Number of impacts</td>
<td>Balls Ø 4/5 cm: 17/2</td>
</tr>
<tr>
<td></td>
<td>Teton T3/T4 cm: -/13</td>
</tr>
<tr>
<td></td>
<td>Balls Ø 4/5 cm: 8/-</td>
</tr>
<tr>
<td></td>
<td>Teton T3/T4 cm: 5/17</td>
</tr>
<tr>
<td>Observation</td>
<td>Balls: indents but no perforation of overlay</td>
</tr>
<tr>
<td></td>
<td>Teton: overlay perforated but no tearing, no damage on cushion</td>
</tr>
<tr>
<td>Weakest zone</td>
<td>Corners</td>
</tr>
<tr>
<td>Evaluation</td>
<td>Protection against 4 and 5 cm ice projectiles is given</td>
</tr>
</tbody>
</table>

4.3 Roof light top frame

The aluminium top frames covering the fixation zones of the ETFE-membranes are exposed to hail as well. Therefore a series of 7 tests were conducted in order to establish if the profile meets the requirements HIR 4. None of the 7 shots with 4 cm ice balls caused any changes in the visual appearance.

5 COMPLEMENTARY TESTS

5.1 Butyl tape tension test

Butyl tapes [8] were adhered in all corners of the base aluminium profile of the roof lights (Figure 9). The joint of the subsequent rubber and spacer profiles were filled with a SI sealant. Finally, it was covered by an additional layer of butyl tape on top of the aluminium profile. As a result of the roof light length and due to daily and annual temperature changes, profile dilatations may reach ±4 mm. As a consequence, the rigidly clamped butyl tapes between the profile and the silicone rubber are periodically stressed.

Fig. 9: Profile joint bridged by butyl tape
Fig. 10: Si-sealant filled in the profile gap
Fig. 11: Final sealing of top frame by butyl tape
For the tension test the butyl mass of the tape was removed and the bare tape was tested. The tape consists of polyester-aluminium-polyester foil and a 1 mm thick uncured butyl mass. It showed in the tension tests the following behaviour:

- Stress @ rupture: $105 \pm 1.9$ MPa
- Extension at rupture: $35.2 \pm 2.1\%$
- Stress @ stretching: $\sim 83$ MPa
- Stretching limit: $\sim 2.5\%$
- Fracture type: ductile, tough

### 5.2 HIR test of adhered butyl tape

This tape adhered over the silicon sealant filled profile joint is sought to prevent instant water penetration when the silicon sealant separates with time. It is therefore exposed to hail as well (Figure 10, 11). A series of HIR tests were conducted on such an application in order to explore the water tightness of the joints.

Results: All 3 cm ice balls impacting the tape by 3.5 J compresses the butyl tape and squeezes the butyl mass at the profile edge (Figure 12). Shearing-off and total quenching was the result when 4 cm ice balls hit the joint area with a kinetic energy 11 joule (Figure 13). Despite this damage, the joint did not leak and remained waterproof when leak tested by vacuum bell (Figure 14).

### 6 CONCLUSIONS

- Recent weather experiences have shown that the greater area of Zurich suffers more frequently from severe hail storms with hail stones up to 5 cm. All sides of buildings can be hit.
- Inflated ETFE-membrane structures susceptible to hard body impact leaks have to be protected against hail stone impact.
- Sufficient dimensioning and testing is mandatory to evaluate the foil thickness while also having regard to wind driven storm debris.
- This has been realized by an independently pressurized overlay above the roof light cushions.
To prevent damage to the inflated cushions the overlay should have a clearance of >10 cm. In the fixation zone the overlay of >4 cm at least.

- A 250 μ-ETFE overlay foil resists 5 cm hail stones with smooth surfaces. Point shaped hail stones may perforate but will be stopped by the overlay because of the high tear resistance of ETFE.

- The 4 mm thick aluminium cover profile over the fixation zone will not show indents by 4 cm-hail stone impact.

- Special attention was given to joint sealants because of high thermal dilatations of long aluminium profiles and narrow joint gaps.

- Tapes of uncured butyl sealant as additional sealing in the corners of jointed frame profiles do not withstand 3 cm hail stone impact. If the butyl mass remains tacky and flexible the joint may still be water tight if not, easy replacement is possible.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the contribution of Vector Foiltec (H.C. von Düiring and co-workers) for financial support and providing the test specimens.

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REFERENCES

CHARACTERIZATION OF MORTARS WITH CERAMIC RESIDUES

Matias, G. (1), Torres, I. (2) and Faria, P. (3)

(1) ITeCons, Coimbra - Portugal, ginamatias@itecons.uc.pt
(2) DEC - FCTUC, Coimbra – Portugal, itorres@dec.uc.pt
(3) DEC – FCT UNL, Lisboa – Portugal, paulina.faria@fct.unl.pt

Abstract

Mortar coatings are usually the first elements to deteriorate, once they are the most exposed construction elements. Taking into account their protective function, it is easily understood the importance of the development of a correct conservation / rehabilitation intervention.

Old mortars were essentially air lime based and their rehabilitation must be executed with compatible mortars, so that they not only recover the protective function, but also contribute to maintain the integrity of the whole wall-render element and the aesthetic image of the building.

As it is known, many old lime-based mortars had ceramic residues in their composition, which are very abundant residues, particularly in Portugal. Thus, in an attempt to combine the interest of reusing waste ceramic, reducing the amounts that are typically landfilled, with the technical benefits that they can bring to rehabilitation mortars, experimental research has been developed, at the University of Coimbra, in collaboration with the NOVA University of Lisbon, on the behavior of lime mortars with incorporation of ceramic residues.

In this paper, it is presented a comparison between air lime mortars with ceramic residues with volumetric proportions of 1:3 and 1:2 (binder:aggregate), showing the advantages of the reintroduction of the ceramic residues into the productive chain. Studied mortars revealed a quite satisfying behavior as the inclusion of ceramic residues may improve the characteristics of common air lime mortars.

1 INTRODUCTION

The use of ceramic residues on mortars’ production dates, at least, from the Roman Period. In Portugal it is still possible to find several rendering examples from that period with this type of practice, a proof of its longevity and reliability. Ceramic fragments were introduced in mortars as dust or/and grain particles, and reacted with the most common binder in the past – air lime. Pozzolanic reactions developed, between lime and the fine ceramic particles, induced an improved behavior and revealed mortars with hydraulic characteristics. Larger fragments also developed chemical reactions with the binder, not yet well known, but their essential role was to partially substitute common aggregates. This type of mortars was used with very
different functions, being the most common, especially during Roman Empire, the water proofing requirement. They were largely used for baths, tanks and aqueduct coverings [1].

High specific surface of ceramic dust and high amounts of silica and alumina in the amorphous phase are the main factors that potentiate pozzolanic reactions. The use of ceramic dust may allow to reduce the content of binder in mortars, as it improves their behavior. This will bring benefits from the environmental point of view, once it may enhance a reduction on the exploitation of natural resources and also on the emission of carbon dioxide from the production of binders.

As aggregate, and with adequate particle’s size distributions for mortar’s preparation and similar to commonly used sand, ceramic residues allow a significant reduction in the extraction of natural resources. It has been verified, especially in cement mortars, that this substitution improves their behavior [2]. It must be considered, however, that the characteristics of the aggregate greatly affect the performance of the mortars: roundness, angularity and texture of the grains conditions the binding structure and, consequently, the workability on fresh state and the water amounts required [3]. In hardened state, particles’ distribution has impact especially on mortars’ microstructure and mechanical behavior [4]. Using this type of materials as aggregate replacer might contribute for the reduction of natural resources consumption and, consequently, prevent hazardous environmental impacts caused by natural sand’s extraction.

To the development of this experimental work, three types of residues from ceramic industries of the central region of Portugal were selected. These industries produce ceramic bricks, roof tiles and decorative pots. According to producers’ information, calcination temperatures usually go around 900ºC, in the case of bricks and roof tiles, to 1100ºC in the case of decorative pots. But the period of time at the highest temperature may be restricted. Residues were milled and the resultant material, considered as adequate after particle’s size distribution characterization, was incorporated in air-lime mortars as a partial substitute of the aggregate. This paper presents the results of an experimental campaign developed with mortars with 1:2 and 1:3 volumetric proportions of binder and aggregate. Results from tests performed after 60 days and 120 days of curing are presented: mechanical strength, water vapor permeability and water absorption due to capillary action.

With this work it is intended to enlarge the knowledge about ceramic residues’ incorporation in mortars for repair / rehabilitation purposes or, eventually, for actual construction, considering particularly their durability and longevity. It is expected to obtain compositions with improved behavior reusing materials that are considered waste and disposed into landfills, contributing, in this way, for a more sustainable construction.

2 EXPERIMENTAL PROCEDURE

2.1 Materials characterization

Mortars were prepared using hydrated powder air lime as binder, a river sand and ceramic residues.

Air lime, designated as CL 90 according to EN 459-1:2010 standard, was produced by Lusical (designated as H100). Their Ca(OH)$_2$ minimum content is 93%. Ceramic residues were constituted of brick, roof tile and decorative pot from Portuguese industries and were simply crushed, with a laboratory Retsch jaw crusher, with a 10mm cribble. The ceramic material and the sand were characterized in what concerns to particle size distribution according to EN
and the obtained results are presented in Figure 1. Loose bulk density was also determined to allow weight proportions’ evaluation. Values obtained for lime, sand and for each one of the ceramic residues, determined according to EN 1097-3:1998, are presented in Table 1.

Analyzing Figure 1 it can be noticed that the particle’s size distribution of the residues, after being crushed, is quite similar to the one of the river sand. They only differ because residues contain about 10% of particles under 0.063mm, while smaller particles from river sand are almost residual.

Table 1: Loose bulk density, $\rho_b$, of the materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Brick (B)</th>
<th>Tile (T)</th>
<th>Pot (P)</th>
<th>River Sand</th>
<th>CL</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\rho_b$ (Mg/m$^3$)</td>
<td>1.10</td>
<td>1.17</td>
<td>0.99</td>
<td>1.54</td>
<td>0.36</td>
</tr>
</tbody>
</table>

In what concerns to loose bulk density of the dry materials all residues present quite similar values, lower than river sand. Air lime bulk density has the lowest value obtained.

2.2 Mortars characterization

Several mortars’ compositions were prepared with the materials described above. Volumetric proportions considered were 1:3 and 1:2 (binder/aggregate) and corresponding mortars were attributed prefixes 3 and 2, respectively. For each type of residue, 20% and 40% of the natural sand was replaced - mortars with 20% of sand’s substitution were designated with an L (Low) and mortars with 40% of sands substitution were designated with an H (High). Mortars with brick residues were attributed B suffix, mortars with tile sharks, T, and mortars with decorative pot were attributed a P. Besides the indicated compositions, two reference mortars without residues were also prepared and they were referenced with an R. These mortars allowed the evaluation of the influence of each residue, as well as the influence of the substitution percentages.
All mortars were prepared with water amounts that allowed a consistency in fresh conditions of 135-165 mm, a range that assure workability to this type of lime-based mortars. This parameter was determined according to EN 1015-3:1999 standard (flow table method). Water/binder proportions were also determined and results are presented in Table 2. Table 3 indicates the volumetric and weight proportions of the mortars.

Table 2: Water/binder ratio (w/b) by weight and flow table consistency (fv)

<table>
<thead>
<tr>
<th>Mortar</th>
<th>3R</th>
<th>3LB</th>
<th>3HB</th>
<th>3LT</th>
<th>3HT</th>
<th>3LP</th>
<th>3HP</th>
<th>2R</th>
<th>2LB</th>
<th>2HB</th>
<th>2LT</th>
<th>2HT</th>
<th>2LP</th>
<th>2HP</th>
</tr>
</thead>
<tbody>
<tr>
<td>w/b</td>
<td>2.85</td>
<td>2.21</td>
<td>2.56</td>
<td>2.62</td>
<td>2.35</td>
<td>2.35</td>
<td>2.63</td>
<td>1.66</td>
<td>1.66</td>
<td>1.87</td>
<td>1.66</td>
<td>1.66</td>
<td>1.66</td>
<td>1.51</td>
</tr>
<tr>
<td>fv</td>
<td>147.6</td>
<td>150.8</td>
<td>152.9</td>
<td>151.3</td>
<td>153.2</td>
<td>151.4</td>
<td>151.0</td>
<td>164.8</td>
<td>152.4</td>
<td>164.3</td>
<td>163.1</td>
<td>148.2</td>
<td>149.2</td>
<td>156.1</td>
</tr>
</tbody>
</table>

Table 3: Volumetric and weight proportions

<table>
<thead>
<tr>
<th>Mortar</th>
<th>CL</th>
<th>Residues</th>
<th>Sand</th>
<th>CL</th>
<th>Residues</th>
<th>Sand</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>B</td>
<td>T</td>
<td>P</td>
<td>CL</td>
<td>B</td>
<td>T</td>
</tr>
<tr>
<td>3R</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>3</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td>3LB</td>
<td>1</td>
<td>0.6</td>
<td>-</td>
<td>2.4</td>
<td>1</td>
<td>1.8</td>
</tr>
<tr>
<td>3HB</td>
<td>1</td>
<td>1.2</td>
<td>-</td>
<td>1.8</td>
<td>1</td>
<td>3.6</td>
</tr>
<tr>
<td>3LT</td>
<td>1</td>
<td>-</td>
<td>0.6</td>
<td>2.4</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td>3HT</td>
<td>1</td>
<td>-</td>
<td>1.2</td>
<td>1.8</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td>3LP</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>0.6</td>
<td>2.4</td>
<td>1</td>
</tr>
<tr>
<td>3HP</td>
<td>1</td>
<td>-</td>
<td>1.2</td>
<td>1.8</td>
<td>1</td>
<td>-</td>
</tr>
<tr>
<td>2R</td>
<td>1</td>
<td>-</td>
<td>-</td>
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<tr>
<td>2LB</td>
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<td>0.4</td>
<td>-</td>
<td>1.6</td>
<td>1</td>
<td>1.2</td>
</tr>
<tr>
<td>2HB</td>
<td>1</td>
<td>0.8</td>
<td>-</td>
<td>1.2</td>
<td>1</td>
<td>2.4</td>
</tr>
<tr>
<td>2LT</td>
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<td>0.4</td>
<td>1.6</td>
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<tr>
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<td>-</td>
<td>-</td>
<td>0.4</td>
<td>1.6</td>
<td>1</td>
</tr>
<tr>
<td>2HP</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>0.8</td>
<td>1.2</td>
<td>1</td>
</tr>
</tbody>
</table>

With the fresh mortars, based on EN 1015-11:1999/A1:2006, cylindrical specimens for water vapor permeability determination and prismatic specimens, with 40mm x 40mm x 160mm dimensions, for mechanical strengths and capillary water absorption, were prepared. Specimens were kept at 20°C and 95% relative humidity (HR) for the first five days. They
remained for two more days in 20°C and 65% HR and were demolded. They remained in these standard conditions until the ages of test - 60 and 120 days. The incorporation of ceramic residues also acted as a natural pigment and the mortars became slightly reddish colored.

2.3 **Mechanical strengths**

Flexural and compressive strength were determined according to EN 1015-11:1999/A1:2006 standard specifications. For this purpose, three prismatic specimens were tested, for each sample and curing period. Compressive strength resistance was determined with one of the halves resultant from flexural strength tests. Flexural strength results are indicated in Figure 2 and compressive strength results are presented in Figure 3. Standard deviation is also graphically represented.

![Figure 2: Flexural strength at 60 and 120 days of curing](image)

![Figure 3: Compressive strength at 60 and 120 days of curing](image)

It was observed that flexural and compressive strength did not present a significant increase through time, except for most of the mortars with high percentages of residues. All mortars with high percentage of residues clearly revealed higher strength and this difference was more evident for 1:2 mortars. In all cases, mortars with ceramic waste register higher strength than the reference mortars, 3R and 2R.

2.4 **Water absorption due to capillary action**

Water absorption due to capillary action was determined according to EN 15801:2009 standard, appropriate to slow curing mortars, such as air lime ones. One of the two resultant
halves from flexural tested specimens was used and weightings were carried out at 5, 10, 15, 30, 60, 90, 120, 180 minutes and each 24 hours after a first contact of the specimens with the water slide; the test went on until constant mass was reached. Water absorption coefficient and corresponding standard deviation are represented in Figure 4.

All mortars obtained quite uniform values. Differences observed are very slight; however, higher capillary coefficients correspond to mortars with higher percentages of residues.

2.5 Water vapor permeability

Circular specimens used to determine water vapor permeability had an approximated diameter of 100 mm and thickness of 17mm. The procedure indicated by ISO 12572:2001 standard was followed and the wet cup method was selected. Figure 5 presents the water vapor diffusion-equivalent air layer thickness values, $S_d$, which allows thickness evaluation of an equivalent air layer in terms of water vapor resistance.

Also for water vapor diffusion-equivalent air layer thickness significant differences between mortars were not verified. Mortars with higher water vapor resistances are the ones with higher percentage of residues, despite these differences are very slight, and all mortars obtained lower values than equivalent reference ones. In what concerns to binder:aggregate proportions, results were quite similar and no evolution related to curing period seemed to occur.
3 DISCUSSION

In what concerns to mechanical behavior, the increase of flexural and compressive strengths with the increase of the percentage of ceramic residues might be related with two aspects: in one hand, the increase of residues, which contain 10% more fines than common sand, reduces the porosity of the mortars due to filling of pores; on the other hand, the presence of fine ceramic particles may develop pozzolanic reactions with the binder, also leading to the increase of strength. This hypothesis is reinforced with the fact that mortars with 1:2 (binder:aggregate) proportion present more expressive differences between samples with lower and higher substitution percentages. The fact that all mortars present higher strengths than corresponding reference ones may also be related with the aggregates’ shape. Substituting common siliceous sand, rounder shaped, for ceramic fragments, generally more angular and rough, might contribute to the increase of the binder-aggregate cohesion.

Mortars 3LT, 2HB and 2HP presented a decrease of strength at 120 days of curing. Any explanation was found for this fact and, for that reason, more studies should be developed to understand eventual instable products that may be formed at specific curing periods.

Regarding to water absorption due to capillary action, slight differences are observed. The increase of absorption with the increase of residues percentages is related with the increase of smaller particles, which affects the amount and size of the pores and also with the water absorption capacity of the residues - higher than siliceous sand.

In terms of water vapor permeability, despite the fact that all mortars present uniform results, samples with higher amounts of residues revealed higher water vapor resistances, once again hypothetically related with changes in the porous structures. The increase of smaller pores may be directly related with water absorption results.

In general, the absence of significant changes thought time in terms of behavior in the presence of water (liquid or vapor) might be an indicator of unchanged porous structures due to carbonation and hydration processes.

Several researchers established requirements for substitution mortars for old buildings, applied as renders, plasters or for repointing. Veiga et al. [5] defined the interval of 0.2 N/mm² to 0.8 N/mm² as adequate for flexural strength and 0.4 N/mm² to 3 N/mm² for compressive strength. Generally, it is observed that results obtained for the tested mortars with higher percentages of residues fulfill this requirement. The same researchers indicate that water vapor diffusion-equivalent air layer thickness should present values inferior to 0.1 mm. None of the tested mortars satisfied this requirement. However, obtained values are close to this limit and they might be considered adequate for the effect that is purposed. Veiga et al. [5] also consider that capillary coefficients between 1.0 kg/(m²/min¹/2) and 1.5 kg/(m²/min¹/2) are adequate for substitution mortars. Tested mortars with lower residues’ substitution percentages obtained values close to the maximum defined, despite all mortars presented slightly higher values.

It should be taken into account that test methodologies might have some differences and, as so, direct results comparison must be careful.

4 CONCLUSIONS

Studied air lime-ceramic waste mortars presented quite satisfying results in what concerns to mechanical behavior. The presence of residues, particularly in higher percentages, revealed
quite beneficial when compared to reference mortars. Despite eventual changes in the porous structures, it might be induced that some kind of pozzolanic reaction may occur.

In what concerns to physical behavior it was observed a straight relation between results obtained for water absorption and water vapor permeability and the hypothetical changes introduced in the porous structure. The increase of fine particles of ceramic substitution sand lead to an eventual decrease of pores sizes and, consequently, increased capillarity effect and also increased water vapor resistance. However, values obtained were considered quite adequate for rehabilitation mortars.

No significant differences were observed related to curing period or even to binder:aggregate proportions. Both studied proportions seemed adequate for their purpose, being the 1:3 more eco-efficient.

Globally, it is considered that studied mortars have a quite satisfying behavior and that the inclusion of ceramic residues may improve the characteristics of common air lime mortars. Having mortars with improved behavior when formulated with ceramic residues, that are generally landfilled, encourage the continuity of this study.

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5 REFERENCES


STUDY OF THE DURABILITY OF MORTAR FOR STRUCTURAL CONCRETE MADE WITH FINE POWDER OF WHITE GLASS

Fernando A. Sales(1), Rosemary B.C. Sales(2) Elaine C. S. Corrêa(3) Neley D.S. Molhallem(4) and Maria Teresa P. Aguilar(5)

(1) Departamento de engenharia Mecânica, Centro Federal de Educação Tecnológica de Minas Gerais, Belo Horizonte, Brazil – fdosales@gmail.com
(2) Departamento de ciência e Tecnologias, Universidade do Estado de Minas Gerais, Belo Horizonte, Brazil - rosebcs@gmail.com
(3) Departamento de Química, Centro Federal de Educação Tecnológica de Minas Gerais, Belo Horizonte, Brazil - elaine@deii.cefetmg.br
(4) Departamento de Química, Universidade Federal de Minas Gerais, Belo Horizonte, Brazil - nelcydsm@yahoo.com.br
(5) Departamento de Engenharia de Materiais e Construção, Universidade Federal de Minas Gerais, Belo Horizonte, Brazil - teresa@ufmg.br

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Abstract
Waste content reactive silica is often used as a supplementary cementitious material. It is common to use granulated slag from blast furnaces, silica fume, and fly ash in partial replacement of Portland cement materials. Another alternative material with reactive silica is glass waste. This substance is commonly used as a raw material in the manufacture of new glass, but can also be used as an aggregate for Portland cement concrete or asphalt concrete, or as a raw material for products made of clay. However, the fine powder waste with a particle size below 100 μm requires careful handling and is usually disposed of in landfills. Different studies indicate that these powders could partially replace cement in the manufacture of concretes. These studies are not conclusive, though.

This study is aimed at evaluating the durability of mortars made with fine powder (less than 30 μm) of colorless glass, and water-cement ratio of 0.5. Results indicate that the use of colorless glass residue to replace cement in 10 and 20% decreases the permeability of the cementitious compound to oxygen, whereas increases the thickness of carbonated layer, inhibits the alkali-silica reaction, and promotes the decrease of wear resistance.

Keywords: Waste glass, Portland cement, mortar, Mechanical properties
1 INTRODUCTION

The current need to obtain eco-efficient building materials has stimulated a number of studies involving the use of industrial and/or agricultural waste in the manufacture of cement and/or concretes. Depending upon factors such as the particle size, silica content, and degree of crystallinity, these residues can be used as aggregates or mineral additions of cementing, pozzolanic and/or filler action. The most common mineral additions of residues are blast furnace slag, fly ash, rice husk ash house ash and sugarcane bagasse ash [1-3]. Recently, the addition of glass waste in Portland cement compounds has drew the interest of both researchers and industries [4-9].

Glass waste is an inert non-biodegradable material [10] with high rates of utilization and recycling, and can be found in the form of cullet or powder. Wastes in the form of cullet are commonly used as raw material in the manufacture of new glasses [11-13]. The primary recycling of glass exhibits rates ranging from 47% to 96% depending on the continent, country or state [14-15]. In Brazil, near one third of all produced glass is recycled. In addition, the packaging and bottles sectors are responsible for recycling approximately four hundred and seventy thousand tons of waste glass [16]. Nevertheless, the glass waste powder resulting from the cutting and polishing of edges and holes usually goes to landfills.

The first studies on the inclusion of waste glass in cementitious materials have evaluated its use as aggregate and extend up to the present day [17-19]. Yet, these studies have revealed that the occurrence of alkali-silica reaction limits the use of this waste as aggregate [20-22]. The literature highlights the potential use of glass micro-particles as a pozzolanic material [19, 23, 24-25]. Moncea et al. [26] suggest the filler effect for glass waste used as a supplementary cementitious material. In effect, several studies address the reaction of alkali-silica associated with glass micro-particles [21, 22, 27]. As regards permeability and carbonation depth, there are few studies, though [28-29]. Works on the influence of these residues over the resistance to abrasion barely exist. Furthermore, most of these studies do not evaluate the influence of the chemical composition expressed by staining on the performance of the cement compounds. In this connection, this work is aimed at evaluating the durability of Portland cement compounds manufactured with clear-glass micro-particles and without the addition of coarse aggregate - in place of 10 and 20% - to cement mass. The performed assays addressed permeability to oxygen, accelerated carbonation, alkali-silica reaction and wear resistivity.

2 MATERIALS AND METHOD

The used cement was Portland of high initial resistance (Table 1) and low content of admixtures, thus decreasing the analysis variables. Moreover, it has high tricalcium silicate content, which provides higher amount of calcium hydroxide for pozzolanic reactions within a shorter period of time.

The residual colorless glass was produced in laboratory in order that the possible contaminants could be eliminated (Table 2). Specific mass was 2.60 g/cm³, specific surface was 371.8 m²/kg by Blaine method, and 5.218 m²/kg by BET method - Brunauer, Emmett and Teller. The particle size distribution of glass micro-particles was obtained by scattering the laser beam, and revealed that 50% of all particles had a diameter of 4.70 μm, whereas 90% had 28.35 μm. X-ray diffraction showed that micro-particles had a diffuse spectrum, which is typical of non-crystalline phases.
Table 1: Physical and chemical characterization of Portland cement

<table>
<thead>
<tr>
<th>Assays</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Insoluble residue</td>
<td>0.42%</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>2.03%</td>
</tr>
<tr>
<td>Magnesium Oxide</td>
<td>1.15%</td>
</tr>
<tr>
<td>Sulfur Trioxide</td>
<td>2.79%</td>
</tr>
<tr>
<td>Carbonic Anhydride</td>
<td>0.99%</td>
</tr>
<tr>
<td>Specific Mass</td>
<td>3.11 g/cm³</td>
</tr>
<tr>
<td>Specific surface (Blaine method)</td>
<td>470.8 m²/kg</td>
</tr>
</tbody>
</table>

Table 2: Chemical composition of colorless glass micro-particles

<table>
<thead>
<tr>
<th>Assays</th>
<th>(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Insoluble residue</td>
<td>92.72</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>0.22</td>
</tr>
<tr>
<td>Silicon Dioxide</td>
<td>73.93</td>
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<tr>
<td>Calcium Oxide</td>
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</tr>
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<td>Magnesium oxide</td>
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</tr>
<tr>
<td>Sulfur Trioxide</td>
<td>0.23</td>
</tr>
<tr>
<td>Sodium Oxide</td>
<td>9.18</td>
</tr>
<tr>
<td>Potassium Oxide</td>
<td>0.02</td>
</tr>
<tr>
<td>Alkali Equivalent</td>
<td>9.19</td>
</tr>
</tbody>
</table>

As fine aggregate, we have used the natural siliceous sand provided by the Institute of Technological Research of the State of São Paulo (standardized sand) with density of 2,650 kg/m³ for particle sizes of 1.2, 0.6, 0.3 and 0.15 mm. In the assays of alkali-silica reaction, we have used sand produced by crushing basalt rocks in the proportions indicated by ASTM C-1260 [30].

The used water was that available for consumption in the metropolitan region of Belo Horizonte by COPASA (Minas Gerais Sanitation Company).

In the manufacture of cementitious compounds, the used ratio was 1 binding agent (cement + cement or glass micro-particles): 3 sand (equal amounts of the four different particle sizes) and water-cement factor of 0.5. Mortars with a mean workability of 210mm in spreadability, and average compressive strength of 55 MPa have been produced.

As a criterion for durability assessment, oxygen permeability was determined in line with the recommendation of RILEM [31] to study concrete permeability. According to Neville [32], oxygen permeability serves to better depict the actual conditions in relation to the armature and is more representative so as to evaluate concrete in work. A permeameter built at the Department of Mechanical Engineering of UFSJ from the cell developed by Cabrera and Lynsdale in 1988, as described by Panzera Assisi and [33], has been used. This assay has measured the speed of gas percolation (flow) through the porous materials confined and subjected to the pressure difference between the considered points. The permeability of the
sample can be calculated by Equation 1, which takes into account the compressibility and the viscosity of oxygen, as well as the selected [34-35] units.

\[ k = \frac{4.64 \times 10^{-16} Q P_1 \cdot L}{A (P_1^2 - P_2^2)} \]  

(1)

\( K \) denotes intrinsic permeability (m²); \( L \) is the length of the sample (m); \( A \) stands for the cross sectional area of the sample (m²); \( Q \) means flow rate (cm³/s); \( P_1 \) is the applied absolute pressure (in bar), and \( P_2 \) is the pressure through which flow is measured (in bar). For the tests, three test specimens with dimensions of 5x5 cm have been fabricated for each type of mixture. The samples were placed in a chamber at 80°C for 24 hours to dry and the tests were performed after cooling.

The assay for resistance to carbonation was carried out in a climatized Thermo Fisher Scientific chamber, model RCO 3000T-5-VBC. The samples were immersed in water and were cured for 28 days. After curing, the sample surface was brushed and washed to remove eventual residues of demolding agents or cement laitance, which could hinder the diffusion of carbon dioxide into the interior of cementitious compounds. Before being introduced into the carbonation chamber, the samples were dried in a chamber. The assay parameters (saturated environment within the equipment itself in an atmosphere with 5% CO\(_2\) at 27.5 °C±2) employed in this study were defined in consonance with the work of Nobrega and Araújo [36]. The samples remained in a saturated environment and were exposed to CO\(_2\) for 90 days. After this period, the samples were longitudinally sectioned, and the depth of carbonation was determined from the application of a solution of 70% absolute ethanol, 29 % distilled water and 1% phenolphthalein by spray irrigation. Using a parking meter of 0.01 mm accuracy, three measurements by test specimen were obtained.

The assay of alkali-silica reaction had two aims. The first aim was to evaluate if the alkali content in the glass could contribute to further expand the test specimen as a result of the alkali-silica reaction of the aggregate. The second aim was to assess whether the silica present in glass and that was not consumed in the pozzolanic reaction would later react with the cement alkali (hardened compound). Such phenomenon occurs when glass is used as aggregate [19, 37].

The procedure for the assay of alkali-aggregate reaction was based on ASTM C-1260 [30]. A set of equipment (bath/chamber and extensometer) of the Soloteste brand has been used. Three test specimens with dimensions of 25x25x250 cm were prepared for each type of compound. The used plan was 1:2.25 in mass, water-cement ratio of 0.47 and basaltic sand. The expansion limits were measured 16 days after molding, whereas other steps were carried out in staggered dates (0, 3, 6, 10, 19 and 30 days). The final value was calculated from the mean expansion of the three mortar bars as a function of age.

The test for resistance to abrasion was carried out following the procedures established by the Brazilian standardization [38]. Three prismatic test specimens of each type of material measuring 70x70x30mm have been prepared. After 28 days of curing, the samples were subjected to wear with quartz sand under a load of approximately 66 N and speed of (30±1) rpm. Measurements were held after 500 laps, which means 1,000 meters along the way. After 500 meters, the samples were inverted on the coupling bracket of the apparatus so as to prevent distortion (bias) of the material under test. After 1,000 meters, heights were measured at all the four sides of the samples to evaluate wear. The test was carried out using Amsler equipment manufactured by Contenco.
3 RESULTS

Table 3 exhibits the average values of the coefficient of oxygen permeability of samples with and without addition of glass, as well as the standard deviation of measurements. According to Lee, McCarthy and Dhir [39], the durability of a concrete with an oxygen permeability coefficient below $10^{-16}$ m$^2$ is considered good; between 10 and 16 m$^2$ and $3 \times 10^{-6}$ m$^2$ is moderate; and above $3 \times 10^{-16}$ m$^2$ is poor. Data reveal that the use of glass micro-particles in place of cement did not alter the performance of cementitious compounds (moderate) assessed by the assay of oxygen permeability. Nonetheless, the use of higher levels of substitution appears to contribute to reduce the permeability, thus improving the durability of composites in this aspect. The chemical composition and the non-crystalline structure of silica probably favor the occurrence of pozzolanic reaction when the glass is brought in contact with calcium hydroxide. Products resulting from this reaction promote the refinement and increase of tortuosity in the pore network, whereas decrease their connectivity [29, 40, 21].

<table>
<thead>
<tr>
<th>Sample</th>
<th>K</th>
<th>Mean</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>1.86</td>
<td>1.96</td>
<td>0.09</td>
</tr>
<tr>
<td></td>
<td>1.96</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.05</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Colorless 10%</td>
<td>1.47</td>
<td>1.73</td>
<td>0.24</td>
</tr>
<tr>
<td></td>
<td>1.96</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.78</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Colorless 20%</td>
<td>1.61</td>
<td>1.48</td>
<td>0.17</td>
</tr>
<tr>
<td></td>
<td>1.29</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1.55</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 1 shows the results of the accelerated carbonation assay of control samples and those with substitution of cement by 10-20% with micro-particles of colorless glass. The graph shows mean measurements and standard deviation after curing inside a carbonation chamber for 60 days. The results show that all samples were subjected to carbonation. Nevertheless, the depth of those made with glass micro-particles was greater as compared to those without glass micro-particles. Moreover, depth was seen to have been more pronounced in those with higher glass content. After 90 days of assay, the increased thickness of the carbonated layer was near 1.3 mm when compared with the control sample. Similar carbonation results were found by Matos and Coutinho [28] in a study using waste glass of different natures. According to some authors, the consumption of calcium hydroxide due to the pozzolanic reaction of compounds containing waste glass can be responsible for such meaningful carbonation depth [28, 41-42].
The mean assay results of alkali-silica reaction are exhibited in Figure 2 and Table 4. According to ASTM C-1260 [30] between 0.10 and 0.20 % occurred at 16 days an expansion and may indicate aggregates both with innocuous and reactive behaviors. Thus, we have decided to perform the test up to the 30 days of age: samples with 10 and 20% of colorless glass micro-particles were in class II: potentially reactive material.
Table 4: Results from alkali-silica reaction in 16 and 30 days

<table>
<thead>
<tr>
<th>Mixtures</th>
<th>mean expansion (%)</th>
<th>16 days</th>
<th>Standard Deviation</th>
<th>30 days</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>0.32</td>
<td>0.01</td>
<td>0.88</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>Colorless 10%</td>
<td>0.15</td>
<td>0.01</td>
<td>0.32</td>
<td>0.02</td>
<td></td>
</tr>
<tr>
<td>Colorless 20%</td>
<td>0.12</td>
<td>0.00</td>
<td>0.23</td>
<td>0.01</td>
<td></td>
</tr>
</tbody>
</table>

Results reveal that the use of glass micro-particles inhibits alkali-silica reaction even with the use of highly reactive aggregates. First, this means that the alkali content above the limit indicated for pozzolanic materials did not contribute to the occurrence of alkali reaction in the glass. This assertion can be related to the fact that the alkalis were encapsulated in gels contained in the cement paste, and were unable to react [43]. Secondly, the high silica content of the glass seems not to have promoted the occurrence of alkali-silica reaction in concrete. These effects are related to the fact that the high specific surface and the amorphicity of silica have led it to react with calcium hydroxide in the first ages. Thus, no silica content was left for the further reactions, i.e., when the concrete was hardened and unable to accommodate the reaction products.

Results of the resistance assay for abrasion carried out after 1,000 meters are shown in Figure 3, where the average values of thickness variation relative to the three test specimens measured crosswise at the four sample points after 28 days in a moist chamber are located. After 1,000 m, a meaningful variation was observed for the abrasion results. The replacement of Portland cement by 10 to 20% of micro-particles of colorless glass and amber has caused wear resistance to decrease. The glass content in the mixture clearly influenced this result. These data show that a weak layer was formed on the concrete’s surface probably due to the great exudation, which was reinforced by the presence of glass particles [44-45].

![Figure 3: Results of the resistance assay to wear by abrasion.](image-url)
4 CONCLUSIONS

The substitution of 10 and 20% of cement by colorless micro-particles contributes to reducing the oxygen permeability of compounds, while promotes increased thickness of the carbonated layer. In addition, it inhibits the alkali-silica reaction even with the use of reactive aggregates, and reduces wear resistance.

ACKNOWLEDGEMENTS

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REFERENCES


ADHESIVE TAPES FOR BUILDING PURPOSES: FRAMEWORK FOR EVALUATION OF PERFORMANCE AND DURABILITY

Selamawit M. Fufa (1), Marius Kvalvik (1) and Petra Rüther (1)
(1) Department of Materials and Structures, SINTEF Building and Infrastructure, Trondheim, Norway – selamawit.fufa@sintef.no; marius.kvalvik@sintef.no; petra.ruther@sintef.no

Abstract
There are different types of adhesive tapes applied in the building envelopes on the market which demand documentation of the properties of the adhesive tapes. In order to determine whether the adhesive tape will perform for its intended use, standard test methods which consider both the adhesive tape and the surface to which it is applied is important. The durability of the adhesive tapes is evaluated using 2 weeks of accelerated ageing in a climate simulator with four repeated cycles, according to NT Build 495, and 24 weeks of heat aging at 70 °C in accordance with EN 1296. The tensile strength and elongation, peel resistance and shear strength of the tape applied to different surfaces are evaluated before, during and after ageing. The durability test results give an overview on the performance of the adhesive tape when used in building envelopes. This study presents adhesive tape durability testing, evaluation and service life estimation methods, which hence form a basis for further improvements of the existing accelerated ageing methods.

1 INTRODUCTION
Adhesive tapes are used to seal joints or overlaps in the wind barrier layer to ensure adequate air tightness of the building. In Norwegian buildings, the wind barrier layer is often exposed to quite severe environmental conditions during the building phase. These can be rain, snow, dew and sunlight. An exposure of the wind barrier layer for three to six months is not unusual, though not desirable. The adhesive tapes used in this layer must be able to withstand these conditions without mentionable long term damage and additionally fulfil their performance requirements for the service life of the whole building. Lack of durability of these building products may result in energy loss and consequently increase CO₂ emissions and the costs of maintenance or replacement of building products, and any possible consequential building damage. In order to analyse the durability of joints, it is important to evaluate the adhesive and substrate properties, and evaluate the performance using accurate and reproducible durability test methods.
Standard test methods along with internationally accepted test procedures established by different adhesive manufacturing associations, like The European Association for the Self Adhesive Tape industry (AFERA), Pressure Sensitive Tape Council (PSTC) and the international association for the self-adhesive label industry (FINAT), have been used for evaluation of adhesive tapes [1, 2]. These test methods consider various types of stress as well as environmental conditions in which the adhesive tape will face during its service life. However, there remain significant differences between the test methods. For example, FINAT specifies glass as the most suitable test plate to evaluate the bond strength of the adhesive tapes due to smoothness and stable chemical nature of its surface, while AFERA recommends steel [1, 3]. The difference in the test methods can be due to additional or modifications of the test methods required representing the actual end use of adhesive tapes.

Specifically, there is lack of reliable test methods, accelerated ageing procedures and long term performance prediction methods for adhesive tapes used in buildings. Hence, long term performance prediction of adhesive tapes used on buildings, reliable durability test methods which consider the actual service condition is required. Accelerated ageing tests provide information about the durability of the adhesive tapes and joints (adhesive tapes/ substrate interface) to environmental stress within a reasonable timeframe by reproducing the basic degradation mechanisms that are expected to occur in long term real life in-use conditions.

This work presents test methods used by SINTEF Building and Infrastructure to evaluate the adhesive tapes used in Norwegian buildings. The effect of adhesive and surface properties and environmental factors are also presented in some detail since both the long term and short term performance of joints mostly depends on these factors.

2 BACKGROUND

2.1 Overview of the overall study

The work presented in this paper is one part of ongoing research project in SINTEF Building and Infrastructure, where evaluation of adhesive tapes durability testing, evaluation and service life estimation methods is the main focus area.

A test programme was designed to evaluate existing test methods by applying four different acrylic tapes (labelled as S, E, I and W) and different substrates; polyethylene based wind barrier membranes (WT), polypropylene based wind barrier membranes (WI), coated spruce (CS), uncoated spruce (S), glass (G), galvanized steel (GS) and vapour barrier membranes (VT). The test specimens were preconditioned at (23±0.2) °C and (50±3) % relative humidity before ageing.

Two ageing test series were used; 2 weeks of climate ageing followed by 24 weeks of heat ageing (Series1) and 24 weeks heat ageing (Series2). The performance of the test specimens before and after 2 weeks, 8 weeks, 12 weeks and 24 weeks of ageing were evaluated using tensile strength, peel resistance and shear resistance test methods. The experimental setup is summarized in Table 1.
Table 1: Experimental setup

<table>
<thead>
<tr>
<th>Standards</th>
<th>EN 12311</th>
<th>EN 12316-2</th>
<th>EN 12317-2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grip distance (mm)</td>
<td>120±2</td>
<td>100±2</td>
<td>200±2</td>
</tr>
<tr>
<td>Grip separation speed (mm/min)</td>
<td>100±10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample size (width x length)mm, for tapes and/or substrates</td>
<td>Tapes=(50x300)±0.5</td>
<td>Tapes=(50x300)±0.5</td>
<td>Tapes=(50x220)±0.5</td>
</tr>
</tbody>
</table>

ii. Accelerated ageing and test interval

<table>
<thead>
<tr>
<th>Ageing</th>
<th>Tensile strength, peel resistance and shear resistance tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>Series 1: Climate and heat ageing</td>
<td>Fresh</td>
</tr>
<tr>
<td>Series 2: Heat ageing</td>
<td>-</td>
</tr>
</tbody>
</table>

iii. Number of test specimen replicas used for each ageing series and test method

<table>
<thead>
<tr>
<th>Tapes</th>
<th>Fresh</th>
<th>Series 1</th>
<th>Series 2</th>
<th>Fresh</th>
<th>Series 1</th>
<th>Series 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>S, E, I, W</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Substrates</td>
<td>Peel resistance</td>
<td>Shear resistance</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fresh</td>
<td>Series 1</td>
<td>Series 2</td>
<td>Fresh</td>
<td>Series 1</td>
<td>Series 2</td>
<td></td>
</tr>
<tr>
<td>WT</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>WI</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>VT</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>G</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>GS</td>
<td>5</td>
<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
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</tr>
<tr>
<td>S</td>
<td>5</td>
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<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
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<tr>
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</tr>
<tr>
<td>WT</td>
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<td>5</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

Here, existing test procedures and evaluation methods is presented, the remaining results of the project will be presented elsewhere.

2.2 Factors influencing the durability of adhesive tapes

Adhesive tapes mainly consist of tacky adhesive coated on one or both sides of a backing or carrier material such as paper, polymeric film, cloth or metal foil. Natural or synthetic rubber, acrylic and silicon are the common adhesives used in adhesive tapes. Adhesive tapes are tacky because of viscoelastic properties of the adhesive. Because of their liquid property they can have some properties of water, so that they will wet a surface to which they are applied. They will also resist separation under stress due to their elasticity. The backing
provided the necessary strength to the adhesive tape and needs to have good tensile strength, elongation, conformability, moderate heat resistance, moisture and chemical resistance properties.

Thus, the performance of adhesion in the joints mainly depends on both the viscoelastic property of the adhesives and surface properties of the adhesive tape and substrate it is adhered to [1, 3-5]. In addition, most in-service failures of bonded joints are caused by environmental degradation of the interface between the adhesive tape and the substrate it is adhered to. In order to assess the durability of the joints, it is important to understand main factors that can influence the bond.

**Adhesion-cohesion balance:** The property of the adhesion bond depends on the proper balance between adhesion (holding power of the adhesive on substrates) and cohesion (holding power of the internal components of the adhesive) properties [1, 6]. Adhesive possesses adhesion, for bonding and debonding, and cohesion against debonding. The adhesion is characterized by tack and peel, whereas cohesion is described by shear resistance[4]. Tack represents the adhesive's ability to adhere quickly, peeling measures the resistance to peeling and shear measures its ability to resist removal. Therefore, the right balance between high tack, high peel adhesion and high shear resistance is one of the factors that influence the properties of adhesive tapes [5, 7, 8].

**Surface properties:** The physical properties and the chemical composition of the surface of adhesive and substrate has significant effect on the quality of the bond formed [4, 9]. Good wettablility is one of the requirements for good adhesion, as it increases the actual contact area between the surface of the substrate and the adhesive tape, and it depends mainly on the balance between the surface energy of adhesive and substrate.

Quantitative determination of surface energy of both substrates and adhesives is important to investigate the magnitude of adhesion and for selection of suitable substrate/adhesive pairs for bonding, but it is a difficult task. The determination of contact angle at the solid/liquid phase boundary is one of the methods used to indirectly estimate the surface energy of solid materials [9, 10].

For the adhesive to wet the substrate effectively (i.e, for a lower contact angle), the surface energy of the adhesive should be lower than the surface energy of the substrate [9]. Partial wetting and non-wetting conditions are characterized by high values of contact angle and with lower surface tension of the substrate. The texture and cleanliness of the surface are also other factors that can affect adhesion [4].

**Climate exposures:** Besides the properties of the adhesives and substrate, the end-use environment is another important factor which can affect the function of the adhesive tapes. Adhesive tapes used in buildings can be subjected to different climate strains including UV light, high or low temperatures and moisture. Ultraviolet light and heat are key factors that degrade adhesive tape. UV light may initiate the degradation process by breaking the bonds of the adhesive, while the degradation processes exponentially increasing with respect to increasing temperatures. Exposure to elevated temperature affects the adhesion by reducing the bond strength in joints. Water, in its various forms, is another factor which can lead to failure in bonded structure. Dissolution reactions and dilutions, mechanical degradation and freezing/thawing cycles may also contribute to this degradation.
3 DURABILITY TEST METHODS

The durability test method comprises standard test methods that consider environmental factors and stress to which the adhesive tapes can be subjected. The test specimens are exposed to environmental conditions in accelerated laboratory ageing for 26 weeks before tensile strength, peel resistance and shear resistance tests.

3.1 Test plates

The surface of the substrate or test plate plays a key role in the final adhesion properties [3]. Although the use of glass (by FINAT) and steel (by AFERA) are suggested as standard test plates, in order to evaluate the actual substrate/interface property main end-use substrates are used. Vapour barrier membranes, wind barrier membranes, wood, gypsum board and bitumen fibreboard are some of the substrates used for testing the adhesive tapes based on adhesive tapes specification.

3.2 Accelerated ageing

Adhesive tapes and joints are exposed for two weeks in a vertical climate simulator according to NT Build 495 [11]. The test apparatus consists of a rectangular rotating central unit where test specimens are mounted and subjected to four different climate strains: UV and IR radiation (at a black panel temperature of (63 ± 5) °C), water spray (15 ± 2 l/(m² h)), freezing ((-20 ± 5) °C), and thawing in ambient laboratory climate. The exposure time is 1 hour in each climate zone according to the above given order (Figure 1).

![Figure 1: Working principles of climate simulator [11].](image)

After the climate simulator exposure, the test specimens are exposed to 24 weeks of heat ageing in oven, at a temperature of 70 °C, according to EN 1296 [12].

3.3 Test methods

The test method was developed for testing mechanical properties of adhesive tapes applied in buildings by using standard test methods used for testing of roof membranes. Since
adhesive tapes are exposed to similar climatic conditions like roof membranes, these test methods are considered to be most relevant to adhesive tapes used for building purposes. Tensile strength of the adhesive tapes (Figure 2a) as well peel resistance (Figure 2b) and shear strength (Figure 2c) tests of the joints are evaluated in a tensile testing machine. The testing is performed before ageing, after 2 weeks of ageing in climate simulator, after 12 weeks of heat ageing and after 24 weeks of heat ageing. The test results are evaluated by calculating the percentage change caused by the exposure compared to fresh test specimens.

![Figure 2: Schematic diagram of tensile and elongation (a) peel resistance ((b) for flexible substrate (left) and rigid substrate (right)) and shear resistance (c) tests.](image)

The tensile strength of adhesive tapes is evaluated according to EN 12311-2 [13] standard test method. The test specimens are tightly clamped in a tensile test machine with a grips distance of (200±2) mm and tested at a constant grip separation speed of (100±10) mm/min. The test results present the maximum tensile force in N/50mm and the corresponding elongation in percentage.

In order to investigate the performance of the joints, peel resistance and shear resistance tests are performed for adhesive tape glued to essential end-use substrates according to EN 12316-2 [14] and EN 12317-2 [15] standard test methods, respectively.

For the peel resistance test, the test specimens are tightly clamped in a tensile test machine with a grips distance of (100 ± 2) mm and tested at a constant grip separation speed of (100 ± 10) mm/min. 180 degree peel tests are used when an adhesive tape is adhered to a more rigid substrate while T-peel is used for tape applied to thin, flexible substrates. The mean peel resistance value expressed in N/50 mm is used to evaluate the adhesion performance of the joints.

The shear resistance test is performed in a tensile tester with a grips distance of (200 ± 2) mm and a constant grip separation speed of (100 ± 10). The shear resistance of the joint is the maximum force required until the joint breaks or separates.

3.4 Service life estimation

Service life is defined as the period of time after installation during which a building or its parts meets or exceeds the performance requirements [16]. Service life prediction methods can be used by manufacturers to offer warranties on performance of their products to
customers. One of the critical aspects of durability testing is prediction of the equivalent service life from accelerated ageing tests. Actually, it is not easy to give a precise comparison between artificial and natural aging as the real-life service conditions of products cannot be predicted or accurately simulated.

The rate of degradation in the accelerated ageing test chamber can be compared with actual outdoor exposure using an acceleration factor, a number correlating the ageing time in the test chamber with actual natural outdoor ageing exposure [17, 18].

With some uncertainty, an artificial aging of 2 weeks in climate simulator and 24 weeks in heat chamber at 70 °C can be compared to approximately 25 years of natural exposure.

4 CONCLUSIONS

This work presents test methods used to evaluate the long term performance of adhesive tapes used in buildings. Accelerated ageing procedure and tensile, shear and peel test methods along with corresponding standards have been discussed. Further work will include investigation of the durability test and evaluation methods based on the test results from the experimental work.

ACKNOWLEDGEMENTS

This paper is developed as part of BEST project, and we gratefully acknowledge the financial support and contribution from colleagues at Sintef Building and Infrastructure.

REFERENCES


INVESTIGATION METHODS OF THE RESISTANCE OF UHPC TO CHEMICAL ATTACK AND ITS CLASSIFICATION IN THE EXPOSURE CLASSES OF THE EUROPEAN STANDARD

Dr.-Ing. Holger Schmidt (1), Prof. Dr.-Ing. Frank Schmidt-Döhl (2), Prof. Dr.-Ing. Lutz Franke (2) and Dr.-Ing. Gernod Deckelmann (2)

(1) MC-Bauchemie Brasil, São Paulo – holger.schmidt@mc-bauchemie.com.br
(2) Institute of Materials, Physics and Chemistry of Buildings; Hamburg University of Technology, Hamburg - frank.schmidt-doehl@tuhh.de; lutz.franke@tuhh.de; gernod.deckelmann@tuhh.de;

Abstract

The meaning of the behaviour of ordinary concrete and UHPC with respect to chemical attack is characterized and discussed in this work. Hereby of special interest is the discussion of the possibility of using UHPC without additional external protection, e.g. coatings, in settings of higher acid and salt concentrations than exposure class XA3 of the European standard EN 206. In this context the difference in durability of an ordinary reference mortar and various UHPC mixtures will be examined. The experimental results show a higher resistance of the considered UHPC mixtures against acid and salt solutions as an ordinary concrete or high performance concrete. To predict the damage depth for acid attacks a new method was developed with the possibility to determine effective equivalent pH values of the UHPC mixtures in comparison with an ordinary mortar, which attends completely the requirements of exposure class XA3. The final results of investigations show the recommendation of a new exposure class for chemical attacks (XA4) in attendance of the European standard EN 206. Furthermore the ultra high performance concrete could be used in the exposure class XA3 without any additional protection system.

1 INTRODUCTION

In the construction industry ultra high performance concrete (UHPC) is becoming increasingly important. Until now it has been regarded as a material with high durability. The aim of the research project is to evaluate if UHPC has a defined higher resistance as an ordinary concrete or high performance concrete with respect to the durability. In the test series the behaviour of UHPC against sulphuric acid, lactic acid, sulphate solutions and ammonia solutions was investigated. These experiments were carried out on various UHPC mixtures in comparison with an ordinary reference mortar, which attends the requirements of XA3.

Furthermore were carried out investigations to the influence of the composition and curing of the UHPC mixtures with respect to durability. Also was elaborated a new method to evaluate the chemical behaviour of UHPC and its classification in the exposure classes XA of the DIN EN 206-1. Because of the significant higher resistance of UHPC to chemical attacks
are recommended modifications of the classes. It is considered in the evaluation method, that UHPC mixtures can be a “black box”, which means the lack of knowledge of its composition. In view of a guideline of UHPC mixtures should be answered the question:

- How can be classified the UHPC mixtures in the exposure classes?

2 INVESTIGATIONS AND RESULTS OF THE UNDAMGED MATERIALS

All experiments were carried out with different UHPC mixtures and an ordinary reference mortar. The ordinary reference mortar (REF) consists of Portland cement CEM I 42.5 R-HS and is produced according to Sielbau-Richtlinie [3]. This mortar complies with the requirements of exposure class XA3 of DIN EN 206. The powder of the UHPC mixtures M2Q and B4Q is composed of ordinary Portland cement CEM I 52.5 R-HS/NA, microsilica and quartz powder. The fineness of the quartz powder is between silica fume and cement. It was used as a micro filler to optimize the packing density of the powder mixture. The mixture H75-1 is optimized by adding granulated slag. Superplasticizer on the basis of polycarboxylateether ensured the flowability of the investigated compacted UHPC. Table 1 gives more details on the composition of the ordinary reference mortar and the UHPC mixtures. On the UHPC mixtures was applied a curing under water (WL) and a thermal curing (90), more details are in [1]

<table>
<thead>
<tr>
<th>Material</th>
<th>unit</th>
<th>Ord. mortar</th>
<th>M2Q 90 / WL</th>
<th>B4Q 90</th>
<th>H75-1 90</th>
</tr>
</thead>
<tbody>
<tr>
<td>cement</td>
<td>kg/m³</td>
<td>832</td>
<td>650</td>
<td>222.5</td>
<td></td>
</tr>
<tr>
<td>cement CEM I 52.5 R-HS/NA</td>
<td>kg/m³</td>
<td>512</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>cement CEM I 42.5 R-HS</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>aggregates</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CEN sand</td>
<td>kg/m³</td>
<td>1536</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>quartz sand 0,125/0,5</td>
<td>kg/m³</td>
<td>975</td>
<td>354</td>
<td>1000.9</td>
<td></td>
</tr>
<tr>
<td>basalt split 2/8</td>
<td>kg/m³</td>
<td>597</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>additives</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>quartz powder Q I</td>
<td>kg/m³</td>
<td>207</td>
<td>325</td>
<td>221.4</td>
<td></td>
</tr>
<tr>
<td>quartz powder Q II</td>
<td>kg/m³</td>
<td>131</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>microsilica</td>
<td>kg/m³</td>
<td>135</td>
<td>177</td>
<td>144.4</td>
<td></td>
</tr>
<tr>
<td>granulated slag</td>
<td>kg/m³</td>
<td></td>
<td></td>
<td>602.9</td>
<td></td>
</tr>
<tr>
<td>admixtures</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>superplasticizer</td>
<td>kg/m³</td>
<td>29.4</td>
<td>15.2</td>
<td>13.3</td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>water to cement ratio (w/c)</td>
<td>0.45</td>
<td>0.20</td>
<td>0.24</td>
<td>0.81</td>
<td></td>
</tr>
<tr>
<td>water to binder ratio (w/b)</td>
<td>0.45</td>
<td>0.17</td>
<td>0.19</td>
<td>0.24</td>
<td></td>
</tr>
</tbody>
</table>

Furthermore are investigated commercial UHPC products (K1, K2 and K3) with unknown composition.
Of all materials was determined the characterization in the zero state. All investigations were applied in the age of 28 days of the specimens.

- Compressive and flexural strength
- Value of free Ca(OH)2 (Portlandit) by DTA/TG
- total porosity by the bulk density using the dipping and weighing method and the skeletal density using helium pycnometer
- porosity and the distribution of pore radio by mercury intrusion porosimetry. The capillary pore fraction is in the range of 1 µm to 25 nm and the fraction of raw pores in the range of 1 to 10 µm.

The values of compressive strength and the porosity values are presented in Table 2. The concentration of calcium hydroxide was determined via differential thermal and thermogravimetric analysis (DTA/TG) for all UHPC mixtures. As expected a low value of 0.2% was detected in all specimens of ultra high performance concrete via this testing method. In contrast the value of portlandit measured for the ordinary reference mortar amounts to about 19 mass percent.

Table 2: Measured material parameters (age 28 days) (data partly from [1])

<table>
<thead>
<tr>
<th>Material</th>
<th>Compressive strength [MPa]</th>
<th>Total porosity [Vol.-%]</th>
<th>Fraction gel pores [Vol.-%]</th>
<th>Fraction capillary pores [Vol.-%]</th>
<th>Fraction raw pores [Vol.-%]</th>
<th>Hg-porosity (MIP) [Vol.-%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>M2Q 90</td>
<td>192</td>
<td>11.89</td>
<td>11.03</td>
<td>0.76</td>
<td>0.22</td>
<td>4.61</td>
</tr>
<tr>
<td>M2Q WL</td>
<td>188</td>
<td>11.64</td>
<td>10.77</td>
<td>0.73</td>
<td>0.14</td>
<td>8.05</td>
</tr>
<tr>
<td>B4Q 90</td>
<td>192</td>
<td>12.81</td>
<td>11.94</td>
<td>0.71</td>
<td>0.16</td>
<td>4.93</td>
</tr>
<tr>
<td>H75-1</td>
<td>174</td>
<td>12.32</td>
<td>12.08</td>
<td>0.12</td>
<td>0.12</td>
<td>1.97</td>
</tr>
<tr>
<td>K1</td>
<td>194</td>
<td>18.38</td>
<td>14.05</td>
<td>3.46</td>
<td>0.87</td>
<td>8.90</td>
</tr>
<tr>
<td>K2</td>
<td>148</td>
<td>11.77</td>
<td>11.02</td>
<td>0.62</td>
<td>0.13</td>
<td>10.03</td>
</tr>
<tr>
<td>K3</td>
<td>147</td>
<td>9.56</td>
<td>7.15</td>
<td>2.01</td>
<td>0.40</td>
<td>7.31</td>
</tr>
<tr>
<td>REF</td>
<td>63</td>
<td>18.66</td>
<td>8.23</td>
<td>9.90</td>
<td>0.53</td>
<td>15.60</td>
</tr>
</tbody>
</table>

More detailed information about the phase composition (XRD), element analyses (XRF spectroscopy) and the transport coefficients (capillary water absorption, water vapour diffusion, ionic diffusion) in the undamaged building material matrix are in [1].

3 INVESTIGATION METHODS AND RESULTS OF DURABILITY TESTS

To characterise the resistance of ultra high performance concrete to acid or salt solutions, “corrosion” experiments were carried out. Various UHPC mixtures and one ordinary reference mortar were investigated. The experimental design is divided into two parts: experiments by using static boundary conditions and experiments by using non static boundary conditions. The test program and the results of the experiments in static boundary conditions are shown in the following. More details about the experiments by using non static boundary conditions are in [1], [4] and [5].
An important part of the investigations are experiments with static boundary conditions according to Sielbau-Richtlinie [3]. The specimens (40 x 40 x 80 mm³) of ultra high performance concrete and ordinary reference mortar are immersed in aggressive acid and salt solutions. This testing method was also used in [6] and [7]. The exposure time of each experiment is set to more than 4000 hours. The aggressive solution has a constant temperature of T = 21.5 °C and is constantly stirred by using magnetic mixers. In this article are presented the results about the damaged zone caused by acid attack. There are a lot of investigations to the determination of transport parameters and the distribution of elements and phases in the damaged zone additionally to these results. [1] is reporting in detail about these results.

3.1 Method of proton consumption and determination of damage depth

In this research project was developed the method of consumptions of protons to determine the damaged zone after an attack of acid. It is based on the recorded consumption of protons in the immersing experiment and the total consumption of protons of a powered specimen. Sulphuric acid and lactic acid are used to investigate the behaviour of the specimens with respect to acid attack in these investigations. By using an automatic titration system (MultiT) the pH value is kept constant and the consumption of protons is recorded. The investigations are performed via the following acids in detail:

- chemical attack by using sulphuric acid with a pH value of 3, 4 and 5
- chemical attack by using lactic acid with a pH value of 4 and 5

The advantages of this special method are the easy and practical application, the knowledge of the time depended development of the damage depth X_{VG} during the investigation and the easy classification of the damage depths in the exposure classes XA. It is possible to extrapolate the damage depth for various years. A detailed description of this method is given in [1] and [2]. This method is also used in the future German standard DIN 19573 (‘Mortar for construction and renovation of drainage systems outside of buildings’).

[2] shows a lot of examples of the proton consumption curves about a period of more than 2 years. To minimize the influence of foreign ions in the acid, the acidic solution is renewed every 1000 hours during the complete exposure time. At the end of the experiment the specimens are brushed to remove incoherent particles. The alteration of the external dimensions is represented by the depth X_{A}. The visual neutralization depth X_{V,Ph} is measured by using the phenolphthalein method and light microscope.

It is necessary to evaluate in combination the proton consumption of the powered specimen to discuss the resistance to acid of the specimens. So it is possible to determine the damage depth X_{VG}, which is used for evaluation and classification.

The damage depth X_{VG} in mm by using the method of proton consumption is calculating by the following equation.

\[ X_{VG} = \frac{H_{M,t} \cdot 100}{H^*M \rho_{M, tr}} \]  

In this equation is H^*M [mol H⁺/kg_{mortal}] the total consumption of protons of a powdered specimen after drying at 105°C. The dissolution of the powdered mortar is realized by using the equal constant pH value like in the immersing experiment. \( \rho_{M, tr} \) [kg/dm³] is the dry bulk density.
H_{Mt} [mol H⁺/dm²] is the consumption of protons of the immersed specimens in acid by using a constant pH value during an interested exposure time.

\[
H_{Mt} = n_{prot,S} \cdot \alpha_{diss,S}(pH_{Bad}) \cdot \frac{m_s}{M_S} \tag{2}
\]

\[
m_s = \frac{V_{gem}}{A_0} \cdot c_{S,verd} \left( \frac{\%}{100} \right) \cdot \rho_{S,verd} \tag{3}
\]

In these equations are \(n_{prot,S}\) the quantity of dissociated protons in [\(-\)], \(\alpha_{diss,S}(pH_{Bad})\) the dissociation degree of the attacking acid in [\(-\)], \(M_S\) the molar mass of the attacking acid in [g/mol], \(V_{gem}\) the volume of titrated acid in [mL], \(A_0\) the reactive surface of the immersed specimens in [cm²], \(c_{S,verd}\) [\(\%\)] the concentration of acid in [\%] and \(\rho_{S,verd}\) the density of the diluted acid in [g/cm³].

In general the damage depth calculated by equation (1) is more reliably than the determination of the neutralization depth by an indicator like phenolphthalein.

3.2 Attack by sulphuric acid

In the following are presented examples of results after an attack by sulphuric acid after 4000 hours. This type of degradation is accompanied by the dissolution of the cementitious binder matrix and the formation of new mineral phases like gypsum and ettringite. The following Tables 3 and 4 show the damage depth \(X_{VG}\) by the method of consumption of protons, the visual neutralization depth \(X_{V,Ph}\) by the phenolphthalein method and the consumption of protons \(H_{Mt}\) and \(H^*_M\).

**Table 3: Results with sulphuric acid pH3 after 4000 h (data partly from [1])**

<table>
<thead>
<tr>
<th>Results</th>
<th>unit</th>
<th>M2Q90</th>
<th>M2Q WL</th>
<th>B4Q90</th>
<th>H75-1</th>
<th>K1</th>
<th>K2</th>
<th>K3</th>
<th>REF</th>
</tr>
</thead>
<tbody>
<tr>
<td>(X_{VG})</td>
<td>mm</td>
<td>0.86</td>
<td>0.79</td>
<td>1.1</td>
<td>0.89</td>
<td>1.13</td>
<td>1.05</td>
<td>1.31</td>
<td>1.57</td>
</tr>
<tr>
<td>(X_{V,Ph})</td>
<td>mm</td>
<td>0.8</td>
<td>0.8</td>
<td>-</td>
<td>0.9</td>
<td>0.9</td>
<td>1.0</td>
<td>1.0</td>
<td>1.5</td>
</tr>
<tr>
<td>(H_{Mt})</td>
<td>molH⁺/dm²</td>
<td>0.1731</td>
<td>0.1526</td>
<td>0.1888</td>
<td>0.1825</td>
<td>0.2554</td>
<td>0.1966</td>
<td>0.183</td>
<td>0.194</td>
</tr>
<tr>
<td>(H^*_M)</td>
<td>gH⁺/kgmortar</td>
<td>9.085</td>
<td>8.852</td>
<td>7.076</td>
<td>9.00</td>
<td>9.2</td>
<td>8.75</td>
<td>6.12</td>
<td>5.86</td>
</tr>
</tbody>
</table>

**Table 4: Results with sulphuric acid pH4 after 4000 h (data partly from [1])**

<table>
<thead>
<tr>
<th>Results</th>
<th>unit</th>
<th>M2Q90</th>
<th>M2Q WL</th>
<th>B4Q90</th>
<th>H75-1</th>
<th>K1</th>
<th>K2</th>
<th>K3</th>
<th>REF</th>
</tr>
</thead>
<tbody>
<tr>
<td>(X_{VG})</td>
<td>mm</td>
<td>0.52</td>
<td>0.46</td>
<td>0.59</td>
<td>0.3</td>
<td>0.49</td>
<td>0.46</td>
<td>0.67</td>
<td>1.06</td>
</tr>
<tr>
<td>(X_{V,Ph})</td>
<td>mm</td>
<td>-</td>
<td>0.4</td>
<td>-</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>-</td>
</tr>
<tr>
<td>(H_{Mt})</td>
<td>molH⁺/dm²</td>
<td>0.0917</td>
<td>0.0858</td>
<td>0.0901</td>
<td>0.0519</td>
<td>0.1070</td>
<td>0.077</td>
<td>0.0828</td>
<td>0.111</td>
</tr>
<tr>
<td>(H^*_M)</td>
<td>gH⁺/kgmortar</td>
<td>8.048</td>
<td>8.395</td>
<td>6.132</td>
<td>7.452</td>
<td>8.84</td>
<td>7.87</td>
<td>5.43</td>
<td>4.980</td>
</tr>
</tbody>
</table>

4 EVALUATION OF RESULTS TO CHEMICAL ATTACK

There are presented two graphics summarizing the results with sulphuric acid pH3 and pH4 after 4000 h to realize conclusions of the UHPC mixtures about of the resistance to chemical attacks. Results of the other investigations like lactic acid, ammonia solutions and sulphate solutions are presented in [1].
All investigated UHPC mixtures have a compressive strength of at least 150 N/mm². A direct correlation of the compressive strength about 28 days with the damage depth could not be found. But it can be summarized that the maximum damage depth of the “building material” UHPC after 4000 hours for pH3 is 1.3 mm and for pH4 it is 0.67 mm. The damage depths of the reference mortar, which attends the requirements of the maximum exposure class XA3, are 40% higher than the damage depth of UHPC in sulphuric acid pH3 and circa 65% higher for pH4. Thus, the building material UHPC has a defined higher resistance than an ordinary concrete with respect to chemical attacks. However, this conclusion can only be assumed if the high compressive strength of the material of at least 150 N/mm² is achieved by low water-binder-ratio and an optimization of the packing density. This must be clearly emphasized, as well as other building materials with higher water-binder-ratios are possible, which can be achieve the high compressive strength of UHPC. But, the low permeability and higher durability are not ensured.

Figure 1: Results of immersing experiment with sulphuric acid pH3 after 4000h

Figure 2: Results of immersing experiment with sulphuric acid pH4 after 4000h

Another aim of the project is the relevance for the praxis. For that is developed a method for classification of the building material UHPC in the exposure classes XA (chemical attack) of the European standard. The concept, which is linked to the method of the consumption of protons, is described in detail in [2] and [1].
The evaluation concept for the building material UHPC in case of chemical attack is based on equivalence or rather comparative survey of typical damage depths according to the standardized concrete in the exposure classes XA of the European standard. Typical damage depths are determined by using the reference mortar (REF), which corresponds to the requirements of the exposure class XA3 (w/c = 0.45; cement ≥ 320 kg/m³; CEM I 42.5R HS/NA; compressive strength ≥ C35/45). The question about the classification of UHPC in the exposure classes without laboratory testing and without exact knowledge of the composition of commercial products, which is posed in the beginning, can be answered with the evaluation concept as follows.

A quick look at the current standardization to the boundary conditions of the exposure classes XA for resistance to acidic attacks shows that the exposure class XA2 is applied in the range of < pH5.5 and ≥ pH4.5 and the exposure class XA3 for the scope of < pH4.5 to ≥ pH4. But for the exposure class XA3 the utilization of additional protective measures is necessary like for example plastic coatings.

With the presented results above, the UHPC based on a very low water-binder-ratio and an optimization of the packing density with a compressive strength of at least 150 N/mm² after 28 days can be used without any kind of additional protection systems in the conditions of exposure class XA3 for acid attacks.

Furthermore, it may be suggested, the exposure class XA4 for the acidic range. Here the building material UHPC can be used for the scope of pH4 to pH3.5 regularly, but only by using additional protective measures in accordance with the current regulation at XA3. This additional safety measure can be omitted with a corresponding experimental proof of equivalent performance by using the method of the proton consumption, as described in [1] and [2]. The compilation of relevant data for such an equivalence analysis is exemplified in the following figure.

![Figure 3: Damage depth of different UHPC and the reference mortar in sulphuric acid as a function of the pH value after 4000 hours](image-url)
The effective equivalent pH values of several UHPC are documented in the following table. That means that the damage depths of the materials are equal at the pH values given in one row. For example, the UHPC mixture H75-1 has the same damage depth with a pH value of 3.00 like the reference mortar (REF) with a pH value of 4.5.

Table 5: Material specific pH values with equal damage potential, measured by the method of proton consumption (data partly from [1])

<table>
<thead>
<tr>
<th>pH value</th>
<th>REF</th>
<th>M2Q90</th>
<th>M2Q WL</th>
<th>B4Q90</th>
<th>H75-1</th>
<th>K1</th>
<th>K2</th>
<th>K3</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.5</td>
<td>4.5</td>
<td>3.09</td>
<td>2.80</td>
<td>3.39</td>
<td>3.00</td>
<td>3.29</td>
<td>3.20</td>
<td>3.51</td>
</tr>
<tr>
<td>4.0</td>
<td>4.0</td>
<td>2.79</td>
<td>2.51</td>
<td>3.08</td>
<td>2.71</td>
<td>3.08</td>
<td>2.99</td>
<td>3.30</td>
</tr>
</tbody>
</table>

5 CONCLUSIONS

The experimental results show that the investigated UHPCs have an increased resistance compared to ordinary strength concrete to chemical attack. Based on the proton consumption of the immersing experiments in acidic waters a practical test method (method of proton consumption) is developed. It could be determined the effective equivalent pH values for the UHPCs. The classification in the exposure classes according to DIN EN 206 is realized by using this procedure. It could be concluded, that the ultra high performance concrete can be used in the exposure class XA3 without additional protective measures. Furthermore, is also proposed an exposure class XA4 for the attack of acid, which regulates the use of UHPCs up to pH3.5.

ACKNOWLEDGEMENTS

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REFERENCES

REDUCTION OF AUTOGENOUS SHRINKAGE IN OPC AND BFSC PASTES WITH INTERNAL CURING

Mateusz Wyrzykowski (1,2), Pietro Lura (1,3)

(1) Concrete/Construction Chemistry Lab, Empa, Swiss Federal Laboratories for Materials Science and Technology, Duebendorf, Switzerland – mateusz.wyrzykowski@empa.ch
(2) Department of Building Physics and Building Materials, Lodz University of Technology, Lodz, Poland
(3) Institute for Building Materials, ETH Zurich, Zurich, Switzerland – pietro.lura@empa.ch

Abstract
Self-desiccation occurring in low water-to-cement ratio (w/c) concretes and the resulting autogenous shrinkage may lead to early-age cracking and consequently jeopardize the durability and service life of high performance concrete elements. Among different methods developed to reduce the negative effects of self-desiccation, internal curing by means of superabsorbent polymers (SAP) recently gained a lot of attention. In this work, influence of SAP on the performance of cement pastes and mortars of w/c 0.30 prepared with Ordinary Portland Cement (OPC) or Blast Furnace Slag Cement (BFSC) is studied.

In particular, self-desiccation and autogenous shrinkage, and their reduction thanks to SAP addition, are investigated. Internal relative humidity (RH) evolution during early stages of hydration is measured using water activity sensors. It is observed that addition of SAP reduces or even practically eliminates the RH depression. As a consequence, autogenous shrinkage (measured using a linear method with corrugated tubes) is also considerably limited and even some initial expansion is promoted in the case of OPC. For BFSC, a considerable part of autogenous shrinkage is eliminated, but the remaining shrinkage is still very high. In addition, the effect of internal curing for the two types of cement, regarding the influence on hydration, mechanical properties and microstructure is also investigated.

1 INTRODUCTION

In High Performance Concrete (HPC), due to its low w/c and fine pore structure, the RH decrease (or self-desiccation) and the resulting autogenous shrinkage [1] may lead to macro- and microcracking and as a consequence seriously jeopardize the performance and the durability. One of the methods to mitigate autogenous RH decrease and shrinkage is internal curing with SAP [2]. In this method, SAP are added dry to the mixture and absorb water during mixing. This water is then gradually released from the SAP and distributed in the hardening concrete due to the action of capillary forces arising as the material undergoes self-desiccation. Internal curing water compensates for the chemical shrinkage and allows for
maintaining high levels of saturation in the pores and consequently high RH and limited shrinkage [3, 4]. At the same time, SAP have been found to promote development of hydration due to the effect of additional water entrained into the system [2, 5]. Further advantages due to addition of SAP are improved frost resistance [6, 7] and reduced thermal expansion [8].

Advantages of using SAP in cementitious systems made with OPC has been already well recognized in a series of publications, e.g. [2, 3, 8]. At the same time, BFSC have been shown [9-11] to induce considerable deformations due to autogenous shrinkage. Therefore, the possibility of applying internal curing with this type of cement is the main motivation for the work presented here. Internal curing with light weight aggregates (LWA) in concretes with blended OPC and BFSC has been shown to limit the autogenous shrinkage [9], however no published study could be found on the action of SAP in pure BFSC systems. One of the crucial factors in terms of internal curing efficiency is swelling capacity of SAP when mixed with cement and water. This property will govern the sizes of the water reservoirs and consequently also the efficiency of internal curing. The maximum swelling capacity occurs when the SAP absorb pure (deionised) water [12]. On the other hand, already during concrete mixing, rapid dissolution of alkali-sulphate cement phases will lead to especially high concentrations of K, Na and S in the mixing water; these ions will limit the absorption capacity of the SAP compared to pure water. It is noted that the composition of the pore solution and therefore the swelling capacity of the SAP can be expected to differ between OPC and BFSC [9, 13].

In this work, the effect of internal curing with SAP was compared for cement pastes of basic w/c of 0.3 prepared with two types of cement: OPC and BFSC. The tests performed focus on reducing autogenous phenomena: self-desiccation and self-desiccation shrinkage. Self-desiccation was measured using water-activity sensors and shrinkage using the corrugated tube method [14] with automatic deformation measurements, similarly as in [15]. In addition, the heat evolution was measured with isothermal calorimetry to determine the influence of additional water on the hydration process. The static elastic modulus allowed studying the effect of entrained SAP and the resulting increase in porosity on the mechanical properties, see e.g. [4]. Finally, the microstructure of the cement pastes with SAP addition was studied using Back Scattered Electron Microscopy (BSE) with a focus on the pores being the remnants of the swollen SAP.

RH measurements showed that self-desiccation can be eliminated when SAP are added. For OPC pastes, autogenous shrinkage was practically eliminated and some slight expansion occurred. For BFSC pastes, very high autogenous shrinkage was observed in the reference system, which was reduced considerably due to SAP addition, but remained high nevertheless. The effect of SAP on the E-modulus was similar for both types of cement, with slightly higher reduction for BFSC. A clear improvement of SAP on the hydration evolution could be observed in the OPC system, while hardly any influence could be found for BFSC. No clear difference could be detected regarding the size of the macropores (the remnants of the swollen SAP) observed with BSE for both types of cement. It is therefore deduced that absorption of pore solution by SAP was similar for the two cement types. Based on the presented results it can be concluded that a positive effect of SAP addition could be observed for the applied BFSC. Nevertheless, special care should be taken regarding cement composition and SAP when cements different than OPC are used.
2 MATERIALS AND METHODS

2.1 Materials and mixture composition
Cement pastes were prepared using cements CEM I 42.5 N (OPC) and blast-furnace slag cement CEM III/B 32.5 N (BFSC). Deionized water was used and 0.5% (by weight of cement) of polycarboxylate-based superplasticizer was incorporated into the mixing water. Reference mixtures (with no SAP, denoted as REF) had w/c of 0.3, while for mixtures with SAP (denoted as SAP) additional water was accounted for internal curing and the total w/c was equal to 0.35. Solution-polymerized SAP with irregular particles of size 63–125 μm were used, with absorption of 16 g/g of dry SAP [5] in OPC pastes.
Mixing was performed using a vacuum mixer (Twister Evolution) with a 0.5-l vessel. 2 min of wet mixing under vacuum at 450 rpm followed dry mixing of cement (and SAP when applicable). Mixing and all experiments were performed in controlled-climate rooms at 20 ± 0.5 °C and 70 ± 5 %RH.

2.2 Linear autogenous shrinkage
Linear autogenous shrinkage of cement pastes was measured using corrugated tubes [14] with an automatic measuring bench equipped with LVDTs, see [15]. Directly after casting in the corrugated tubes, two specimens per each mixture (obtained from independent mixings) were inserted in an automatic dilatometer immersed in silicone oil bath. The oil bath minimized the risk of water loss from the tubes and helped maintaining constant temperature of the setup and the samples. The measurements started about 1 h after casting; nevertheless, the measurements were zeroed at final set to comply with ASTM standard [14].
The temperature evolution in the middle of a companion corrugated tube during maturing was measured with termocouples in the w/c 0.30 cement paste.

2.3 Internal RH evolution
Measurements of internal RH were performed using water activity sensors (AW-DIO by Rotronic). The nominal accuracy of the sensors was ±1 %RH. The sensors were calibrated at the beginning and at the end of each measurement with three saturated salt solutions with equilibrium RH in the range 85-98%. The temperature of the sample and the sensor was maintained at 20 ± 0.05 °C by means of water circulating in the casings of the measuring chambers and a thermostat-controlled bath.
After mixing, the pastes were poured in sealed plastic containers. At the age of approximately 1 day the samples were crushed into pieces of about 5 mm and about 8 g of crushed material was used for each station (two stations in parallel measured one mixture).
The mass loss in the specimens after the measurement did not exceed 0.05 % of the initial weight of the sample.

2.4 Hydration evolution
The rate of heat release was measured on cement pastes with isothermal calorimetry at 20°C using a TAM Air isothermal calorimeter (Thermometric AB). About 6 g of freshly-mixed paste was put into glass vials, closed and placed in the measuring cells. Due to the external mixing, the initial heat peak immediately after the addition of water to cement could not be measured and the measurements started at 1 h after water addition. Two specimens for
each cement paste were tested, resulting in a difference for the ultimate value of the cumulative heat of hydration of less than 2% for OPC and 9% for BFSC.

2.5 Scanning electron microscopy
7 days old samples were cut with a diamond precise saw and impregnated with epoxy resin under vacuum for 30 min. After hardening of the resin, the sections were polished using diamond suspensions. The samples were carbon coated and studied using a Scanning Electron Microscope (SEM, Fei NovaNanoSEM 230) with Back Scattered Electron (BSE) detector at 15 kV and Energy-dispersive X-ray spectroscopy (EDX).

3 EXPERIMENTAL RESULTS

3.1 Evolution of hydration degree
As can be seen in Fig. 1a, the application of internal curing has promotes hydration at later ages, i.e., starting from 1 – 1.5 days for OPC pastes. A similar result has been recently reported in [5], where it was shown that the effect of water entrained within the SAP is similar to a simple increase in w/c (without adding SAP) in terms of the final degree of hydration reached. On the other hand, the effect of SAP on hydration of BFSC was surprisingly negligible. It thus seems that entraining water could not promote the hydration process of the BFSC. The reason for such behavior is not clear. One possible explanation is that the hydration of BFSC is less influenced by RH than that of OPC.

3.2 Evolution of elastic modulus
In Fig. 1b the influence of SAP on the evolution of elastic moduli is presented. The increase of porosity due to the application of SAP leads to a visible reduction of mechanical properties. For the case of OPC it was found in [8] that the effect of an increase in the initial porosity due to internal curing water addition is very similar to a conventional increase of w/c. For BFSC, a slightly higher reduction of the elastic modulus was observed. This may be explained by the absence of a promotion of the hydration degree for the BFSC paste (as discussed in section 3.1), whereas further hydration allowed for a partial compensation of the increased initial porosity in the OPC paste.
3.3 Self-desiccation

Fig. 2. a) Internal RH evolution in cement pastes with OPC and BFSC measured on duplicate samples. For specimens with SAP the reading was 100% RH over the whole period of measurement (presented together with the error bars showing nominal accuracy of the sensors); b) autogenous deformation in cement pastes with OPC and BFSC measured on duplicate specimens. Time from water addition.

While self-desiccation can be clearly observed for reference cement pastes made with the two types of cement, for the pastes with SAP addition readings were 100%RH along the whole period of measurements. Readings at such high RH levels may have been the effect of vapor condensation on the sensors. As soon as this happens, the sensor needs to be dried and the measurement is invalid. Nevertheless, the results clearly show that self-desiccation in materials with SAP addition can be effectively reduced, at least at the initial stages of hardening.

3.4 Self-desiccation shrinkage

In Fig. 2a autogenous deformation results are presented. Referencing the deformations to the length at final setting is a standard, however very unreliable procedure. The high scatter in setting time usually obtained with the Vicat needle will have a large influence on the absolute value of shrinkage, since the rate of shrinkage is generally high around setting time. For example, a 10-min uncertainty in the determined setting time corresponds to a shift of the deformation curve by about 40 $\mu$m/m for the OPC-SAP cement paste, see [8]. Moreover, shrinkage occurring around setting time as determined with the Vicat needle is of lower importance in terms of the risk of microcracking. The high compliance of the material at this stage can lead to pronounced deformation even under very low forces, but at the same this deformation is not expected to lead to the buildup of considerable self-induced stresses.

In order to overcome this artifact, we referenced the shrinkage deformations to the point where the very steep initial shrinkage phase, starting from casting and continuing until about 0-4h after the setting time determined with Vicat needle, ends. A change of the slope of shrinkage curve could be clearly resolved at that time point.

One of the possible origins of expansion occurring short after setting for OPC-REF, OPC-SAP and BFSC-SAP pastes may be re-absorption of bleeding water [16]. Some bleeding could be observed on the surface of sealed specimens during the initial stages of maturing. Thermal expansion should not contribute considerably to the expansion since the temperature increase in the middle of the corrugated tube filled with w/c 0.3 cement paste occurring after
setting was $0.5 \pm 0.1 \, ^\circ\text{C}$ (average of two samples) with the temperature peak at about 16 hours after water addition. For a coefficient of thermal expansion of the OPC-REF paste equal to 16 $\mu\text{m/m/}^\circ\text{C}$ [8] this corresponds to a maximum expansion of only 8 $\mu\text{m/m}$. Sant et al. [17] investigated crystallization pressure of calcium hydroxide (CH) as one possible mechanism contributing to swelling of cement pastes after setting.

It can be seen for all materials, that after approximately 1.5 day of hardening, self-desiccation shrinkage started surpassing the competitive swelling. For both types of cement the effect of SAP is clearly visible in Fig. 2b. For OPC pastes, the shrinkage deformations occurring after the aforementioned expansion phase in the reference paste were limited when SAP were added and only very moderate shrinkage occurred. For BFSC pastes, the effect of SAP addition is also clearly visible, however the amount of internal curing was not sufficient to completely eliminate the very high autogenous shrinkage deformations, even though self-desiccation seemed to be eliminated, see Fig. 1a.

### 3.5 Microscopy study

The negligible influence of SAP on the hydration and only partial reduction of autogenous shrinkage for BFSC pastes leads to questioning the reasons for the reduced efficiency of SAP with this type of cement. Such question was especially relevant since no data on absorption of SAP in pastes prepared with BFSC was available (the 16 g/g absorption was obtained for OPC only [5]). This is why a microscopy study of the two cement pastes was performed, Fig. 3-4. It needs to be noted that only limited data is presented here, based on which the sizes of the SAP-originated voids were similar in the two types of cement pastes. A broader study where the total absorption and size distributions of SAP-originated voids are analyzed for larger sections of the pastes is currently in progress.

The grey matter forming in the SAP-originated voids is portlandite, as revealed by EDS. No considerable difference could be observed at a glance regarding the shapes of the pores created by swollen SAP in the two cement pastes, Fig. 8.

![Fig. 3 Polished sections of cement pastes prepared with a) OPC and b) BFSC observed with BSE. Magnification 200 ×. Irregular black shapes are the remnants of the swollen SAP filled partially by calcium hydroxide.](image-url)
4 CONCLUSIONS

Internal curing with SAP has been studied for cement pastes with two different types of cement: OPC and BFSC. The reference cement pastes without SAP had w/c of 0.30, while in internally cured pasted additional water was entrained within SAP to obtain w/c 0.35. In particular, the limiting effect of SAP on self-desiccation and self-desiccation shrinkage was investigated. In the OPC paste, autogenous shrinkage was practically eliminated. Very high levels of autogenous shrinkage observed in BFSC paste (over 1300 $\mu$m/m) could only be partially reduced due to addition of SAP (to approximately 700 $\mu$m/m). Even though autogenous shrinkage could not be eliminated, such reduction would decrease the risk of early-age cracking. A further important difference in the action of SAP regarded its influence on hydration evolution. Whereas the well-known increase of the degree of hydration could be observed in the OPC paste, hardly any influence of SAP on the hydration of BFSC was measured. A microscopy study performed with BSE showed no clear differences between the pores created by the SAP in the two types of cement pastes analyzed. However, a broader study focused on size distribution of the macropores and amounts of hydration products (calcium hydroxide) built into the SAP voids is currently in progress.

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REFERENCES


QUALITY CONTROL OF REINFORCED CONCRETE STRUCTURES FOR DURABILITY

Gladwell W. Nganga (1), Mark G. Alexander (1) and Hans D. Beushausen (1)

(1) Concrete Materials and Structures Integrity Research Unit (CoMSIRU), Department of Civil Engineering, University of Cape Town, South Africa - ngngla001@myuct.ac.za

Abstract

The extensive deterioration of reinforced concrete (RC) structures in the recent past has raised worldwide concerns. To ensure efficient utilization of resources (sustainable construction) there is a need to construct durable RC structures. The current quality control approach of measuring strength of standard specimens such as cylinders or cubes is inadequate as it is not directly related to durability. Concrete cover quality can be reliably assessed through the use of a performance-based approach which also allows for flexibility in selection of materials by the concrete producer on the condition that the required performance is complied with. The performance-based approach developed and used in South Africa is based on the durability index (DI) tests. A review of implementation of the specifications in the production of precast median barriers which were used in a large scale infrastructure project in Gauteng Province is provided. The material provisions, strength and durability indicators are considered and discussed. A second study based on determining the effects of reducing the cement content on the properties of concrete (strength and durability) is also provided. From the two studies, it is shown that high binder content may result in high strength which does not necessarily ensure compliance with durability requirements. It is possible to reduce the cement content, ensuring its efficient use, while still attaining the required durability and strength properties.

Keywords: Durability; quality control; performance-based approach; sustainable construction

1 INTRODUCTION

Concrete is a widely used construction material, its consumption has been described to be second to that of water [1]. Its popularity can be attributed to various reasons such as: its versatility, readily available materials, fair cost in comparison to other construction materials, and durability under most conditions, to name but a few. However, in the recent past, there have been increasing concerns with the lack of durability of concrete structures, more so the pervasive corrosion of reinforced concrete (RC) structures resulting in high costs of repair and rehabilitation [2,3].
Sustainable development using concrete requires the efficient use of resources with consideration of environmental, economic and social aspects. The production of cement, one of the main constituents in concrete, has been identified to emit approximately 5% of the world’s anthropogenic CO$_2$ emissions which raises environmental concerns [4].

There is a projected increase in population in developing countries e.g. in Asia, South America and Africa. With this growth, the demand for infrastructure will increase. To ensure sustainable construction to meet these rising infrastructure requirements there is a need to: a) ensure the design and construction of durable structures that will adequately perform over their service life with acceptable levels of repair and rehabilitation [5] (b) reduce the CO$_2$ emissions that arise from cement production through use of alternative fuels, alternative raw materials and use of by-products from other industries to produce blended cements [6].

2 SUSTAINABLE CONSTRUCTION AND DURABILITY

Sustainable construction for concrete structures requires that they be durable, and that cement usage is ‘efficient’. Consequently, this section will consider durability of RC structures, contrasting the current ‘prescriptive’ approach with the use of a performance-based approach in the design of durable RC structures, and application of this approach to ensure efficient use of cement in concrete.

2.1 Durability of concrete structures based on the ‘prescriptive’ approach of design

The durability of RC structures is largely dependent on the quality of the concrete cover, its depth and resistance to penetration of aggressive agents that cause deterioration in a particular environment. This quality is influenced by the material properties and construction practices which should ensure proper placing, compaction and curing of concrete [7]. Provisions in design standards e.g. BS EN 206-1 [8] and SANS 10100-2 [9] outline material requirements (maximum water/cement ratio and minimum cement content), minimum cover depth and minimum strength for a given environment so as to attain the required durability of a RC structure. The limitations with this current prescriptive approach in the standards are:

a) The limiting values of maximum water/cement ratio and minimum cement content are difficult to verify for compliance on structures on site.

b) The material provisions of maximum water/cement ratio and minimum cement content are based on past empirical studies. These provisions fail to consider changes in recent cements e.g. their physical properties (increase in surface area due to fine grinding) and chemical properties (increase in tricalcium silicate (C$_3$S) and tricalcium aluminate (C$_3$A) content) which results in rapid hydration reactions and an increase in the rate of strength development. With these new cements the specified 28 day strength can be attained with a lower cement content resulting in high water/cement ratios which leads to an increase in penetrability [10].

c) Strength is an inadequate quality control measure as it does not verify the penetrability of the concrete cover. Strength measurements are done using cube/cylindrical specimens which are prepared in ideal laboratory conditions with proper compaction and are mostly water-cured. This measure does not provide verification on the quality of the cover concrete which is dependent on proper placing, compaction and curing of the concrete mix on site to ensure the required resistance to penetration is attained [11,12].
These limitations in the current prescriptive standards have been identified with several proposals by researchers to shift to a performance-based approach that provides verification of the concrete cover properties of depth and resistance to penetration.

2.2 Performance-based approach

The performance-based approach is a quantitative design methodology that involves the use of mathematical models that describe the rate of transport of aggressive agents which initiate deterioration in a particular environment [13]. The models have been developed for exposure conditions of, inter alia, carbonation-induced and chloride-induced corrosion. From these models, measurable output parameters, geometric properties (cover depth) and material properties (diffusion coefficient) are determined for a specified service life. Performance specifications are based on these output parameters and provide a set of clear, achievable, measurable and enforceable instructions of the functional requirements of hardened concrete [14]. The required performance is then verified for compliance with the use of performance test methods which should be reliable, repeatable and accurate [15].

The performance-based approach allows for flexibility in the choice of materials used and facilitates innovation by the contractor and concrete producer provided that the required performance is met. This should enable a savings in costs as a concrete producer can use readily available materials that are inexpensive. It also enables the concrete producer and contractor to work together in developing a concrete mix that meets the required performance [14]. The performance-based approach considered in this paper is the South Africa’s durability index performance approach.

2.3 Efficient use of cement in concrete

The production of cement is projected to increase so as to meet the increasing infrastructure needs in developing countries [5]. The CO₂ emissions from cement production would also be expected to increase with such a rise in production. These emissions are derived from two main sources in the production process: calcination of limestone to produce calcium oxide, and from fuels which are burnt to provide energy that propels the production process [16]. To reduce CO₂ emissions, cement producers have undertaken several measures such as: a) modification of the production process making it less energy intensive, b) reduction in the clinker/cement ratio through the use of supplementary cementitious materials such as slag and fly ash which are by-products from other industries to produce blended cements, c) use of alternative fuels such as sewage or industrial sludge, biogas etc. [4, 6, 16].

A further approach that has been used to reduce the CO₂ emissions from cement-related activities is a reduction in the cement content in concrete. Provisions in the design standards outline minimum cement contents required to attain durability of a RC structure in a given environment. From studies reported in Wassermann et al. [17] and Dhir et al. [18], it was observed that the cement content can be reduced from the minimum provided in the standards without having a negative influence on durability parameters. In some cases the durability parameters were observed to improve with a lower cement content.

The replacement of cement in concrete with supplementary cementitious materials has also been undertaken in several studies in binary, ternary or quaternary mixes. For example Bilodeau and Malhotra [19] explored the use of high volume fly ash concrete where the cement content was reduced by as much as 50%. The main limitation identified with such
mixes is the slow development of strength which has been addressed through the use of fine materials that react rapidly such as limestone powders or silica fume [20, 21].

From a recent study reported in Fennis et al. [22, 23], the compaction interaction packing model (CIPM) was used in the design of a defined-performance ecological concrete. The particle packing density of a mixture was determined and applied in a cyclic design process where a cement spacing factor (CSF) was determined which is related to strength. If the strength obtained was higher than that required, the cement content was reduced and the cyclic design process repeated until the required strength value was obtained. From experimental studies undertaken, the cement content was reduced from the minimum specified in the standards of 260 kg/m$^3$ to 110 kg/m$^3$ with addition of fly ash and quartz powders in the low cement concrete mixes. This concrete complied with the required strength specified and was found to have higher resistivity in comparison to the reference concrete with a cement content of 260 kg/m$^3$.

With the use of the performance-based approach, the cement content can be reduced through addition of supplementary cementitious materials, inert fillers, chemical admixtures and design approaches such as developed by Fennis et al. [23] to optimize material properties and obtain the required concrete properties. This ensures the efficient use of cement in concrete with a corresponding reduction in CO$_2$ emissions.

3 THE DURABILITY INDEX PERFORMANCE-BASED APPROACH: PRACTICAL IMPLEMENTATION

The durability index tests developed and used in South Africa provide measures of permeability (oxygen permeability index, OPI), sorptivity (water sorptivity index, WSI) and porosity, and chloride conductivity (chloride conductivity index, CCI) of a sample of concrete. The tests are carried out using cylindrical concrete specimens with a diameter of 70 mm and thickness of 30 mm. The concrete specimens are cored from test panels or from the actual structure typically 28 days after casting, then oven dried at 50°C prior to testing. The OPI and CCI tests are used in service life design as good correlations have been established between OPI values and carbonation depth (carbonation-induced corrosion model) and CCI and diffusion coefficient in Fick’s second law of diffusion (chloride-induced corrosion model). Limiting values of the durability index tests (OPI and CCI) that are used in performance-based specifications have been established using these models with consideration of environmental conditions, cover depth, material properties and the required service life [24].

The durability index tests have been implemented in a large scale infrastructure project, the Gauteng Freeway Improvement Project (GFIP) in Gauteng Province, South Africa. The total expenditure on the project was approximately US$ 2 billion. The entire project was undertaken over 185 km of the road network which was divided into sub-projects. One of the sub-projects considered in this paper involved the construction of precast elements that were used as median barriers on the highways. The material provisions for the median barriers are summarized in Table 1.
The limiting values for the project which is located in a carbonation exposure environment were a mean cover depth value of 40 mm, design strength of 30 MPa and minimum OPI value of 9.70 (log scale). From Table 1, it is observed that the total binder content is high at 586 kg/m$^3$ with a high cement content of 410 kg/m$^3$ [25]. This cement content is considerably higher than minimum cement content in the design standards which is 300 kg/m$^3$ [8].

Additional data was obtained from the project, i.e. the strength, OPI and cover depth values. The average values complied with limit values provided in the specifications with an average strength of 49.4 MPa and an average OPI value of 10.25 (log scale). A comparison of the strength and OPI values from the project is provided in Figure 1.

It is observed that no relation exists between OPI and compressive strength. A high strength value does not always result in a corresponding high OPI value (indicating a higher resistance...
to penetrability). The strength values measured were considerably high, maximum obtained of 75 MPa which far exceeded the limit value in the project specifications. The high binder content resulted in low water/binder ratios that led to high strength values. However, the specified OPI values could be obtained at lower strength values indicating that the cement content can be reduced while meeting the required limit values of strength and OPI provided in the specifications [25].

4 INFLUENCE OF CEMENT CONTENT ON DURABILITY PARAMETERS

The provisions for material properties in the standards specify a minimum cement content and maximum water/cement ratio to ensure durability of a RC structure in a particular environment [8, 9]. A study that sought to determine how these provisions in the standards are interpreted in the South African construction industry is reported in Angelucci [26]. From the study, it was observed that the provisions for cement content were usually higher than those in the standards as it was perceived that a high cement content resulted in high strength and a corresponding increase in resistance to penetrability, which would increase the durability of a RC structure.

To determine whether this practice of cement provisions as used by the construction industry is valid, experimental work was undertaken which aimed at determining the influence of mix design parameters such as water/cement ratio and cement content on strength and durability parameters [26]. The variables in the experimental work were cement type (CEM I 52.5 N, 30% fly ash and 50% slag), the water/cement ratio (0.40, 0.50 and 0.60), four different cement contents and two curing regimes (water-cured and laboratory-cured (temperature of 20°C and 50% relative humidity) concrete mixes). The concrete mix properties for a water/cement ratio of 0.40 are considered and are provided in Table 2; the coarse aggregate content was constant at 1100 kg/m³ with addition of Sika superplasticizer in some cases to maintain workability.

Table 2: Concrete mix properties for a water/binder ratio of 0.40

<table>
<thead>
<tr>
<th>Mix design parameters</th>
<th>Mixes</th>
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<td>Binder content</td>
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<td>C</td>
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<td>319</td>
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<td>D</td>
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<td>341</td>
<td>146</td>
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The tests carried out involved the 7, 28 and 90 day compressive strength, and durability index tests - oxygen permeability index, water sorptivity index and chloride conductivity index. From the concrete mix properties summarized in Table 2, the highest cement content was 488 kg/m$^3$ for Mix I-D while the lowest was 194 kg/m$^3$ for Mix III-A. A comparison of the effect of cement content on the compressive strength and OPI at 28 days is provided in Figure 2 for the water/cement ratio of 0.40. The compressive strength obtained was high with values ranging from 57 to 63 MPa. The OPI values were also considerably high ranging from 9.98 to 10.84, on a log scale. This indicates concrete with a low permeability.

![Figure 2: Comparison of the effect of binder content on compressive strength and OPI for a water/cement ratio of 0.40](image)

From Figure 2, the lowest compressive strength was obtained at the highest binder content of 488 kg/m$^3$. This indicates that a high cement content does not always result in high strength. There is no linear relationship between the OPI value and strength, that is, a high strength value does not always lead to a high OPI value. The highest OPI value was obtained at a binder content of 341 kg/m$^3$; an increase of cement content beyond this content led to a reduction in OPI values. Similar observations on strength and durability parameters were made in Wassermann et al. [17] and Dhir et al. [18] indicating that the required concrete properties can be obtained with a lower cement content. Thus, there is justification for application of the performance-based specifications in construction industry practice since the provision of high cement content does not always result in the required concrete properties of strength and durability. This would ensure efficient usage of cement in addition to a reduction in costs of construction.
5 Conclusion

Concrete is without doubt the construction material of choice that will enable the much needed infrastructure development in developing countries. However, there is a need to ensure sustainable construction with concrete which will be achieved by design and construction of durable structures and the efficient use of cement. The current quality control provision of strength measurement is inadequate and a shift to a performance-based approach is key to construction of durable structures. The application of this approach in South Africa has been presented in the two case studies considered. It was observed that strength is not directly related to durability. There is need for a shift in provisions for cement in the current South Africa’s construction industry to lower the cement content. The application of the durability index performance-based approach will therefore facilitate the construction of durable RC structures and the efficient use of cement.

REFERENCES


A NUMERICAL INVESTIGATION OF AGGREGATE SHAPE EFFECT ON THE DIFFUSIVITY OF MORTAR BASED ON ELLIPSOIDAL PARTICLE PACKING

Lin Liu (1), Huisu Chen (2), Wenxiang Xu (3)

(1) College of Civil and Transportation Engineering, Hohai University, Nanjing 210098, China -linliulinliu@gmail.com

(2) Jiangsu Key Laboratory of Construction Materials, School of Materials Science and Engineering, Southeast University, Nanjing 211189, China - chenhs@seu.edu.cn

(3) Institute of Soft Matter Mechanics, College of Mechanics and Materials, Hohai University, Nanjing 210098, China - xwxfat@yahoo.com.cn

Abstract

The diffusivity of cement-based materials is a key factor related to its service life and durability issue for concrete structures. In recent years, a number of numerical models have been developed to predict the diffusivity of cement-based materials from its corresponding meso-/micro-structures since the great reduction in cost and experimental work. Nevertheless, most modeling works have focused on the packing systems of spherical particles. Investigations about the particle shape effects on the diffusivities of cement-based materials are rarely reported. Based on ellipsoidal particles packing, by coupling with a 3D lattice approach, the aggregate shape effects on the diffusivities of mortar are studied in this paper. By the random packing model of ellipsoidal particles, mortars with aggregates of different aspect ratios (e.g. \( \kappa = 0.4, 0.6, 0.8, 1, 2, 3 \)) are obtained first. Then, the mesostructures of mortars are digitalized by a specific algorithm. Consequently, 3D lattice networks composed of diffusive lattice elements are constructed from the digital mesostructures of mortars. From the 3D lattice networks, the diffusivities of mortars can be predicted. The simulations are compared with experimental results. Finally, the dependences of mortar diffusivities on the aggregate shapes at various aggregate volume fractions are discussed. The influences of ITZ zone on the diffusivities of mortars of aggregates of different aspect ratios are also discussed.

Keywords: shape effect, mortar diffusivity, ellipsoidal particle packing, numerical simulation

1. INTRODUCTION

Diffusivities of cement-based materials are closely related to the service life and durability of concrete structures. In recent years, a number of numerical models [1-6] have been developed to predict the diffusivity of cement-based materials from its corresponding meso-
micro-structures, since the great reduction in cost and testing work compared to experiments. By using random walk algorithm, the diffusivities of concrete and cement paste have been investigated by Bentz et al. [1] and Liu et al. [2] respectively. By converting the micro-/meso-structure of cement-based composite material into a transport network and using a lattice approach, Liu et al. [3] have revealed the diffusivity changes of cement paste with microcracks and Zheng et al. [4] have indicated the effect of elliptical aggregate shapes on the diffusivity of concrete. By finite element method, Kamali-Bernard et al. [5] have computed the diffusivity of mortars from a digitized 3D mesostructure, and Li et al. [6] have obtained the diffusivities of concrete using 2D mesostructures with several aggregate shapes. Nevertheless, most modeling works have focused on the packing systems of spherical particles in three dimensions [1-3,5] or limited to simple various shaped particles (ellipses or convex polygons) in two dimensions [4,6]. Investigations about the particle shape effects on the diffusivities of cement-based materials in three dimensions are rarely reported. Following the present studies, this paper is going to investigate the influences of aggregate shape on the diffusivity of mortar by random packing models of ellipsoidal particles.

In order to investigate the effect of aggregate shape, aggregate particles are modeled as ellipsoids with varying aspect ratios (e.g. $\kappa = 0.4, 0.6, 0.8, 1, 2, 3$). A lattice approach is utilized to predict the diffusivities of mortars. The mesostructures of mortars of ellipsoidal particles packing are first digitalized. Then, lattice networks composed of diffusive lattice elements are constructed from the digital mesostructures of mortars. From the 3D lattice networks, the diffusivities of mortars can be obtained. The simulations are validated with experimental results from literature. Finally, the dependences of mortar diffusivities on the aggregate shapes for mono-sized particle packing systems, multi-sized particle packing systems at various ITZ thicknesses are discussed.

2. RANDOM PACKING OF ELLIPSOIDAL PARTICLES

In this work, aggregate particles are modeled as a number of ellipsoids with a given particle size distribution (PSD). An equivalent diameter $D_{eq}$, defined as the diameter of a sphere having the same volume as that of a non-spherical particle [7-9], is introduced to connect the ellipsoidal PSD with the spherical PSD in cement-based composite materials [9-10]. For ellipsoids, $D_{eq}$ can be expressed as [10],

$$D_{eq} = \begin{cases} 2\kappa \kappa^{2/3} & \kappa < 1 \\ 2\kappa^{1/3} & \kappa \geq 1 \end{cases} \quad (1)$$

where $D_{eq}$ is the equivalent diameter of the ellipsoid or the polyhedron. $c$ and $\kappa$ (i.e., if the ellipsoid shape is prolate, $\kappa = a/c$; if the ellipsoid shape is oblate, $\kappa = c/a$) are the semi-minor axis and aspect ratio of the ellipsoid, respectively.

The particle size distribution of aggregates can be determined either experimentally in a conventional sieve analysis or generated from a theoretical gradation. In concrete, two particular distribution functions, the Fuller distribution and the equal volume (EVF) distribution, are commonly used to represent the PSD of aggregates. The Fuller and the EVF distribution functions, which stand for the lower and upper bound of aggregates respectively, can be given as [10],

$$\text{Fuller distribution: } \frac{1}{2\kappa^{1/3}} \left( \frac{x}{c} \right)^{(1/3)(\kappa + 1)}$$

$$\text{EVF distribution: } \frac{1}{2\kappa^{1/3}} \left( \frac{x}{c} \right)^{(1/3)(\kappa - 1)}$$

where $x$ is the particle size, and $c$ and $\kappa$ are the semi-minor axis and aspect ratio of the ellipsoid, respectively.
where $F_N(D_{eq})$ is the cumulative number-based probability function of aggregates, $D_{eq}$ is the equivalent diameter of a non-spherical aggregate (mm), and $D_{max_{eq}}$ and $D_{min_{eq}}$ are the maximum and minimum equivalent diameters (mm), respectively. Thus, according to equation (2), the particle number of ellipsoidal aggregates with various sizes can be obtained with a given volume fraction of aggregates $V_f$ in concrete. Details of the determination of the number of ellipsoidal particles are described in literature [9].

After the total number of non-spherical particles with various sizes is known, the mesostructure model of mortar or concrete can be constructed by random packing of such ellipsoidal or polyhedral particles in the bulk cement paste. The key issue of the random packing of aggregate particles is to ensure that each particle does not overlap with the others, while the neighboring interfacial layers around the aggregate particles may overlap. A contact detection algorithm of ellipsoids is presented and the accuracy and efficiency of the algorithm are tested by Xu et al. [7-10]. To eliminate wall effects, periodic boundary conditions are applied.

By the random packing model of ellipsoidal particles, the mesostructures of mortar with different aspect ratios (e.g. $\kappa = 0.4, 0.6, 0.8, 1, 2, 3$) can be obtained, see figure 1. In the random packing models of ellipsoidal particles, the mesostructure is represented by vectors. The geometric information of an ellipsoid $E$ in the random packing model is described by the nine degrees of freedom, i.e., semi-major axis ($a$), semi-intermediate axis ($b$), semi-minor axis ($c$), center ($x_0, y_0, z_0$) and three Euler angles ($\alpha, \beta, \gamma$). It is a so-called nine parameters method.

3. DIGITALIZATION OF MESOSTRUCTURES

In this section, the generated mesostructures of mortars with non-spherical aggregates are digitized into voxel-presented mesostructures, represented by bulk cement paste, ITZ, and aggregate. The digitalization of mesostructures is implemented based on a nine points controlling algorithm, which is utilized to digitize the microstructure of cement paste with spherical particles [3]. For each voxel, its phase is determined as the major phase among the phases of nine points. The nine points are the vertices of the cubic voxel and its center. Figure 2 shows the digitized mesostructures of mortar with ellipsoidal aggregates of $\kappa=0.6$. The thickness of ITZ is 50 $\mu$m. In order to capture the ITZ zones, a resolution of 10 $\mu$m/voxel is utilized.
4. 3D LATTICE APPROACH FOR DIFFUSIVITY

In this section, a lattice approach is employed to evaluate the diffusivity of mortar in question, which has been utilized for modeling the diffusivity of cement-based composite materials in previous studies [3-4].

In the 3D lattice modeling of the diffusivity of mortar, the first step is to convert the voxel-presented mesostructure of mortar into a lattice network consisting of diffusive lattice elements. In the voxel-presented mesostructure, the diffusivity of each voxel is equal to the diffusion coefficient of its composing phase. Two diffusive phases (i.e., bulk cement paste and ITZ) exists in mortar, while aggregate phase is assumed to be non-diffusive. A diffusive lattice element can be formed by connecting any two adjacent diffusive voxels. Figure 3 illustrates how to construct a lattice network which consists of diffusive lattice elements.

![Figure 2: Digitized mesostructure of mortar with ellipsoidal aggregates.](image)

![Figure 3: Determination of the diffusive lattice elements.](image)

For the diffusive lattice element \( i-j \) relating voxel \( i \) and voxel \( j \), its diffusion coefficient \( D_{i-j} \) is [3],

\[
D_{i-j} = \frac{2}{D_i + D_j}
\]  

(3)

where \( D_i \) and \( D_j \) is the diffusion coefficient of voxel \( i \) and voxel \( j \), respectively. The flow through the element \( i-j \) is satisfied with Fick’s first law,

\[
q_{i-j} = -D_{i-j} \frac{dc_{i-j}}{dx}
\]  

(4)

where \( q_{i-j} \) is the flow density through element \( i-j \). \( c_{i-j} \) is the concentration gradient from voxel \( i \) to voxel \( j \). \( x \) is the center distance from voxel \( i \) to voxel \( j \).

According to equation (4), the matrix equation for a diffusive lattice element can be written by [3],

\[
-\frac{D_{i-j}A_{i-j}}{l} \begin{bmatrix} 1 & -1 \\ -1 & 1 \end{bmatrix} \begin{bmatrix} c_i \\ c_j \end{bmatrix} = \begin{bmatrix} q_{i-j} \\ q_{j-i} \end{bmatrix}
\]  

(5)

where \( A_{i-j} \) and \( l \) represents the area and length of the element \( i-j \), respectively. The whole matrix equation for the lattice network is assembled one element by one element.

After the lattice network is constructed and whole matrix equation for the lattice network is assembled, the next work is to impose concentration boundary conditions. Concentrations \( c_i \)
and \( c_0 \) are assigned to the inlet surface and the outlet surface, respectively. The inlet and outlet surfaces are assumed as two parallel surfaces of the cubic mesostructure.

The concentration distribution in the lattice network can be obtained by solving the whole matrix equation. There are several methods to solve the whole matrix equation, for instance, finite difference method (FDM) and conjugate gradient method (CGM). The FDM can give the concentration changing with time but time consuming. The CGM can not give concentration changing with time, but can calculate the concentration distribution at steady-state with a high efficiency \(^5\). CGM is utilized in this study. After the steady-state diffusion is achieved, the diffusion coefficient of mortar, \( D_m \), can be obtained by,

\[
D_m = \frac{Q}{A} \frac{L}{c_t - c_0}
\]

where \( Q \) is flux through the outlet surface, mol·mm\(^{-2}\)·s\(^{-1}\); \( L \) is the length of the mesostructure; \( A \) is the cross-sectional area of the mesostructure, mm\(^2\); \( c_t \) and \( c_0 \) is the concentration of the diffusive species on the inlet and outlet surface, mol·mm\(^{-3}\).

According to experimental results measured by Delagrave et al. \(^{11}\), the tritiated water diffusion coefficient of the water/cement paste is \( 9.8 \times 10^{-12} \) m\(^2\)/s. In this paper, this value is assigned to paste voxels, and a mean value of \( D_{ITZ}/D_{paste} = 6.4 \) is used for ITZ voxels. The value of \( D_{ITZ}/D_{paste} = 6.4 \) corresponds to a homogeneous value associated to a given ITZ thickness and has been previously identified by the authors \(^5\). As to boundary conditions, 1.0 mol/mm\(^3\) for \( c_t \) and 0 for \( c_0 \) are assumed.

5. COMPARISON WITH EXPERIMENTAL RESULTS

A number of experiments have investigated the influence of aggregate on the diffusivity of mortar, which could be utilized to validate the simulation results at different aggregate volume fractions. Five sets of experimental results \(^{11-15}\), given in figure 4, generally exhibit the same trend that the relative diffusivities of mortar decrease with the increasing aggregate volume fraction. While the data from literature \(^{16}\) indicate that the relative diffusivities of mortar increase with the increasing aggregate volume fraction when a fine sand grading was utilized. The experimental results also show a certain variability attributed to different test methods (under steady-state regime \(^{11-14}\) or non-steady-state regime \(^{15-16}\)), different diffusion species (chlorides \(^{12, 14-16}\), oxygen \(^{13}\), tritiated water \(^{11}\)), different sand grading \(^{53, 57}\), different w/c, different hydration ages and so on.

Simulations from ellipsoidal particles packing system with different aspect ratios are compared with these experimental results. With a Fuller distribution and \( D_{eq} \) in the range of 0.2−2 mm, the simulated results may show different trends with different aspect ratio and ITZ thickness. The upper curve by simulation is obtained from spherical particle packing systems with a 50 µm ITZ thickness, while the lower curve by simulation is obtained from oblate spheroidal particle packing systems with \( \kappa = 0.4 \) and no ITZ zone. The upper curve by simulation show an increase in the relative diffusivities of mortar with the increasing aggregate volume fraction, exhibiting the same trend as the experimental results from Caré \(^{15}\) where a fine sand grading was utilized. The lower and middle curves show the same trend as other experimental results \(^{11-14}\), that is, the diffusivities of mortar decrease with the increasing aggregate volume fraction. Except for the aggregate shape effects, this may be
attributed to the change in ITZ content and connectivity in mortars no matter by simulation and by experiment.

6. RESULTS AND DISCUSSIONS

By applying the computational methods presented above, the dependences of the relative diffusivities of mortar on oblate ellipsoidal (aspect ratio of $\kappa = 0.4, 0.6$ and 0.8), spherical and prolate ellipsoidal ($\kappa = 2$ and 3) particles are investigated. To characterize the particle shape, sphericity is used to unify the shape. Sphericity $s$ is defined as the ratio of surface area between spherical and non-spherical particles with the same volume [17].

The shape effects for mono-sized particle packing systems, multi-sized particle packing systems at various ITZ thicknesses are illustrated in figure 5 to figure 7. It could be found that at $s=1$, the maximum relative diffusivity of mortar is achieved by simulation. The predicted relative diffusivity of mortar decreases with the decreasing sphericity. By comparing the oblate to the prolate ellipsoidal particle packing systems, it is found that the influence of sphericity on prolate ellipsoidal particle packing systems is weaker than on the oblate one. At the same $s$, the predicted relative diffusivity by prolate ellipsoidal particle packing system is higher than by the oblate one.

Figure 5 can also indicate that at the same aggregate volume fraction, a lower diffusivity of mortar can be obtained by using coarse aggregate. For multi-sized particle packing systems, the shape effect under high aggregate volume fraction is significant than that under a relative lower aggregate volume fraction, see figure 6 and 7. The shape effects at different ITZ thicknesses (t=25µm, 50µm) illustrate the same trend, that is the predicted relative diffusivity of mortar decreases with the decreasing sphericity. The predicted value under a thick ITZ thickness is higher than that with a relative thinner ITZ thickness, see figure 7.

Figure 4: Comparison of relative diffusivities of mortar by simulations and by experiments [11-16].

Figure 5: Dependences of relative diffusivities on $s$ for mono-sized particle packing systems.
Figure 6: Dependences of relative diffusivities on $s$ for multi-sized particle packing systems at different aggregate volume fractions.

Figure 7: Dependences of relative diffusivities on $s$ for multi-sized particle packing systems at different ITZ thicknesses.

7. CONCLUSION

To quantitatively evaluate the influences of particle shapes on the diffusivity of mortar, aggregate particles are modeled as ellipsoids with varying aspect ratios (e.g. $\kappa = 0.4, 0.6, 0.8, 1, 2, 3$). These particles’ shapes are unified by a shape parameter sphericity, which could be further utilized to compare with polyhedral particle packing systems in the future. By coupling the 3D lattice approach with the random packing of ellipsoidal particles, the diffusivity of mortar can be predicted. Simulations indicate that at $s=1$, the maximum relative diffusivity of mortar is achieved, and the relative diffusivity of mortar decreases with the decreasing sphericity. By comparing the oblate ellipsoidal particle packing systems to the prolate ones, it is found that the influence of sphericity on the prolate ellipsoidal particle packing systems is weaker than on the oblate ones. At the same $s$, the predicted relative diffusivity by prolate ellipsoidal particle packing system is higher than by the oblate one.

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BIOGENIC CORROSION OF CONCRETE SEWER PIPES: A REVIEW OF THE PERFORMANCE OF CEMENTITIOUS MATERIALS

Moses W. Kiliswa (1) and Mark G. Alexander (1)

(1) Concrete Materials and Structural Integrity Research Unit (CoMSIRU), Department of Civil Engineering, University of Cape Town, Cape Town – klswop001@myuct.ac.za; mark.alexander@uct.ac.za

Abstract

The paper reviews the performance of cementitious materials under conditions of biogenic corrosion of concrete sewer pipes. It draws on data from samples analysed in the laboratory and also installed in an experimental sewer in South Africa, as well as data from sewers in France and work from simulation chambers in Germany. Cementitious materials, including supplementary cementitious materials (SCMs), have been used widely in sewer networks to counter different mechanisms of deterioration, with varying success. However, under certain conditions, sewage that is conveyed through outfall sewers becomes septic and hydrogen sulphide gas is generated and biogenically converted to sulphuric acid, which reacts with the acid-soluble components of concrete causing it to corrode. The concrete sewer pipe materials under review comprise those made with Portland Cement and Calcium Aluminate Cement. Results indicate that the formation of alumina gel, as a product of a chemical reaction between the acid and certain cementitious materials, and densification of the concrete matrix, have a significantly positive impact on the rate of deterioration of concrete sewer pipes. The experimental scope of a related current research project is previewed, focusing on the expected outcomes.

Keywords: concrete sewers, biogenic acid generation, biogenic corrosion, cementitious materials.

1 INTRODUCTION

Concrete sewer pipe deterioration due to biogenic sulphuric acid (H₂SO₄) attack is not a new problem. As far back as 1918, attempts were made to combat corrosion in a range of sewers in Cairo [1] and since then, researchers have studied the problem of sewer corrosion to establish the mechanisms involved and factors having an effect on the rate of corrosion [2, 3].
Initially, in order to combat microbially induced concrete corrosion (MICC) of sewer pipes, authorities \[4\] imposed tighter regulations on the nature and toxicity of industrial wastewater that was discharged to sewers and this led to significantly lower levels of biologically toxic metals such as lead, chromium and mercury, and as a result, bacterial levels and MICC increased. This trend prompted further research which subsequently revealed the complexity and the environment of many species of bacteria as well as fungal species involved in MICC \[5, 6\]. Increase in MICC was also linked to increased sewage temperature, sulphate-containing detergents, and long sewer line lengths to service growing urban populations \[7\].

Fresh sewage entering a wastewater collection system contains organic sulphur compounds that are capable of being oxidized to sulphates (SO\(_4^{2-}\)). Initially, aerobic species flourish in a sewer but as oxygen used for catabolizing organic waste in the sewage becomes depleted, anaerobic species occur, with two types of bacteria being involved in MICC. Firstly, there are sulphate reducing bacteria (SRB) in the liquid sewage which reduce SO\(_4^{2-}\) to sulphide as an alternative source of oxygen; sulphide exists in the stale sewage as sulphide ions (S\(^{-2}\)), hydrogen sulphide ions (HS\(^{-}\)), and hydrogen sulphide molecules (H\(_2\)S) in both the aqueous and gaseous states, depending primarily on pH. At pH less than 7, H\(_2\)S predominates in aqueous solutions, and is readily released into the sewer headspace especially under turbulent flow conditions \[8\]. Secondly, there are sulphide oxidising bacteria (SOB), typically on the moist walls of the sewer pipe above the flowing sewage. It therefore follows that oxidation of H\(_2\)S to H\(_2\)SO\(_4\) occurs in two ways, through auto-oxidation by a spontaneous reaction of H\(_2\)S with O\(_2\) in an aqueous solution, or biologically due to SOB, namely acidithiobacilli bacteria, oxidising H\(_2\)S to generate energy for assimilation of carbon for synthesis of cell materials. The latter process is generally referred to as biogenic acid formation. Figure 1 shows these significant sewer characteristics.

\[
\text{H}_2\text{S} + 2\text{O}_2 \rightarrow \text{H}_2\text{SO}_4
\]

This paper reviews the performance of cementitious materials under conditions of biogenic corrosion of concrete sewer pipes. It draws on data from samples analysed in the laboratory and also installed in an experimental sewer in South Africa, as well as data from sewers in France, and work from simulation chambers in Germany. The data are placed in a design context by relating them to expected deterioration rates, and subsequently the implications for use of these materials in such aggressive environments are drawn. Further, the experimental...
scope of a related current research project is previewed, focusing on the expected outcomes and benefits.

2 FUNDAMENTALS OF ACID ATTACK ON CONCRETE SEWERS

2.1 Colonization of concrete sewer pipe surfaces by bacteria

Newly cast concrete pipes have a pH of about 12 to 13, which is too high for microbial activity to take place. Nonetheless, through the auto-oxidation of $\text{H}_2\text{S}$ to $\text{H}_2\text{SO}_4$, and also as CO$_2$ dissolves into the condensate film that forms on the walls of the sewer to form various forms of carbonic acid (H$_2$CO$_3$) [9], these acids react with the alkali species in the concrete, and over time lower the pH of the concrete surface. In the presence of sufficient oxygen, nutrients and moisture, neutrophilic sulphur oxidising microorganisms (NSOM) such as *acidithiobacillus thioparus* [10] can colonize the concrete surface once the pH falls to ~9. These organisms produce H$_2$SO$_4$ through oxidation of the H$_2$S diffusing into the condensate film, which reacts with the concrete lowering the surface pH further, thus facilitating colonization by new bacterial strains adapted for lower pH conditions. When the pH of the concrete falls to ~4, acidophilic sulphur oxidising microorganisms (ASOM) such as *acidithiobacilli thiiooxidans* can begin colonising the concrete surface and continue to oxidise H$_2$S to H$_2$SO$_4$, but can also oxidise thiosulphate and elemental sulphur that is deposited on the sewer walls after H$_2$S is directly oxidised by O$_2$ that is present in the headspace. These species lower the pH of the concrete surface to values of ~1 [11].

2.2 Characteristics of concrete subjected to H$_2$SO$_4$ attack

In Portland Cement (PC) concretes, H$_2$SO$_4$ initially converts the calcium hydroxide (Ca(OH)$_2$) to calcium sulphate (CaSO$_4$) (Equation 1).

$$\text{Ca(OH)}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{CaSO}_4 + 2\text{H}_2\text{O} \quad (1)$$

This attack presents two somewhat opposing scenarios. Initially, H$_2$SO$_4$ destroys the polymeric nature of Ca(OH)$_2$ and substitutes a larger molecule into the matrix causing pressure and spalling of the adjacent concrete and aggregate particles, and in another instance, CaSO$_4$ precipitates as gypsum (CaSO$_4$.2H$_2$O), which may bind together the insoluble residue in the gel layer and create a barrier on the concrete surface thus slowing the rate of attack; this means that ultimately, the rate of attack will depend on the ionic diffusion coefficients and the thickness of the gypsum layer [12].

Most researchers agree that attack in sewers is mainly caused by H$_2$SO$_4$ produced by S-oxidising bacteria, and that large differences are found between tests involving purely chemical (mineral) H$_2$SO$_4$ and those involving microbially produced H$_2$SO$_4$ [13]. Bacteria play a distinctive role, especially their relationship with the concrete substrate which influences their metabolic activity [14]. The main products of corrosion when PC concrete is subjected to either mineral or biogenic H$_2$SO$_4$ attack are gypsum (Equation 1) and ettringite (3CaO.Al$_2$O$_3$.3CaSO$_4$.32H$_2$O - Aft) (Equation 2) which is formed by a secondary sulphate attack of aluminophase by the gypsum.

$$3\text{CaSO}_4.2\text{H}_2\text{O} + 3\text{CaO.Al}_2\text{O}_3 + 26\text{H}_2\text{O} \rightarrow 3\text{CaO.Al}_2\text{O}_3.3\text{CaSO}_4.32\text{H}_2\text{O} \quad (2)$$
Subsequently, a soft and pulpy layer with an increased porosity forms on the corroded concrete surface. In the case involving mineral $\text{H}_2\text{SO}_4$ attack, this layer constitutes an extra barrier, while in the case of biogenic $\text{H}_2\text{SO}_4$ attack this layer creates excellent conditions for the growth of SOB$^{[13]}$.

In Calcium Aluminate Cement (CAC) concretes, the rate of $\text{H}_2\text{SO}_4$ attack is much slower than in PC concretes. The key to CAC’s better performance lies in the nature of the hydrates, particularly alumina hydrates, which are stable down to a pH of $\sim$3. The dissolution of the calcium component of the other hydrates leads to the formation of additional quantities of this phase, which infill pores, thereby protecting the concrete from further attack and generally giving a smoother corroded surface with less aggregate loss (Equation 3).

$$\text{C}_3\text{AH}_6 + 6\text{H}^+ \rightarrow 3\text{Ca}^{2+} + 2\text{Al(OH)}_3\downarrow + 6\text{H}_2\text{O}$$  (3)

Below a pH of $\sim$3.5, the alumina hydrate dissolves and neutralises more acid as shown in Equation 4; the overall neutralization reaction is shown in Equation 5.

$$\text{AH}_3 + 6\text{H}^+ \rightarrow 2\text{Al}^{3+} + 6\text{H}_2\text{O}$$  (4)

$$\text{C}_3\text{AH}_6 + 2\text{AH}_3 + 24\text{H}^+ \rightarrow 3\text{Ca}^{2+} + 6\text{Al}^{3+} + 24\text{H}_2\text{O}$$  (5)

At these lower pH values, there still exists a zone of intermediate pH between that of the surface and of the unaffected core concrete, in which deposition of alumina hydrate provides a barrier to penetration of the acid$^{[15]}$.

3 CEMENTITIOUS MATERIALS STUDIED

3.1 Studies undertaken in South Africa

There was extensive research on sewer corrosion in South Africa by the National Building Research Institute (NBRI) of the Council for Scientific and Industrial Research (CSIR) that culminated in the publication of a book on the subject in 1959$^{[16]}$, in which several recommendations were made. The most significant of these recommendations was to pay attention to sewer hydraulics in order to avoid high velocities and turbulence so as to minimize $\text{H}_2\text{S}$ release, and to use dolomitic (DOL) aggregate which provides more alkalinity to a concrete matrix thereby contributing to the neutralization of any acid attack. It was felt that facilities were required to allow the monitoring of actual performance of various materials that are used in the manufacture of concrete sewer pipes based on the CSIR recommendations.

For this reason, an experimental sewer section was installed alongside a bypass line in Virginia, the so-called Virginia Experimental Sewer (VES), in the Free State Province, South Africa. This location was proposed due to the nature of the effluent (high in sulphates), the flat terrain necessitating pumping, and the generally mild to warm climate (daily temperatures up to 35°C in summer, occasionally up to 27°C in winter) that results in conditions ideal for $\text{S}^{2-}$ generation and acid attack. Under such conditions, an extremely corrosive $\text{H}_2\text{SO}_4$ environment (pH <1.0) develops and $\text{H}_2\text{S}$ levels in excess of 100 ppm are common$^{[17]}$.

Three categories of sewer pipe material samples were installed in 1988 and the sewer was commissioned in March 1989. These samples included unprotected cementitious, protected cementitious and high density polyethylene (HDPE) pipes. The unprotected samples consisted of Portland Cement and siliceous aggregate (PC/SIL) concrete, Portland Cement and
dolomitic aggregate (PC/DOL) concrete, Calcium Aluminate Cement and siliceous aggregate (CAC/SIL) concrete, and asbestos fibre reinforced cement (FC). The protected samples consisted of PC/SIL with a post-installed HDPE lining and FC coated with epoxy tar, polyurethane and epoxy \[^{18}\].

The corrosion rates estimated by taking in-situ measurements on the unprotected pipes after 5 and 12 years, and actual measurements taken on some pipe sections removed from the sewer after 14 years are given in Table 1.

### Table 1: Estimated and measured corrosion rates at Virginia Experimental Sewer \[^{19}\]

<table>
<thead>
<tr>
<th>Material</th>
<th>Measured solubility*</th>
<th>5 year estimate (mm)</th>
<th>12 year estimate (mm)</th>
<th>14 year measured (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Total</td>
<td>Ave./yr.</td>
<td>Total</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.20</td>
<td>&gt; 30.0</td>
<td>&gt; 6.0</td>
</tr>
<tr>
<td>PC/DOL</td>
<td>0.70</td>
<td>10 – 15</td>
<td>2 – 3</td>
<td>20 – 30</td>
</tr>
<tr>
<td>CAC/SIL</td>
<td>0.25</td>
<td>5 – 10</td>
<td>1 – 2</td>
<td>10 – 15</td>
</tr>
<tr>
<td>FC</td>
<td>0.65</td>
<td>10 – 12</td>
<td>2+</td>
<td>20 – 25</td>
</tr>
</tbody>
</table>

*Values based on insolubility tests by NBRI \[^{20}\]

**Measurements not taken on FC pipes as walls were swollen and soft due to products of corrosion

The corrosion rates estimated at 5 years varied slightly from the 12 year estimates and the 14 year measurements. The most significant finding was that the CAC based concrete performed better than other cementitious materials. From experimental work undertaken after 2000, CAC/DOL concrete was found to be about four times as effective in dealing with biogenic corrosion as the PC/DOL concrete and at least ten times as effective as PC/SIL concrete \[^{21}\]. This was attributed to the bacteriostatic effect of CAC binders that stifle the growth of bacteria thus reducing acid generation, and the greater alkalinity provided by DOL to the cement matrix that contributes to the neutralization of any acid formed.

### 3.2 Studies undertaken in France

In situ experiments undertaken within Veolia Water sewers in Arcachon Bay showed better performance of CAC based mortar samples when compared to PC based ones \[^{22}\]. Cylindrical mortar samples were exposed in two sewer networks; Site 1 had H\(_2\)S concentration of between 0 and 20 ppm and a temperature range of between 18 and 25°C, while Site 2 had H\(_2\)S concentration of between 100 and 200 ppm and a temperature range of between 25 and 30°C. Also, in order to measure the pH and detect Adenosine Triphosphate (ATP) as an indicator of microbiological activity in moisture on sample surfaces, a PVC tube was anchored to gather condensate from the samples.

After 4 months of exposure, the surface pH of PC based samples in Site 1 was ~5.5 and no mass loss was recorded, while the pH of PC based samples in Site 2 was ~2 and a mass loss of 1.59% was recorded. For CAC based samples, those installed in Site 1 had a surface pH ~6 with no mass loss, while those installed in Site 2 had a surface pH ~3 and a mass loss of 0.35% was recorded. Quantification of the total DNA extracted after 4 months from the biofilm on the sample surfaces showed that PC based samples were more bioreceptive (10 ng cm\(^{-2}\) for Site 1 and 155 ng cm\(^{-2}\) for Site 2) than CAC based samples (4 ng cm\(^{-2}\) for Site 1 and 7.5 ng cm\(^{-2}\) for Site 2).
48 ng cm\(^{-2}\) for Site 2). The analysis of condensed water in the PVC tube showed that the pH levels were \(~1\). Notably, ATP concentrations from PC based samples (1.1 \times 10^8 RLU) were much higher than those from CAC based samples (3.8 \times 10^4 RLU), indicating greater microbial activity on PC based samples.

### 3.3 Studies undertaken in Germany

Intensive research work on the corrosion of the Hamburg sewer system led to the construction of a totally controlled breeding chamber in which biogenic H\(_2\)SO\(_4\) corrosion was simulated\(^{[23]}\). This test had the major advantage of allowing colonization of bacteria to occur on the surface of concrete samples in conditions as near to reality as possible. The only accelerating factors were the maintenance of an optimal air flow and temperature of 30°C for the bacteria, and the provision of \(~20\) ppm of H\(_2\)S gas and nutrients in the form of an aerosol spray. At set times between 3 months and 1 year, samples were removed and tested for surface pH and mass loss. The increase in temperature and optimal supply of H\(_2\)S and nutrients were estimated to accelerate the corrosion rate by at least 16 times. Results showed that the surface pH of PC based samples reduced to \(~1\) in 6 months, after which mass loss increased rapidly leading to complete destruction of the samples after \(~350\) days. On the other hand, CAC based samples showed a more rapid decrease in surface pH which tended to stagnate at pH \(~3\) after \(~100\) days, and the mass loss of these samples was considerably lower. This pH level coincides with that at which Al(OH)\(_3\) increases in solubility and suggests that the entry of aluminium ions into solution inhibits the activity of SOB\(^{[13]}\).

Another simulation system built by the Research, Development and Consulting Department of Heidelberger Zement, in which time spans of 3 to 5 months are sufficient to investigate the resistance of several concrete types against biogenic H\(_2\)SO\(_4\) has been used successfully\(^{[24]}\). The test system has two parts; a growth and a reaction part. In the growth part, a 25 L fermentor, monoculture of *acidithiobacilli thiooxidans* is cultivated at optimal conditions. The reaction part consists of a glass bio-reactor where test specimens are stored, and a warm and humid environment is sustained in this part. After 1 month of testing, it was possible to see the difference in deterioration between CAC and PC mortars. After 5 months of testing CAC based samples showed 3 – 4% mass loss, whereas PC based samples showed 18 – 31% mass loss.

### 4 DISCUSSION

Reviewed literature indicates that the composition of cementitious materials is the key factor to resistance against biogenic H\(_2\)SO\(_4\) attack. This resistance is attributed to two fundamental mechanisms of defence – neutralization capacity of the cementitious material and its ability to stifle growth of SOB. The latter seems to be more critical when the measured solubility recorded in Table 1 is put into perspective. For example, the PC/DOL samples had a higher solubility value when measured in an acid digestion test than CAC/SIL samples, and yet the CAC based samples had a lower average measured corrosion rate of 1.9 mm yr\(^{-1}\) as compared to PC based samples with an average measured corrosion rate of 3.1 mm yr\(^{-1}\).

Higher mass loss is related to lower surface pH, particularly when the pH falls below \(~4\). Initially, the surface pH of CAC based samples reduce at a higher rate than that of PC based samples, but levelled off at \(~3.5\). Thus the surface pH on cementitious materials is significant as it indicates the rate of microbial activity.
5 SCOPE OF CURRENT EXPERIMENTAL STUDY

The Virginia Experimental Sewer is a ‘live’ laboratory that presents an aggressive H$_2$SO$_4$ environment for studying the performance of cementitious materials used in the manufacture of concrete sewer pipes. Since its inception, the experimental section has enabled researchers to better understand the performance of such materials, and the knowledge gained so far can effectively be used to correlate performances of cementitious materials in other ‘live’ environments with the view of upgrading the existing predictive approaches and better understanding the key parameters influencing sewer corrosion.

The main objectives of the current study are; (a) to critically review the existing empirical and numerical models used for sulphide and corrosion prediction in the design of concrete sewer pipes with the view of improving them, taking into account in-situ measurements of parameters in a range of sewer environments in South Africa, (b) to further characterise rates of deterioration of concrete sewer pipe materials and relate these to an understanding of their material properties, (c) to define the distinction between the neutralization capacity of a concrete mixture and its ability to stifle growth of acid-generating bacteria. Ultimately, the output is expected to yield a range of materials factors for a variety of cementitious materials that will be used in the existing models used for corrosion prediction of cementitious sewer pipes.

6 CONCLUSIONS

- The underlying mechanism of biogenic H$_2$SO$_4$ attack of cementitious materials is well understood.
- CAC based concretes perform better than PC based concretes when subjected to biogenic H$_2$SO$_4$ attack due to their bacteriostatic effect to SOB.
- Opportunities exist to characterise sewer environments with the view of upgrading models used in biogenic corrosion prediction of cementitious sewer pipes.

7 REFERENCES


THE EFFECTS OF VARIOUS MOULD RELEASE TECHNOLOGIES ON CONCRETE SURFACE FINISH

Coventry K A¹, Richardson A E², and Baines C³,
(1) Northumbria University, Faculty of Engineering and Environment, Newcastle upon Tyne, UK kathryn.coventry@northumbria.ac.uk
(2) Northumbria University, Faculty of Engineering and Environment, Newcastle upon Tyne, UK alan.richardson@northumbria.ac.uk
(3) Northumbria University, Faculty of Engineering and Environment, Newcastle upon Tyne, UK c.baines@northumbria.ac.uk

Abstract
Concrete durability is dependent upon the quality of the concrete cover zone. The type of mould release technology used, has a direct effect on the surface finish of the formation of fair faced concrete, and this in turn affects long term durability and surface finish. This paper examines two mould release agents, one permeable formwork liner and Type 1 polypropylene fibres used with a single mould release agent. Cubes were cast in film faced plywood moulds using a C40 concrete that enabled comparisons to be drawn between the mould release agents.

The range of variables tested comprised of, two (FS and CL) commercially available solvent based mould release agents (MRA), permeable formwork and C1 used with polypropylene fibres.

The results showed that the controlled permeability formwork (CPF) liner produced the fewest blowholes and was the best performing technology used in that respect. As a consequence of this, CPF provided the most likely finish that would provide long term durability. None of the technologies employed produced a completely uniform colour throughout, however the CL MRA cubes and polypropylene fibre reinforced cubes produced the most consistent colouring.
1 INTRODUCTION

This paper examines four different techniques and technologies that are designed to produce concrete release from formwork. The objective is to distinguish which of these four techniques and technologies produces the best quality surface finish with the least defects. The mould release technologies investigated are; FS (reactive release agent), CL (reactive release agent), a CPF liner with a C40 concrete mix. One C1 batch has Type 1 polypropylene fibres added to the concrete mix to determine the effect of the fibres on the surface finish of the concrete.

Homes et al [1] suggest, concrete durability is dependent upon the quality of the concrete cover zone, and in particular the surface finish of the cover zone, as this surface is the only barrier to deleterious agents; that may cause degradation of the steel reinforcement. The concrete cover zone is the cross sectional area of concrete that runs from the exposed external concrete surface to the first bar of reinforcement steel. If this area of concrete is of poor quality, then it can allow the ingress of harmful agents such as carbon dioxide, sulphur oxides and chloride ions.

It can be seen that the surface finish of the concrete can be directly linked to the quality and impermeability of the cover zone. If the surface finish is of poor quality then the ingress of harmful agents and water through surface blemishes such as blow holes and micro cracks is inevitable.

2.0 Release agents, application, fibres and formwork.

2.1 Types of mould release agents (MRA)

Baty and Reynolds [2] divide mould release agents into two categories, which are barrier release agents and reactive release agents.

Barrier release agents frequently stain and discolour the concrete [3]. Baty and Reynolds [2] suggest that barrier release agents would not be the best option to use to if a good quality fair faced concrete surface finish is required. As they have deemed to be an inferior MRA they were not tested.

Reactive release agents are also called chemically active release agents. These work because the chemicals in these complex release agents react with calcium in the freshly poured cement paste. The chemical reaction between the calcium and release agent prevents the setting of a minute surface film of concrete. The chemically active ingredient of the release agent is a type of fatty acid. It is this fatty acid that enables the release of the formwork from the concrete. Baty and Reynolds [2] suggest that if reactive release agents are properly formulated and applied, they can create a decreased amount of blowholes, staining and surface irregularities. Reactive release agents can then be broken down into two further sub categories. Mineral oil based release agents and vegetable oil based release agents. Djelal et al [4] claim that mineral oils are being replaced by vegetable oil based release agents. This is because the vegetable oil based formulations are less harmful to the environment. This is the case especially if they are accidentally spilled on site.

2.1.1 Mould oils used

Two dissimilar MRA’s were used throughout this study. These MRA’s are reactive release agents and they produce a better surface finish than barrier MRA’s. Both have different
physical properties, FS is a thick white MRA and is a petroleum distillate. The vegetable based CL is a much thinner translucent MRA and is a chemical formation of complex soaps comprising of fatty acids and hydrocarbons.

2.2 Coating thickness of MRA’s applied to the formwork
When a MRA is applied to a formwork face; it is essential that the correct amount is applied. Too much MRA can create trapped oil and moisture between the concrete and formwork causing surface blemishes. If not enough release agent is applied then the formwork can stick to the cast concrete. The formwork would then have to be forcibly removed and there is a possibility that it may break a surface layer of concrete with it, resulting in a damaged surface finish.

Djelal et al [5] carried out research to determine the optimum thickness of MRA to be applied to formwork to enable a good quality surface finish. They discovered that a very thin film of around 1 µm to 2 µm thick was the optimum thickness for both vegetable oil release agents and mineral oil release agents.

2.3 Controlled permeability formwork (CPF)
CPF is a system that was developed to improve the durability of a concrete surface. When CPF liners are used, MRA’s are not needed. McKenna [6] describes CPF as “thermally bonded permeable liners that are composed of filter and drain elements, attached in tension to the internal face of a structural support (formwork) by staples.”

McKenna [6], Price [7], Dhir and Hewlett [8] and Figueiras et al [9] agree on the explanation of how CPF liners work. They agree that the CPF extracts excess “bleed” or “mix” water and entrapped air from the concrete/formwork interface and it is transported through the liner without removing the cement particles and fines. Schubel, Warrior and Elliott [10] explain that this results in a reduction of near surface water/cement ratio, which may be an advantage, as near surface laitance may provide a smooth finish, but the quality and durability of the concrete is often of a lower quality.

2.4 Formwork materials
Bennett [11] suggests “the character and quality of a concrete finish will be defined by the character of the formwork in contact with the concrete.” Concrete can be cast against many types of formwork material and each can give a different quality of finish. Bennett [11] goes on to divide formwork face materials into five categories:

- Untreated timber
- Film faced plywood
- Oriented strand board
- Chipboard
- Metal.

Choosing the correct type of formwork material for its selected use is essential if the desired finish is to be achieved. Bennett [11] explains that using metal formwork can be economical as it can be re-used repeatedly and has a longer life span than timber. Bennett [11] further suggests that the new metal formwork face should be roughed up first to reduce the risk of discolouration. Monks [12] listed six types of formwork material in order of
permeability, starting with the most impermeable and ending with the most permeable, are:

1. “Steel
2. Glass reinforced plastic
3. Birch plywood with a bonded phenolic resin film overlay
4. Douglas fir plywood with a resin impregnated film overlay
5. Unsealed plywood
6. Plain smooth timber”

Monks [12] stated that these permeability rankings also correlate with the number of times the form face material can be used. For example steel can be re-used many times and plain smooth timber may only be able to be used once. Monks [12] listed the surface finish outcomes of each type of form face material in the same order, where 1 is best and 6 performs worst. Monks [12] argued that impermeable formwork can leave blow holes in the concrete as more air gets trapped in the formwork/concrete interface. However if a permeable plain smooth timber is used there will be a decrease in blow holes but the grain pattern of the timber can be left on the concrete surface. This may be a desirable feature, but it is beyond the scope of this paper.

Many different formwork materials can provide many different finishes. It can be hard to achieve an aesthetically pleasing defect free surface finish as the materials that give the smooth glossy finish are inherently impermeable and do not allow the release of trapped air too easily. Pallett et al [13] stated that plywood and metal are the two most commonly used formwork materials. Bennett [11] Pallett [14] and the Concrete Society [15] agree that the film faced plywood’s provided the best surface finish out of all the available types of wood formwork. However, Pallett et al [13] go on to say that if the condition of the plywood formwork is poor or damaged, then these blemishes can be passed on to the resultant cast concrete.

As previously stated film faced plywood is the most popular choice of form face material to give a good quality finish as this material has a smooth surface like metal but is more permeable and because of these advantages it was chosen for this research.

2.5 Polypropylene fibres
The fibre adopted was 12mm x 6.5 denier; Type 1 extruded polypropylene fibre (Type1), quality assured to ISO 9001-2 and used at 0.9 kg/m$^3$. The presence of fibres in a concrete mix reduces the amount of bleeding and assists in holding aggregate near the surface of fresh concrete so resulting in a better surface integrity. This claim was examined within the test.

3.0 Test programme methodology
3.1 Sieve tests
Sieve test were carried out on the coarse and fine aggregates to enable the mix design to be carried out. Once this was completed the C40 mix design was completed and is displayed in Table 1.

3.2 Mix design
A C40 characteristic strength concrete was used as being representative of a commonly used mix in construction.
Mix design for 1m³ of concrete

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Water / cement ratio</td>
<td>0.55%</td>
</tr>
<tr>
<td>CEM 1 (42,5 R), - Cement</td>
<td>400 kg</td>
</tr>
<tr>
<td>Mixture of coarse medium and fine sand</td>
<td>830kg</td>
</tr>
<tr>
<td>Graded marine rounded aggregate (20mm max)</td>
<td>970kg</td>
</tr>
</tbody>
</table>

Table 1 – Concrete mix design

3.3 Mould design
Twenty film faced plywood 100x100x100mm cube moulds were purpose made. Five of the moulds had one internal side covered with a CPF liner and these five cubes will make up Batch 3.

3.4 Batching sequence
Batch 1 A single batch of concrete was mixed and this was divided into separate batches for cube manufacture. The procedure for cube manufacture is outlined below:
1. Five moulds were made with the ability to have one side detached.
2. The five detached sides were then sprayed with FS at a pressure of between 0.207 and 0.414 MPa. The five sprayed sides were marked on the external face with the letters FS and a number from 1 to 5.
3. The sides were then reattached to the relevant moulds.
4. C40 concrete was placed carefully into each plywood mould.
5. All five moulds were placed on a vibrating flatbed table and they were vibrated for one minute, or until all air bubbles stopped rising to the surface of the plastic material.
6. The remaining excess concrete was scraped off the top of the mould with a trowel and the exposed surface received a trowelled finish.
7. These 5 cubes were set aside and left to cure for 24 hours.
8. After 24 hours the cubes were stripped from their plywood moulds and placed in a heated cube tank (heated to 20⁰C) and left to cure for fourteen days.
9. After the fourteen days the cubes were removed from the tank and tested for their surface finish qualities.
Batch 2 was made the same way as Batch 1 however the FS MRA was replaced by CL and the external face of the sprayed side was marked with CL and a number from 1 to 5.
Batch 3 consisted of 5 cubes, each with one internal side covered in a CPF liner. These cubes were made in the following way:
- The side of the mould covered by the CPF liner had its external face marked with the letters CPF and a number from 1 to 5.
- The process then continued as from step 4 in Batch 1.
Batch 4 included polypropylene fibres. This remaining concrete was remixed with polypropylene fibres added to it at a rate of 0.9g per 1m³ of concrete.

3.5 Methods of testing the surface finish of the cubes
Once the cubes were cast and left to cure in a heated curing tank for 14 days they were tested with regards to their surface finish properties. The test details are described in section 3.6.1.

3.5.1 Modification of the point count method
TR 32 [16] outlines a test for examining the entrained air in concrete. This test however can be performed in a similar way to examine any air voids (blow holes) on the surface of concrete.

The test was performed as follows:
The 100mm$^2$ face of each cube to be tested had a 10mm spaced border drawn onto it. The space inside the border was divided into 20mm strips (Figure 4). Each strip was labelled with the letters A to D. This was to allow identification of where the blow holes are on each cube when analysing the results.

![Gridlines on concrete cube face with a blowhole identified in strip](image)

A visual inspection was carried out on each 20mm strip. Where a blow hole was identified within the boundaries of a strip (circled in Figure 4) a variety of additional parameters were identified. The American Society for Testing Materials [17] state that the conditions of a surface void, (blowhole) can be broken down into various parts:
- The shape. Is it spherical or non-spherical?
- The colour. Is there a colour change between the inside of the void and the external concrete face?
- The grading of the void. What is the size of the void?

Each blow hole identified was examined by a crack detecting microscope at a 40x magnification for the above qualities.

4.0 Results.
The average blow hole surface area and the percentage of the concrete face covered in blow holes was very small using a CPF liner when compared to the other release agents. Figures 7 to 9 visually display the findings.

Figure 7 – Average amount of blowholes found on each batch of cubes

Figure 8 – Average percentage of surface area covered by blowholes for each batch
Tables 6 to 9 compare the amount of spherical to non-spherical blowholes found on each batch of cubes. This is relevant as spherical blowholes cover a smaller surface area and look slightly better than their predominantly larger non-spherical counterparts.

<table>
<thead>
<tr>
<th>Cube reference</th>
<th>Total No. of blowholes</th>
<th>% of these that are spherical</th>
<th>% of these that are non-spherical</th>
</tr>
</thead>
<tbody>
<tr>
<td>CL1</td>
<td>54</td>
<td>94.4%</td>
<td>5.6%</td>
</tr>
<tr>
<td>CL2</td>
<td>63</td>
<td>98.4%</td>
<td>1.6%</td>
</tr>
<tr>
<td>CL3</td>
<td>56</td>
<td>98.2%</td>
<td>1.8%</td>
</tr>
<tr>
<td>CL4</td>
<td>57</td>
<td>98.2%</td>
<td>1.8%</td>
</tr>
<tr>
<td>CL5</td>
<td>52</td>
<td>98.1%</td>
<td>1.9%</td>
</tr>
<tr>
<td>Totals for batch</td>
<td>282</td>
<td>97.5%</td>
<td>2.5%</td>
</tr>
</tbody>
</table>

Table 6 – CL - MRA, spherical to non-spherical blowhole comparison

<table>
<thead>
<tr>
<th>Cube reference</th>
<th>Total No. of blowholes</th>
<th>% of these that are spherical</th>
<th>% of these that are non-spherical</th>
</tr>
</thead>
<tbody>
<tr>
<td>FS1</td>
<td>55</td>
<td>100%</td>
<td>0%</td>
</tr>
<tr>
<td>FS2</td>
<td>53</td>
<td>94.3%</td>
<td>5.7%</td>
</tr>
<tr>
<td>FS3</td>
<td>48</td>
<td>91.7%</td>
<td>8.3%</td>
</tr>
<tr>
<td>FS4</td>
<td>47</td>
<td>74.5%</td>
<td>25.5%</td>
</tr>
<tr>
<td>FS5</td>
<td>19</td>
<td>84.2%</td>
<td>15.8%</td>
</tr>
<tr>
<td>Totals for batch</td>
<td>222</td>
<td>90.1%</td>
<td>9.9%</td>
</tr>
</tbody>
</table>

Table 7 – F1 MRA, spherical to non-spherical blowhole comparison

Figure 9– Average individual blowhole surface area size for each batch
5.0 Conclusions

CPF liner produced a concrete surface finish with by far the fewest blowholes. When a blowhole was found on the CPF cast surface it was small, spherical and was not significant enough to adversely affect the sight of the concrete. The concrete had a slightly matt texture due to the imprint from the liner, but was still the most defect free surface.

The two MRA’s used produced similar results, both producing very similar sized blowholes. The CL MRA produced a greater amount of blowholes than the FS and therefore more of the cubes surface area was covered by these surface voids. However in terms of blowhole shape FS produced a greater percentage of non-spherical blowholes than CL.

Finally the polypropylene fibre reinforced concrete produced the greatest number and largest blowholes out of all four batches of cubes. 4.6% of which were non-spherical. This led to a fragmented, rough looking and non-uniform surface finish.

6.0 References


INFLUENCE OF SERVICE LIFE ON BUILDING LCA

Endrit HOXHA (1), (2), Manuel BAZZANA (1) Guillaume HABERT (3), Jacques CHEVALIER (1) and Robert LE ROY (2), (4)

(1) Université Paris-Est, Centre Scientifique et Technique des Bâtiments (CSTB), Division Environnement et Ingénierie du Cycle de vie, 24 rue Joseph Fourier 38400 Saint Martin D’Hères, France. endrit.hoxha@cstb.fr, manuel.bazzana@cstb.fr.

(2) Université Paris-Est, UMR NAVIER, Ecole des Ponts Tech, 3-8 av Blaise Pascal, Cité Descartes Champs-sur-Marne, 77455 Marne-la-Vallée Cedex 2, France.

(3) Institute of construction and infrastructure management, Swiss Federal Institute of Technology (ETH Zürich), Wolfgang Pauli Strasse 15, 8092 Zürich, Switzerland.

(4) GSA Laboratory, ENSAPM, 14 rue Bonaparte 75006 Paris, France.

Abstract
The current study addresses the relation between service life of building components and uncertainties of Life Cycle Assessment (LCA) results of single houses. LCA practitioners have to deal with service life data and estimation when performing LCA, but they often lack reliable sources of information to do so or face contradictory information. We based the current work on several known Service life data sources that could be used as reference to justify LCA calculations and estimated the influence of these data over whole building LCA results.

After describing current practices regarding the practical uses of the concepts of service life and products building in the LCA community, we present an approach used to define service life intervals used as input data for a series of case studies. These values are applied to 16 distinct LCA studies of single houses. The influence of the service life data used as input is then discussed.

1 INTRODUCTION

Environmental harmful activities differ from one sector to another, but it is well known that one of the biggest contribution related to GHG emissions, ozone depletion, destruction of natural habitats, etc., are due to the building sector [1]. Urgent changes in several fields are required: energy savings, production and use of materials, use of renewable resources, reuse and recycling of building materials, minimization of GHG emissions, etc. [2]. To do so and since more than 30 years, scientific community have developed and validated the life cycle assessment (LCA) methodology [3]. Life cycle assessment is a “cradle-to-grave” approach for assessing industrial systems. LCA is a cradle to grave approach starting from the extraction of raw materials to the production of the product, the transportation and construction activities, the use phase and the end of life. Considering ISO 14040 the LCA process is a systematic,
phased approach and consists of four components: goal definition and scoping, inventory analysis, impact assessment, and interpretation [4]. The built environment is also a primary source of environmental impacts, not just due to initial construction phase but also from emissions occurring during the use phase. While it is well known that energy and water consumption drive an important part of the environmental impacts during the use phase, the existing building stock also requires continuous investments for repair and renovations, which contribute to overall life cycle impacts [5]. In the building LCA field, service lives of products are used to calculate the number of replacements during the service life of the building. To be able to focus on the most relevant aspects of the building, it is fundamental to accurately quantify, for each components, which phase of the life cycle has the main contribution to the environmental impacts. The reliability of LCA in the building sector as in the other industrial sectors is affected by the uncertainties and variability related to the data used for calculation, the methodology used and the user’s knowledge. This study focuses only on the uncertainties and variability of the service life of buildings elements and their influence in the LCA results. Service life prediction of building and building components has been discussed since 1970. Different approaches have been developed, from detailed stochastic model to empiric estimations, each having unique applications and limitations [6]. According to the ISO 15686 service life of a product is function of seven factors: Inherent performance level of components, design level, work execution level, indoor environment, outdoor environment, conditions of use and maintenance level [7]. Service life prediction of building elements, components or product can be a complex and time-consuming process, which might explain the lack of guidance on how to use SL data in building LCA. Yet it is fundamental to estimate the influence of service life in LCA of building as it can affect the reliability of the results. The purpose of our study is the identification of key service life that has the highest influence on the result. The methodology used is based on a statistical method and it includes both sensitivity and uncertainty analysis. As shown in figure 1, a parameter which has a small sensitivity but large uncertainty may be just as important as a parameter with larger sensitivity but smaller uncertainty.

![Figure1: The difference between an input with large sensitivity but small uncertainty and an input with small sensitivity but large uncertainty](image)

We have modelled 16 single houses with the aim of identifying the building elements that have the greatest contribution to the LCA results and their uncertainty, based on service life data collected from literature.
2 CONTRIBUTION ANALYSIS APPLIED TO BUILDING LCA

The contribution analysis is applied to the equation describing the environmental performances of buildings given by the standard 15978 [8]. After Hoxha et al [9] the partial derivative (simple sensitivity coefficients) of the different building material or element is equal to 1:

$$\frac{\partial I_f}{\partial I_{f,i}} = 1 \quad \forall \ i \in [1, c] \tag{1}$$

The equations of normalized sensitivity of materials have the form of:

$$S(I_{f,i}) = \frac{I_{0,i}}{I_f} \quad \forall \ i \in [1, c] \tag{2}$$

where:

$$I_{0,i} = m_i^0 \cdot k_{f,i}^0 \cdot n_i^0 \tag{3}$$

is the nominal value of environmental performance of material i calculated by the nominal value of mass, impact coefficient and number of use of material during the service life of building. The nominal values are here considered as the mean values.

$$I_f^0 = \sum_{i=1}^{c} I_{f,i} \tag{4}$$

is the sum of nominal value of environmental performances of materials. In our case the physical concept of the normalized sensitivity coefficients are that they measure the relative contribution of material impact to the building impact. Also we have to perform the uncertainty analysis for a complete contribution analysis. The relative contributor coefficients after the equation (5) have the form:

$$RC(I_{e,i}) = \frac{\sigma_{I_{f,i}}}{\sum_{i=1}^{c} \sigma_{I_{f,i}}} \times 100 \tag{5}$$

In the next section the methodology is applied to a set of 16 individual houses.

3 STUDY

3.1 Presentation of houses case studies

In this study are modelled a sample of 16 French single houses. Building LCA models of these buildings were taken from CSTB [10]. The surface are of these houses varies between 91 m$^2$ to 298 m$^2$. They have different types of structure (reinforcing concrete structure, wood structure); different types of wall (block concrete, clay brick, cellular concrete block, wood, etc.,); different types of thermo insulation (rock wool, glass wool, wood wool, etc.,); different types of tiles (clay, concrete, slate); etc.

3.2 LCA data and uncertainties sources

The environmental impact of each building material or product is based on values from the environmental products declarations (EPD) available from the INIES database [12]. The mean and standard deviation for the number of replacement of materials and components during the service life of building are calculated using different references from literature. It should be noted that the consistency of these data has been verified, but only to a limited extent. Furthermore, the goal of this data collection work was more to obtain a panel of data a building LCA practitioner would likely consider as a reliable source of information than to seek for SL data that would fit to each of the 16 building model context. By convention, structural materials are assumed to have the same service life as the building (50 years in most
of the cases). SL data used are presented figure 2. Each reference may correspond to several deterministic service life data.

![Diagram showing service life data for various materials](diagram.png)

**Figure 2: Used service life data. Minimum, mean and maximum are calculated from a number of reference values for each material.**

## 4 RESULTS

Figure 2 show the results obtained from the 16 single houses for the GWP, expressed per net floor area. Impacts from the structural and non-structural elements are distinguished, and the variability due to the choice of service life is represented as the minimum and maximum overall impact for each project. Despite some clear differences in terms of absolute values the uncertainties related to the service life of non-structural components would lead to difficulties if one wants to compare the different projects. Maybe the most typical example is “project 13”, which exhibits the lowest minimum and median values (due to taking into account CO₂
storage from the wood structure, hence the negative value) but a rather important maximum value.

![Global Warming Potential](image)

Figure 3: GWP of the 16 building projects (material contribution)

The sensitivity analysis allows identifying the most important contributors to GWP. It is thus possible to isolate products and materials type that exhibit a limited impacts (e.g. natural stone, acrylic products), from those with a more important contribution (e.g. windows). Rock wool and glass wool have a minimum sensitivity equal to zero, which might suggest that there is no building LCA that used both of them at the same time, while plaster products are involved in each study (minimum contribution is 2.5%).

The uncertainty analysis allows identifying the most important contributors to the uncertainty of GWP. It is remarkable from the figure 4 that materials such are: glass wool and non-structural wood and PVC have a greatest influence in the uncertainty of the GWP. The service lives of copper pipes, polyethylene product, linoleum etc., haven’t an important contribution on the uncertainty of GWP.
CONCLUSION AND PERSPECTIVES

The paper was focused on the sensitivity and uncertainty analysis of the single houses. In the uncertainty propagation, only the service life parameter was considered as uncertain. Contribution analysis allowed the identifications of materials and products that have the highest influence on the environmental performances of buildings as well as the identification of materials which have the highest influence on the uncertainties of global warming potential indicator. The uncertainties of service life of materials such as: gravel, linoleum polyethylene
products etc., can be neglected in a LCA modification. Counter to these materials the materials such are PVC, polyurethane etc., should be determined in a more detailed study.

A possible perspective of this study can be the identification of the service lives that have the greatest contributor to the uncertainty of environmental indicator of other type of buildings (apartment building, offices etc.).

REFERENCES


CHARACTERIZATION THROUGH MERCURY POROSIMETRY OF TORTUOSITY FACTORS OF CONCRETES CONTAINING MINERAL ADDITIONS

C. Andrade(1), N. Rebolledo(1), E. Menéndez(1), R. Perez(2), M. Baz(2)

(1) Institute of Construction Sciences “Eduardo Torroja” CSIC-Spain
(2) Grupo Unidos por el Canal –GUPC-(Sacyr) Panama

Abstract
Tortuosity is related to concrete porosity by being a concept appraising departure of the pores from a regular cylindrical shape. One of the manners for its characterization is through the ratio between the resistivity of the porous material and that of the pore solution which, following Archie’s law is equal to the porosity to the power of an exponent which results for concretes around 2. This exponent has been called tortuosity factor in a previous paper where has been shown good agreement with that calculated from a pore size distribution cumulative curve obtained through mercury porosimetry. In present paper the study is extended to concretes containing mineral additions made for the new locks of Panama Canal. The results show that the agreement with present mixes between the calculation of the tortuosity factor from resistivity or from the MIP curve is not good for all mixes and then, it is suggested to use modified Archie’s law until a full enough understanding is found with the pore size distribution.

1 INTRODUCTION
In the last years models for service life prediction, mainly for corrosion initiation, have been developed and are tried to be included in standards [1-2]. The time to corrosion initiation is usually modeled by a diffusion process characterized by a rate parameter, the diffusion coefficient, which can encounter or not the chloride and carbon dioxide binding with cement phases. Transport and reaction with cement phases are then the two main processes to be quantified. The transport process inside the concrete is mainly controlled by the porosity and the so called, “tortuosity” [3-4], which isolated or in combination with another parameter called “constrictivity” tries to model the departure of the pore network from being parallel ideal cylinders, as shown in figure 1-left, to be a complex network of pores of different sizes along its length and with different orientations (figure 1-right). The irregular shape of concrete pores and their variety of sizes makes very difficult to find a manner to characterize the tortuosity/constrictivity and, in consequence, to model the transport properties based in the values of total porosity. Only by having a mathematical expression of the porosity and tortuosity it is possible to predict the transport of species through the porous medium.
Several researchers have tried to define and characterize the tortuosity [3-5] of the concrete pore network. The most useful proposals are based in the empirical law of Archie [6] who developed the expression for the transport in rocks: $\rho = \rho_0 \cdot \varepsilon^{-b}$. This formula expresses that the relation between the transport (resistivity) of the species in a liquid and in the porous material depends on the porosity multiplied or powered by a factor whose value was identified to range around $b=2$ in the case of concrete. Conceptually, looking at equation 1, if the $\tau$ factor is 1 then, the resistivity of the porous material would be the inverse of the volumetric fraction times of the resistivity of the pore solution. Then, if we assume a cylindrical shape in the pores, the porosity would be $V = \pi \cdot r^2 \cdot L$. Being the porosity of the concrete around 10% in volume, that would mean that its resistivity would be around ten times that of the pore solution. However, this relation between the resistivity of the pore solution and of the concrete is more than 100 or 1000 times. In order to try to find that relation, in a previous work [7] of the authors, they have proposed an alternative method to obtain the tortuosity from a mercury intrusion porosimetry, MIP [8]. The method consisted in:

1) To consider the power exponent “$b$” of equation 1 as “tortuosity factor” $= \tau$ encountering all characteristics of the pore network departing from ideal cylindrical shape, 2) To obtain the $\tau$ value from the slope or steepness of the accumulated pore size distribution MIP curve by fitting in that curve the following expression which resembles Archie’s law:

$$\phi_{th} = \phi_0 \cdot \varepsilon^{-\tau}$$

(1)

Where $\phi_{th}$ is the pore diameter at any porosity and $\phi_0$ is the smallest diameter that can be measured in the MIP equipment used (6.6 x10-9 m in present case). In [7] several values of $\tau$ were calculated from MIP curves and from resistivity values for several concrete types and resulted close enough between them. In present work, tortuosity factors are calculated for the concretes of Panama Canal in which several mix proportions with different mineral additions have been used. The old Panama Canal will be centenary in present year 2014. The new locks are built with concretes designed for, at least, the same durability having additions of pozzolanic nature and silica fume.

The reason to have the values of the resistivity of the concretes of Panama Canal is because it has been used for controlling its service life expectation together with a model based in Fick’s law. Due to the relation between resistivity and the diffusion coefficient [9] it has been
postulated in previous works [10] the use of the electrical resistivity for the prediction of service life of concrete structures in order to overcome the limitations of the modeling based in the measurement of the diffusion coefficient. Thus, the electrical resistivity in saturated conditions can be used as an indicator or introduced in a “square root” model relating penetration depth and time. Besides, the resistivity can be measured in a non-destructive manner, and therefore its values can be monitored over time in a relatively easy and inexpensive manner. The model based in the resistivity can be applied not only to the initiation period but also to the corrosion propagation [10]. Present paper presents the values of $\tau$ calculated from new Panama Canal concrete mixes in order to increase the knowledge between tortuosity factor $\tau$ and pore size distribution and resistivity.

2 EXPERIMENTAL PROCEDURE

The study is illustrated with only four mixes made for the both Oceans connections of the Canal: A for Atlantic and P for Pacific (A3 and P3, A11 and P11, A50 and P50 and A56 and P56), representative of the over 50 studied. Their compositions are listed in Table 1.

Table 1. Composition of four concrete mixes studied

<table>
<thead>
<tr>
<th>GUPC Code</th>
<th>Atlantic</th>
<th>Pacific</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixture</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5777</td>
<td>5935</td>
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</tr>
<tr>
<td></td>
<td>7734</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>5481</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5924</td>
</tr>
<tr>
<td>Cement Type kg/m³</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Panamá CEM II</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>282</td>
<td>330</td>
</tr>
<tr>
<td></td>
<td>(0PN 6.7SF)</td>
<td>(12.5PN 5.8SF)</td>
</tr>
<tr>
<td></td>
<td>290</td>
<td>332</td>
</tr>
<tr>
<td></td>
<td>(0PN 0.0SF)</td>
<td>(13.0PN 0.0SF)</td>
</tr>
<tr>
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<td>282</td>
<td>330</td>
</tr>
<tr>
<td></td>
<td>(0PN 6.7SF)</td>
<td>(12.5PN 5.8SF)</td>
</tr>
<tr>
<td></td>
<td>332</td>
<td>282</td>
</tr>
<tr>
<td></td>
<td>(0PN 0.0SF)</td>
<td>(13.0PN 0.0SF)</td>
</tr>
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<td></td>
<td></td>
<td>290</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(0PN 0.0SF)</td>
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<tr>
<td>Pozzolan kg/m³</td>
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<td></td>
<td>43</td>
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<tr>
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<tr>
<td>Silica Fume kg/m³</td>
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<td></td>
</tr>
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<td>19</td>
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<tr>
<td>0</td>
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<tr>
<td>W/B ratio</td>
<td>0.34</td>
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<td>SuperFine Sand kg/m³</td>
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<td></td>
<td>44</td>
</tr>
<tr>
<td>44</td>
<td></td>
<td>86</td>
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<tr>
<td>Fine Sand (0 – 4.75mm) kg/m³</td>
<td></td>
<td></td>
</tr>
<tr>
<td>686</td>
<td></td>
<td>716</td>
</tr>
<tr>
<td>666</td>
<td>702</td>
<td>686</td>
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<tr>
<td>662</td>
<td></td>
<td>703</td>
</tr>
<tr>
<td>703</td>
<td></td>
<td>595</td>
</tr>
<tr>
<td>Coarse Aggregate (4.75 – 19mm) kg/m³</td>
<td></td>
<td></td>
</tr>
<tr>
<td>707</td>
<td></td>
<td>693</td>
</tr>
<tr>
<td>732</td>
<td>720</td>
<td>707</td>
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<tr>
<td>727</td>
<td></td>
<td>701</td>
</tr>
<tr>
<td>701</td>
<td></td>
<td>691</td>
</tr>
<tr>
<td>Coarse Aggregate (19 – 37.5mm) kg/m³</td>
<td></td>
<td></td>
</tr>
<tr>
<td>647</td>
<td></td>
<td>633</td>
</tr>
<tr>
<td>670</td>
<td>657</td>
<td>647</td>
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<td>641</td>
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<tr>
<td>641</td>
<td></td>
<td>630</td>
</tr>
<tr>
<td>Strength at 28 days MPa</td>
<td>52.1</td>
<td>59.3</td>
</tr>
<tr>
<td>42.6</td>
<td>51.6</td>
<td>38.6</td>
</tr>
<tr>
<td>43.2</td>
<td></td>
<td>45.3</td>
</tr>
<tr>
<td>45.3</td>
<td></td>
<td>48.1</td>
</tr>
</tbody>
</table>
Cement type CEM II (ASTM) was used, with additions such as natural pozzolan and silica fume. A “superfine” fraction of sand, also with pozzolanic properties, was employed, although for batching purposes it was regarded as an aggregate rather than a mineral addition. The w/c ratio was on the order of 0.3. The 15x30-cm cylindrical specimens studied were prepared at a real plant in Panama, where they were cured in a humidity chamber for 28 days and later sealed in water-tight packaging for continued curing and shipped to Madrid, where they arrived 38 days after casting.

Upon receipt of the materials, three specimens of each composition were tested by means of the chloride penetration (ASTM C1543) and for voids volume by ASTM C 642. Porosity by mercury intrusion porosimetry (MIP) was measured on a Micromeretics PORESIZER mercury intrusion porosimeter until around $\phi_0 = 6$ nm of pore diameter. Concrete resistivity was determined by Wenner four-point test described in Spanish standard UNE 83988-2 [11] as is illustrated in figure 2.

![Figure 2 Resistivity measured with a four-point resistivimeter](image)

In order to calculate the value of $\tau$ [7], in the MIP cumulative curves it is identified the largest pore existing in certain amount (called pore threshold, $\phi_{th}$), that is, in the larger pore range it is identified the pore diameter at which the curve start to bend or increase. Its selection in the curves is not obvious because the increase in volume is not sudden but smooth in most of pore sizes distributions obtained. With that value and the smallest $\phi_0$ that can be detected with the porosimeter (around 6 nm in diameter) it was calculated the $\tau$ value by means of expression 3. The $\tau$ value from resistivity were calculated from expression 4 in which the $\rho_0$ value was made $= 50 \, \Omega \cdot \text{m}$.

$$\rho = \rho_0 \cdot e^{-\tau}$$

(2)

3 RESULTS

Figure 3 shows the cumulative pore size distributions of the concrete mixes and the differential one. As an example in figure 3, is shown with a black arrow, the pore threshold selected in the MIP curve of mix A50. This visual and approximate selection was also made for the rest of concrete mixes. The pore threshold values are given in Table 2.
In Table 2 are given the values of the Total MIP porosity (in % in volume), the values of the Resistivity (Ω⋅m) at 38 days, the values of pore diameter threshold, $\phi_{th}$, identified in the MIP curves and $\tau$ values obtained through expression 1 and through expression 2. The $\phi_{th}$ values from resistivity were calculated by calculating first the $\tau$ value through expression 2 and introducing these values in expression 1 the corresponding $\phi_{th}$ values are obtained for comparison. The values are given in figures 4 and 5.

Figure 4 Differential Intrusion Porosimetry for Atlantic and Pacific concrete mixes.

<table>
<thead>
<tr>
<th>SMC-A50</th>
<th>SMC-A51</th>
<th>SMC-A56</th>
<th>SMC-A57</th>
<th>SMC-P50</th>
<th>SMC-P51</th>
<th>SMC-P56</th>
<th>SMC-P57</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total porosity (% in volume of sample)</td>
<td>9.34</td>
<td>9.12</td>
<td>10.00</td>
<td>10.70</td>
<td>10.95</td>
<td>11.10</td>
<td>7.55</td>
</tr>
<tr>
<td>Resistivity at 38 days (Ω⋅m)</td>
<td>263.56</td>
<td>226.32</td>
<td>146.33</td>
<td>91.64</td>
<td>152.89</td>
<td>149.78</td>
<td>105.56</td>
</tr>
<tr>
<td>$\phi_{th}$ (µm) from MIP (expression 3)</td>
<td>0.04</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
<td>2.0</td>
<td>0.08</td>
<td>0.1</td>
</tr>
<tr>
<td>$\phi_{th}$ (µm) from resistivity (expression 4)</td>
<td>2.4</td>
<td>1.9</td>
<td>1.9</td>
<td>1.77</td>
<td>3.24</td>
<td>3.28</td>
<td>0.43</td>
</tr>
<tr>
<td>$\tau$ from MIP (expression 3)</td>
<td>0.8</td>
<td>1.54</td>
<td>1.48</td>
<td>1.44</td>
<td>2.39</td>
<td>1.03</td>
<td>1.34</td>
</tr>
<tr>
<td>$\tau$ from Resistivity (expression 4)</td>
<td>2.64</td>
<td>2.55</td>
<td>2.46</td>
<td>2.36</td>
<td>2.59</td>
<td>2.28</td>
<td>2.07</td>
</tr>
</tbody>
</table>
4 DISCUSSION

Both methods of calculation, from resistivity or from MIP, give values of parameters: pore threshold and tortuosity factor, which are of a reasonable order of magnitude, although they differ very much among the two methods in several of the mixes. In spite of the disagreements, the close results found for some mixes indicate the need to continue the study in order to find the reasons of the disagreements. It has to be stressed that the mixes were made with mineral additions and all of them with very low w/c ratios. Then the values of $\tau$ should have been very similar, which is shown better when the calculation is made from the resistivity values.
Figure 6 Relation between the pore threshold $\phi_{th}$ values and the $\tau$ factors calculated from expression 3 (diamonds) or using first expression 4 (squares).

It was already recognized the previous paper [7] the subjectivity of the use of MIP curves for the calculation due the identification of the pore threshold was made as in present paper in an approximate visual manner and that expression 1 do not fully fit into all shapes of the MIP cumulative curves, because these curves are not always of regular slope. At this respect figure 6 gives the relation between the $\tau$ factors calculated through expression 1 from the $\phi_{th}$ visually selected: they seem to be reasonably well selected due to the logarithmic relation found. In the case of the $\phi_{th}$ values obtained calculating first the $\tau$ factor from resistivity values the dependence is exponential also with relatively high regression coefficient. Then, both methods seem to be reasonably correct.

Then, it seems up to present that the best to obtain the tortuosity factor is to use expression 2, as it gives the relation between the resistivity in the porous media, $\rho$, with respect to that in the liquid, $\rho_0$, in an objective manner while the use of MIP curves is still needing further work. It is necessary to continue exploring either to find another mathematical formula different than expression 1 for the MIP curves (which furthermore, in some cases are bimodal with two maxima P50 and A57 in present study), or another possibility, is to fit expression 1 to only the median part of the cumulative curve with the corresponding porosity. They will be tried in a next work.

5 CONCLUSIONS

The conclusions that can be drawn up are:
1. The tortuosity factor values can be estimated by type or family of cement type by means of measuring the porosity and the resistivity. The values found in present research are. For type I cement $\tau$= 1.9, for type II-AV $\tau$= 2.3 and for type II AP $\tau$= 1.6.
2. The tortuosity factor can be deduced from the w/c ratio as it seems that it may be a constant value and then from equation 6 $\phi_{th}= \phi_0 \cdot \varepsilon^{\tau}$, in which the porosity can be calculated through Powers expression.
The conclusions that can be drawn up are:
3. Tortuosity factors can be calculated from a modified Archie’s law, $\rho=\rho_0 \cdot \varepsilon^{\tau}$, in which the exponent of the porosity is considered to encounter of the features of the pore network making not linear the relation between porosity and resistivity.
4. The tortuosity factor can be deduced also from the shape of the MIP curves through the identification of the pore threshold at which the large range of pores start to increase and using expression 3: \( \phi_t = \phi_0 e^{-\tau} \).

5. Up to present a good enough accordance between both methods has not been found for all cases analyzed and then, further mathematical treatment is necessary with the cumulative pore sizes curves through MIP or any other porosimetry method able to give pore size distributions of the capillary pores range.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the assistance received from GUPC and CICP, a private company, in conducting the study, as well as ACP’s willingness to accept new approaches to service life calculations.

REFERENCES

INFLUENCE OF REDISPERSIBLE POLYMER POWDERS ON DAMAGE MECHANISMS OF ETICS RENDER MORTARS

Laura Valdès(1, 2), Marie Aulagnier(2), Evelyne Prat(2) and Mohend Chaouche(1)

(1) Laboratoire de Mécanique et Technologie — Cachan (ENS Cachan/CNRS UMR8535). 61, av. du Président Wilson F-94230 Cachan, France
(2) Centre d’Innovation de ParexGroup, BP 5 — 38, rue du Montmurier, ZI Parc des Chesnes 38291 Saint Quentin Fallavier Lyon, France

Abstract
We consider the impact of redispersible polymer powders (RPP) on the mechanical behavior of mortars used as finishing and decorative coats in ETICS systems. Disk-like mortar samples were submitted to indirect tensile strains through the splitting test (Brazilian test), and the damage state corresponding to various levels of strain was analyzed using two different techniques. A home-developed image analysis technique (Correli-Q4) was used to characterize the deformation field and the crack network percolating at the sample surface, while ex-situ 3D X-ray computed microtomography (\(\mu\)-CT) was used to characterize the crack network in volume. As expected, addition of RPP significantly increased the deformability of the material. On the other hand, the evolution of the peak splitting force versus RPP content was found to be non-monotonic, displaying a minimum value. The \(\mu\)-CT analysis showed that the high deformability of the material containing RPP is not due to eventual micro cracking, at least within the spatial resolution of the \(\mu\)-CT considered. \(\mu\)-CT results also highlighted the high heterogeneity of the main splitting fracture along the sample axis.

1 INTRODUCTION
Durability of ETICS systems, subject to cyclic evolution of the environmental hydrothermal conditions, is determined at great extent by the ability of the system “rendering mortar/ base coat/ mesh and finish” to sustain the very large deformations experienced by the insulation board (polystyrene, mineral wool, etc.). Redispersible polymer powders (RPP) are often included in the mix-design of the mortars in order to obtain such particular mechanical properties. In this investigation we consider the influence of this additive on the mechanical properties of a commercial ETICS rendering mortar. Generally accepted working mechanism of RPP is the following [1-2] (assuming that the temperature is above the minimum film forming temperature MFFT of the polymer). The latex particles first disperse in the kneading water during the mixing process. As the water leaves the poral space through evaporation and hydration, the latex particles agglomerate and then form ordered arrays of hexagonal packing.
of deformed particles. Ultimately a continuous elastomeric homogeneous film forms through molecular interdiffusion giving rise to an organo-mineral composite. Several studies have been reported in the literature regarding the mechanical properties of latex-modified mortars. Most of the studies considered the mechanical behavior in bending (3-points bending) or compression. Although some of the reported results are contradictory, one can retain that latex addition of RPP leads to a significant increase of the flexural strength, but in general a reduction of the compressive strength [3-5]. In most published studies the full mechanical behavior is not reported and only the ultimate resistance is discussed. Mechanical behavior in pure tension is difficult to perform, and to the authors knowledge there are no reported results in the literature regarding this issue.

There is no general agreement regarding the physical origins of the peculiar mechanical behavior of latex-modified mortars. More investigation, including in particular mechanical testing associated with microstructure analysis, is therefore needed. In the present investigation we consider the mechanical behavior of latex-modified mortars, including damage, through the splitting test (also known as Brazilian test). Under some circumstances this test allows to apply an indirect tensile strain.

2 MATERIALS AND EXPERIMENTAL METHODS

2.1 Materials

The reference mortar used is a commercial product reformulated without RPP. Different amounts of RPP (Copolymer of vinyl acetate and ethylene, Mfft = 4°C) were added to the base mortar during mixing. The RPP concentration was varied between 0 and 8% by weight with respect to the total powder. Tap water was used and its dosage rate was fixed to 17% relative to total powder mass for all the mixes. A laboratory planetary mixer (Perrier) was used. The RPP was first mixed with the dry mix. The required amount of water was then added and the paste was mixed following the standard method: 30 s mixing at low rotation speed (∼60 rpm); (iii) stopping during 30 s to gather the mortar into the center of the bowl; and mixing again for 60 s at the same rotation speed.

The mortar pastes were casted in cardboard cylindrical molds with two different diameters (4 cm and 7 cm). The mortar specimens were demolded after 24 h and cured over 28 days under controlled environmental conditions (20°C and 50% humidity). The cylindrical specimens were then cut into two sets of disk-like samples on which splitting tests would be performed. Under some conditions the splitting test allows to apply indirectly an approximate tensile strain. One of these conditions is that the compressive strength of the material should be much larger than its tensile strength. This is the case for instance for concretes or rocks. In addition, the ratio between the diameter of the sample and its thickness should be large enough [6]. The sample sizes were then chosen to be (4 cm diameter/0.9 cm thickness and 7 cm diameter/1.5 cm thickness).

2.2 Experimental procedure

The splitting tests were performed using a servo-hydraulic testing machine from MTS. The specimen was mounted between two parallel plates and subjected to compression along its diameter by displacing one of the plates at constant speed (0.003 mm/s). Since the deformation rate is quite small one can consider that the tests were quasi-static. The compressive force was measured as a function of the instantaneous displacement (or time).
Two types of mechanical tests were performed: Continuous splitting tests until complete rupture of the specimen and cycling tests. The latter consisted of applying a constant displacement rate (at 0.003 mm/s) until reaching a given level of the splitting force, unloading to analyze the damage state of the sample with microtomography, reloading the sample to apply a larger deformation and so on. The relaxation of the sample between two successive loadings is then ignored in these ex-situ tomographic studies.

The strain field was determined using a home-developed digital image correlation (DIC) technique. This technique is described in various publications (see for instance Hild and Roux [7] for a review). In few words, two successive digital images, corresponding to different mechanical loadings and represented by the distribution functions of the gray level \( f(x) \) and \( g(x+u(x)) \) are compared. The displacement field \( u(x) \) is determined such that to get the best match between the two functions. This is obtained by assuming gray level conservation \( f(x)=g(x+u(x)) \). Since in practice between the two images there are always some experimental artifacts that may induce gray level variation (for instance noise), the actual displacement field is rather obtained by minimizing the global residual \( R \) in the region of interest (ROI):

\[
R^2 = \int_{\text{ROI}} [g(x+u(x)) - f(x)]^2 dx
\]

The residual field is related in particular to kinematic discontinuities and is very useful in detecting early appearance of cracking. A finite element type software (Correli-Q4) based on this approach has been developed by Hild and collaborators [7] and used in the present investigation.

Digital images were taken each 6s using a high resolution numerical camera. Although cement-based materials already display natural pattern, a painting was sprayed on the surface of the specimens in order to enhance this pattern and to make the DIC analysis easier. The ROI was concentrated around the axis of compression as sketched in Fig. 1. The pixel size was 51.6 \( \mu \)m.

![Figure 1](image1.png)

**Figure 1**: Sketch of the specimen region analysed with Correli-Q4.

3D microtomographic images of specimens were taken at different loading levels. A laboratory tomograph (NSI-X50) equipped with a 1536x1920 pixels detector was used. 1200
projections were taken within about 40 min for each scan. The spatial resolution was limited to only 20 \( \mu m \) due to the constraints related to the size of the specimen. Indeed, its diameter should be sufficiently larger than its thickness to fulfill Brazilian test requirements, and its thickness should be sufficiently larger than the maximum size of the sand particles (about 2mm) to fulfill continuum hypothesis for a mechanical study. For tomographic analysis only the small samples were used (0.9 cm thickness, 4 cm diameter).

3 RESULTS AND DISCUSSION

3.1 Mechanical behavior

The evolution of the compressive force versus time (or instantaneous displacement since the velocity is constant) for different RPP contents is represented in Fig. 2a. Without RPP the mortar displays almost an elastic-brittle behavior. Addition of RPP leads to both qualitative and quantitative changes of the mechanical behavior. Even for a very small RPP content (2%) the material becomes elasto-plastic. Increasing polymer proportion does not lead to significantly higher plasticity, but the ultimate rupture strain is substantially increased.

![Figure 2](image)

**Figure 2**: (a) Evolution of the compressive force versus displacement for different dosage rates of RPP. (b) Evolution of the peak force versus RPP content.

Fig. 2b represents the peak force versus RPP dosage rate. The error bars indicate the reproducibility of the measurements among 3 different samples. The evolution of the splitting strength versus polymer content is non-monotonic. The peak force first decreases, passes through a minimum and then increases. Such behavior suggests that we are dealing with two opposing (regarding splitting resistance) effects when increasing polymer content. Addition of RPP may indeed lead to two phenomena that may have opposing consequences on the mechanical resistance. On one hand, addition of RPP leads to an increase of air content (see Fig. 3), and on the other hand, this will strengthen the polymer film increasing tensile resistance of the organo-mineral co-matrix. As it is shown in Fig. 3 (through density measurements) increase of air content due to the polymer is significant only at low dosage. This supports the previous argument regarding the evolution of the peak force versus RPP content. An additional argument should be a possible transfer of compressive resistance (that may decrease) to tensile resistance (that increases) when increasing RPP content.
3.2 Mechanical behavior associated with digital image correlation (DIC) analysis

Fig. 4 represents the 2D displacement field as determined using the DIC technique around the compressional axis in the case of low polymer content (2%), 6 s before (Fig. 4a) and 6 s after (Fig. 4b) force drop. V and U are the local displacements respectively along and perpendicular to the compressional axis. Before the peak force the displacement field (U, V) is almost homogeneous along the tensile direction. Along the compressional axis the compressive component V slightly decreases downward, while the tensile one (U) increases. In any case the tensile displacement is an order of magnitude smaller than the compressive one. One can conclude that the mechanical properties of our material do not fulfill the appropriate conditions of a Brazilian test.

The residual, which quantifies the deviation from gray level conservation, is also represented (Fig. 4). Before the peak force the gray level of the residual is two orders of magnitude smaller than the gray level corresponding to the true displacements. Nucleation and growth of a crack will lead to violation of gray level conservation and then an increase of the residual. The latter can be then used to highlight the fracture zone. Although the variations are quite small, one can already distinguish an increase of the residual in the central zone before the peak force, foreshadowing appearance of a crack. The input of the residual is clearer after the force peak (Fig. 4b) when the crack is larger than the resolution of the technique. The fracture can also be perceived in the U displacement field, which displays a discontinuity corresponding to the location of the fracture. Yet the residual is more appropriate in characterizing quantitatively the cracks, including its early initiation, its growth and propagation.

Fig. 5 represents the displacement field and the corresponding residual in the case of 8% RPP content. Only the DIC results beyond the peak force are reported here. A detectable crack was observed only 24s after the ultimate splitting force (Fig 5a). Fig 5b represents the DIC images 48s after the peak force. At high polymer content the cracks become discontinuous and their propagation rate slows down. Indeed the total duration of a fracture spanning the whole ROI is 6s for the 2%RPP mortar while it is more than 48s in the case of 8% RPP content.
3.4 Optical DIC versus microtomography imaging

In this case the samples were submitted to discontinuous increasing loadings in order to analyze their microstructure ex-situ with microtomography for various loading levels. At the same time DIC was used to characterize the deformation field on the specimen surface. Fig. 6 illustrates the loading trajectories in the case of samples with 0% and 6% RPP.

Figure 6: Loading cycles to which the specimens were submitted for ex-situ tomographic analysis. 0% (a) and 6% (b) RPP.
Fig. 7 shows tomographic slices at different positions along the specimen axis in the case of RPP-free mortar and for the last cycle. It can be noticed that the main fracture is quite heterogeneous along the disc axis. The high heterogeneous propagation of the fracture along the disc axis can be better seen on tomographic projections along this axis (Fig. 8).

![Tomographic slices parallel to the specimen base at different locations along its axis, respectively 4mm, 6mm and 8mm.](image1)

**Figure 7:** Tomographic slices parallel to the specimen base at different locations along its axis, respectively 4mm, 6mm and 8mm.

![Tomographic slices perpendicular to the specimen base at different positions relative to the load axis.](image2)

**Figure 8:** Tomographic slices perpendicular to the specimen base at different positions relative to the load axis.

![Zoom in around the main fracture zone at different loading levels.](image3)

**Figure 9:** Zoom in around the main fracture zone at different loading levels.

Fig. 9 represents a zoom in of the central region of the sample for different loading levels. This set of images highlights how the fracture propagates along the weakest path consisting of consecutive large pores and bypassing the sand particles. At small scale the fracture pattern
should depend on the local properties of the material and should then require in any case 3D imaging analysis.

The detailed investigation of the 3D deformation field and facture pattern is still under investigation and will be reported in details elsewhere.

4 CONCLUSIONS

In this paper preliminary results are reported regarding the impact of redispersible polymer powders on the mechanical properties of render mortars. Optical and tomographic imaging were associated to consider the deformation field and damage state as a function of mechanical loading. Although more investigation is needed to draw any definitive conclusions, some qualitative results may be put forward:

- Latex-blended mortars are elastoplastic materials with high deformability
- The influence of RPP content on the mechanical resistance of the mortars under splitting load conditions is non-monotonic, suggesting that two competitive phenomena may come into play: (i) increase of air voids content which is expected to lead to a decrease of the mechanical resistance and (ii) increase of the connectivity and strength of the organo-mineral composite that should enhance the mechanical resistance, in particular the tensile component.
- It is shown that the DIC technique is a particularly valuable tool to analyze the heterogeneous strain field and superficial damage in the case of latex-mortars.
- Volumetric DIC technique using tomographic images is a promising tool to fully investigate the mechanical behavior of latex-mortars and its relation with the evolution of their microstructure. However in-situ mechanical testing in a tomographic set-up is required in order to take full advantage of this tool.

ACKNOWLEDGEMENTS

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REFERENCES

TRANSPORT PROPERTIES OF RECYCLED FINE AGGREGATE CONCRETES

Claudio J. Zega (1)(2), María E. Sosa (2) and Ángel A. Di Maio (1)(2)

(1) CONICET, Argentina – cj.zega@conicet.gov.ar
(2) Laboratorio de Entrenamiento Multidisciplinario para la Investigación Tecnológica (LEMIT), Argentina – ingmesosa@gmail.com; hormigones@lemit.gov.ar

Abstract
The use of crushed waste concrete as a substitute for natural aggregate for concrete production presents economic and environmental benefits. Therefore, several countries have recommendations for the use of recycled coarse aggregate in structural concrete, which limits the use of the fine fraction as a result of the high content of dust and foreign materials. Due to economic factors related to the shortage of natural sands suitable for concrete production, and to achieve an integral use of this type of waste, the use of recycled fine aggregate (RFA) has reached a remarkable international interest.

In this study, the durable behaviour of structural concretes with different strength levels (water/cement ratios 0.40, 0.50 and 0.60), and made with 20% and 40% of RFA was analyzed. Different transport properties such as water absorption, capillary suction and water penetration under pressure were evaluated. The performance of recycled concretes was compared with that of conventional concretes made with crushed fine aggregate in the same percentages. The results indicate that, despite the higher porosity of recycled concretes, their durable performance may be similar to that of conventional concrete.

1 INTRODUCTION
The use of Construction and Demolition Wastes (C&DWs) allows preserving the non-renewable natural resources, the landfills available and decreases the quantity of these wastes. The use of C&DWs as replacement of natural aggregate for the concrete production, particularly those obtained from crushed concrete, presents environmental and economic benefits, and is an important subject nowadays.

Several countries have recommendations for the use of recycled aggregate in structural concrete, but restrict the use of the recycled fine aggregate (RFA) due to its higher water absorption capacity and content of finer materials (lower than 75 μm) [1-4]. However, the use of RFA has become more important in recent years because of economic implications associated with unsuitable properties of natural sands for concrete production, and the need
for a complete use of such waste, since the fine fraction remains when the recycled coarse aggregates are used, which storage and control is more complex.

Different studies have been performed on the effect of RFA in the fresh state properties and resistant behaviour of mortar and concrete. Zega and Di Maio [5] inform that the use of RFA over 50% causes a significant decrease in the slump of the mixes, similar to that produced as consequence of the use of crushed fine aggregate (quarry sand). Khatib [6] concludes that the compressive strength of concretes made with 25% and 100% RFA decrease between 15% and 30% with respect to concretes made with natural aggregate; while Evangelista and de Brito [7] indicate that the compressive strength is not affected by the utilization of RFA up to 30%, although the splitting tensile strength and static modulus decrease as the recycled aggregate content increases. Later studies carried out by Zega and Di Maio [10] indicate that the compressive strength of concretes made with 20% and 30% RFA is similar to that of 100% natural fine aggregate concretes; while the static modulus of elasticity decreases with the increases in the content of RFA.

Although the most important changes in the behaviour of concretes made with RFA are related to higher drying shrinkage and less durability [8, 9], Zega and Di Maio [10] inform that the drying shrinkage of recycled concretes is similar to that of conventional one, and the sorptivity is 13% higher than that of conventional concrete.

The main objective of this study is to analysed the durable behaviour of recycled concretes with different compressive strength level and made with variable content of RFA (20% and 40%), respect to that obtained in concretes made with the same contents of crushed fine aggregate. Transport properties such as water absorption after immersion, sorptivity, capillary suction capacity and water penetration under pressure were evaluated. Complementary, the compressive strength of the studied concretes are presented.

2 EXPERIENCES

Considering the massive use of crushed fine aggregate in ready-mix concrete industry, as consequence of the shortage of natural river sand, the objective of this study is to analyse the possible modifications in some transport properties of concrete when recycled fine aggregate, obtained from crushed concrete, is used in instead of the crushed granitic fine aggregate.

2.1 Materials and mixes

Blended portland cement (type I (SM) ASTM or CEM II-M) and crushed granitic stone as coarse aggregate (6-20 mm) were used. For conventional concrete, the fine aggregate consisted of river sand and crushed granitic fine aggregate (CFA) (0-6 mm), while for recycled concrete, the river sand was used in conjunction with recycled fine aggregate (RFA) (0-6 mm). The RFA was obtained from waste concrete containing only crushed granitic stone as coarse aggregate; this fact allows eliminating the influence of the natural coarse aggregate in the properties of RFA, as was observed in previous studies [11]. Also, a plasticizer was used to avoid the workability lost as consequence of the use of crushed fine aggregates.

The properties evaluated in the river sand, crushed fine aggregate and recycled fine aggregate, such as fineness modulus, specific gravity (saturated and surface dry) (SGssd), 24h water absorption, content of material finer than 75 μm and weight loss by sulphate attack, are presented in Table 1.
Table 1. Properties of fine aggregates.

<table>
<thead>
<tr>
<th>Properties</th>
<th>River sand</th>
<th>CFA</th>
<th>RFA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fineness modulus</td>
<td>1.55</td>
<td>3.82</td>
<td>3.34</td>
</tr>
<tr>
<td>SGssd</td>
<td>2.65</td>
<td>2.77</td>
<td>2.35</td>
</tr>
<tr>
<td>Absorption (%)</td>
<td>0.4</td>
<td>0.6</td>
<td>8.5</td>
</tr>
<tr>
<td>Material finer than 75 µm (%)</td>
<td>1.2</td>
<td>2.8</td>
<td>4.0</td>
</tr>
<tr>
<td>Weight loss by Sulphate attack (%)</td>
<td>5.0</td>
<td>2.1</td>
<td>14.5</td>
</tr>
</tbody>
</table>

The fineness modulus of RFA is slightly lower than that of CFA, and both are higher than that of river sand, due to the crushing process by which they are obtained. Because of the mortar attached to the RFA particles, lower density, higher absorption, material finer than 75 µm and sulphate attack loss are observed with respect to the crushed fine aggregate. These characteristics of RFA were also reported in previous studies [5, 11].

Six mixes of recycled concrete with three different compressive strength levels (35, 27 and 21 MPa) and with two content of RFA (20% and 40% by volume) were produced. Also, six mixes of concrete of the same strength levels and contents of CFA were made as reference concrete. The mixes proportions for each concrete are presented in Table 2.

Table 2. Mixes proportions.

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Water</th>
<th>Cement</th>
<th>River sand</th>
<th>CFA</th>
<th>RFA</th>
<th>Coarse aggregate</th>
<th>Plasticizer</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-35C20</td>
<td>150</td>
<td>380</td>
<td>639</td>
<td>166</td>
<td>-</td>
<td>1102</td>
<td>1.14</td>
</tr>
<tr>
<td>C-35R20</td>
<td>150</td>
<td>380</td>
<td>639</td>
<td>-</td>
<td>141</td>
<td></td>
<td>1.14</td>
</tr>
<tr>
<td>C-35C40</td>
<td>150</td>
<td>380</td>
<td>480</td>
<td>332</td>
<td>-</td>
<td></td>
<td>1.14</td>
</tr>
<tr>
<td>C-35R40</td>
<td>150</td>
<td>380</td>
<td>480</td>
<td>-</td>
<td>282</td>
<td></td>
<td>1.14</td>
</tr>
<tr>
<td>C-27C20</td>
<td>150</td>
<td>304</td>
<td>692</td>
<td>180</td>
<td>-</td>
<td>1102</td>
<td>1.06</td>
</tr>
<tr>
<td>C-27R20</td>
<td>150</td>
<td>304</td>
<td>692</td>
<td>-</td>
<td>153</td>
<td></td>
<td>1.06</td>
</tr>
<tr>
<td>C-27C40</td>
<td>150</td>
<td>304</td>
<td>519</td>
<td>360</td>
<td>-</td>
<td></td>
<td>1.06</td>
</tr>
<tr>
<td>C-27R40</td>
<td>150</td>
<td>304</td>
<td>519</td>
<td>-</td>
<td>306</td>
<td></td>
<td>1.06</td>
</tr>
<tr>
<td>C-21C20</td>
<td>150</td>
<td>250</td>
<td>723</td>
<td>194</td>
<td>-</td>
<td>1102</td>
<td>1.00</td>
</tr>
<tr>
<td>C-21R20</td>
<td>150</td>
<td>250</td>
<td>723</td>
<td>-</td>
<td>165</td>
<td></td>
<td>1.00</td>
</tr>
<tr>
<td>C-21C40</td>
<td>150</td>
<td>250</td>
<td>546</td>
<td>379</td>
<td>-</td>
<td></td>
<td>1.00</td>
</tr>
<tr>
<td>C-21R40</td>
<td>150</td>
<td>250</td>
<td>546</td>
<td>-</td>
<td>322</td>
<td></td>
<td>1.00</td>
</tr>
</tbody>
</table>
The lower content of RFA was adopted based on previous studies where was concluded there was no differences in the mechanical and durable performance of recycled concretes with 20% and 30% RFA, but the slump decreased considerably for concrete with 30% RFA [10]. This last value (30%) is the maximum content of crushed fine aggregate indicated in the Argentinean Regulation for conventional concretes [12], which allows its increase if concrete meets the specified properties for the projected structure. Based on this last fact, 40% was adopted as the maximum content of crushed fine aggregate. The concretes are named according to the strength level, the type of fine aggregate used (C: crushed or R: recycled), and the fine aggregate content (20% or 40%).

With each concrete, three 150x300 mm cylindrical specimens for compressive strength, five 100x200 mm cylindrical specimens for capillary suction and water absorption, and three 150x200 mm cylindrical specimens for water penetration were cast. The specimens were kept in a fog room (temperature: 23ºC±2ºC; relative humidity > 95%) until the age of 28 days.

2.2 Testing methodology

As a reference parameter, the compressive strength was determined in each concrete at the age of 28 days, according to ASTM C39 [13].

The water absorption test is a quick way to assess the durable behaviour of concrete, and was performed according to ASTM C 642 [14].

The capillary absorption test is particularly sensitive to the size of the connected pores. Tests were carried out in cylindrical specimens of 100 mm diameter and 50 mm high, according to IRAM 1871 [15]. To avoid humidity lateral losses and ensure a uniaxial flow into the specimens, the lateral surfaces were sealed with a waterproof paint. The test technique consists of recording the mass increment by capillary suction up to constant weight when the specimens are placed with a face immersed in water (depth 3 mm). The capillary suction rate (sorptivity) and the maximum capacity of absorption (capillary suction capacity) are obtained as results.

The fluid movement through the saturated pore structure of concrete under pressure was evaluated through water penetration test. The determinations were carried out according to IRAM 1554 [16], equivalent to BS EN 12390-8, in 200 mm side cubes. The pressure cycle consists in 0.1 MPa for 48 hours, 0.3 MPa for 24 hours and 0.7 MPa for 24 hours. After that, the specimens are broken by splitting tensile and the humid profile is recorded to obtain the average and maximum depths of water penetration.

3 RESULTS AND DISCUSSION

The compressive strength of all concretes determined at the age of 28 days is presented in Figure 1, where each indicated value is the average of three tests. For each strength level, the compressive strength of concretes made with RFA is similar to that obtained in concretes made with CFA. The content of RFA, as well as that of CFA, no shows a significant influence in the compressive strength. Also, the three strength levels are clearly identified.
Figure 1. Compressive strength.

The results of water absorption after immersion obtained in conventional and recycled concretes are presented in Figure 2. Each indicated value was obtained as the average of five samples.

Figure 2. Water absorption.

As it is expected, the absorption of recycled and conventional concretes increases as the strength level decreases. On the other hand, for the different strength levels studied, the absorption of concretes with RFA is higher than that of reference concrete, and it slightly increases with the content of RFA, due to the higher absorption of this aggregate with respect to the CFA, as was indicated above in Table 1.

The sorptivity and capillary suction capacity obtained in concretes made with crushed and recycled fine aggregates are presented in Table 3, where each value corresponds to the average of five tests.
Table 3. Parameters of capillary absorption test.

<table>
<thead>
<tr>
<th>Concrete</th>
<th>Sorptivity (g/m^2/s^{1/2})</th>
<th>Capillary suction capacity (g/m^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-35C20</td>
<td>2.36</td>
<td>2245</td>
</tr>
<tr>
<td>C-35R20</td>
<td>2.23</td>
<td>1900</td>
</tr>
<tr>
<td>C-35C40</td>
<td>2.22</td>
<td>1995</td>
</tr>
<tr>
<td>C-35R40</td>
<td>2.14</td>
<td>1620</td>
</tr>
<tr>
<td>C-27C20</td>
<td>4.34</td>
<td>2920</td>
</tr>
<tr>
<td>C-27R20</td>
<td>3.60</td>
<td>3235</td>
</tr>
<tr>
<td>C-27C40</td>
<td>5.92</td>
<td>3210</td>
</tr>
<tr>
<td>C-27R40</td>
<td>3.56</td>
<td>3020</td>
</tr>
<tr>
<td>C-21C20</td>
<td>6.61</td>
<td>3520</td>
</tr>
<tr>
<td>C-21R20</td>
<td>4.98</td>
<td>3575</td>
</tr>
<tr>
<td>C-21C40</td>
<td>6.81</td>
<td>3590</td>
</tr>
<tr>
<td>C-21R40</td>
<td>6.00</td>
<td>3995</td>
</tr>
</tbody>
</table>

Sorptivity values increase as the strength level decreases, which is more important that the content of RFA used. The sorptivities determined in concretes C-35R20 and C-35R40 are slightly lower (4-6 %) than those obtained in concretes of the same strength level containing CFA. For concretes C-35, all sorptivity values are lower than the limit (4 g/m^2/s^{1/2}) indicated in the Argentinean Regulation [12] for concretes with durable requirement. For concretes C-27 and C-21, the sorptivity of recycled concretes is lower than that obtained in reference concrete. The better behaviour of recycled concrete can be attributed to the lower effective w/c ratio of this concrete, because of the higher absorption of RFA, and the better interface transition zone of RFA compared with CFA.

With respect to the capillary suction capacity, the values determined in concretes C-35R20 and C-35R40 are 15% and 19% lower than those obtained in concretes of the same strength level made with CFA. For the other strength levels, the capillary suction capacity obtained in recycled concretes is in the same range of magnitude that the registered in the reference concretes. In the particular case of concrete C-21R40, higher capillary suction capacity is noticed, because of the higher porosity of the new cement paste.

The depth of water penetration determined in recycled concretes, as well as that obtained in reference concretes, is presented in Figure 3. The indicated values were obtained as average of three tests. The dotted lines represent the limits indicated in the Argentinean Regulation for the average (30 mm) and maximum (50 mm) depths of water penetration under pressure.

For each strength level, the values of water penetration (average and maximum) in recycled concretes are similar to those determined in reference concretes. For the lower compressive strength level, it can be observed that the average water penetration of concrete
C-21R40 is slightly lower than that of reference concrete, fact that can be attributed to the lower effective w/c ratio of recycled concrete.

It must be noticed that although the water absorption after immersion of recycled concrete was higher than that of the corresponding conventional concrete, the sorptivity and depth of water penetration showed similar performance in both types of concretes. The mentioned above agrees with the indicated in the literature for conventional concrete [17].

4 CONCLUSIONS

From the different transport properties determined in concrete mixes of three different compressive strength level (water/cement ratio 0.40, 0.50 y 0.60) and made with 20% and 40% of recycled fine aggregate (RFA), which were compared with those obtained in reference concretes made with the same content of crushed granitic fine aggregate (CFA), can be concluded that:

- Effectively, the compressive strength obtained in recycled concretes is similar to that of reference concretes, without influence of the content of RFA used.
- The water absorption of recycled concrete is higher than that of reference concrete, for the different w/c ratio, and increases with the content of RFA. This behaviour is attributed to the higher absorption of RFA, respect to CFA, due to mortar from old concrete.
- The sorptivity and capillary suction capacity of recycled concretes increase with the w/c ratio in the same way that for reference concretes. Some influence of the content of RFA was noticed for concretes of the lower strength level, because of the higher porosity of the new cement paste.
- The water penetration test presents similar water depth for recycled and reference concretes, without influence of RFA content, because of the lower w/c ratio and better interface transition zone of RFA.
- For the studied mixes, i.e. strength levels evaluated and contents of RFA used, the achieved results allow concluding that concretes made with recycled fine aggregate may
present a durable performance similar to that of conventional concretes with the same compressive strength level.

REFERENCES

Abstract

Concrete and steel are currently the most widely used construction materials. The main component of concrete is cement, mainly constituted by clinker. The manufacture of clinker consumes non-renewable resources, both energy and raw materials, and releases CO\(_2\). The partial replacement of clinker by supplementary materials minimizes these disadvantages and usually provides suitable mechanical and durable properties of cement based materials. Therefore, the use of supplementary materials as addition to cement is common and utterly accepted by worldwide standards.

Coal fly ash is one of these allowed additions to cement. Fly ash is, like coal bottom ash, a residue from coal-pulverized power plants. While fly ash is usually reused as a high-value by-product in cement manufacture, bottom ash is merely reused as a low-cost filling civil construction, at best scenario. However, some studies have recently reported good pozzolanic and mechanical properties of cements with coal bottom ash as addition.

In this paper, the evolution of the hydration process of cement based materials manufacture with coal bottom ash and fly ash was studied. Several parameters were monitored to this purpose: weight change, electrical resistivity, ultrasonic pulse and porosity. All of them were used to characterize the evolution of the pore structure in cement based materials, an important characteristic that has a fundamental influence on its durable properties. The results showed interesting similarities between cements based materials with bottom ash and fly ash at a same percentage replacement.

Keywords: Coal bottom ash, coal fly ash, cement hydration, resistivity, ultrasonic pulse, porosity
INTRODUCTION

Concrete and steel are currently the most widely used construction materials. The main component of concrete is cement, mainly constituted by clinker. The manufacture of clinker consumes non-renewable resources, both energy and raw materials, and releases CO₂. The partial replacement of clinker by supplementary cementitious materials (SCM) minimizes these disadvantages and usually provides suitable mechanical and durable properties of cement based materials. Therefore, the use of SCM as addition to cement is common and utterly accepted by worldwide standards. Coal fly ash is one of these allow additions to cement. Fly ash is, like coal bottom ash, a residue from coal-pulverized power plants. While fly ash is usually reused as a high-value by-product in cement manufacture, bottom ash is merely reused as a low-cost filling civil construction, at best scenario [1]. However, some studies have recently reported good pozzolanic, mechanical and environmental properties of cements with coal bottom ash as addition [2-8].

The hydration process is an important parameter that influences the final microstructure of the material and hence in its mechanical and durability properties. During the hydration process, calcium silicates (CSH) and calcium hydroxide (portlandite) are formed. The CSH constitutes the cement matrix while portlandite take part both in the cement matrix and the the porous structure. Furthermore, the reaction of aluminates and ferrite aluminates with gypsum produces ettringite, which could react with aluminates to form mono-sulpha-aluminates if there is not enough sulphur. These mono-sulpha-aluminates appear both in the cement matrix than in porous structure. The use of SCM implies more complex hydration process [9]. Partial replacement of cement by FA produces the same hydration products [10] but the kinetic reaction associated is modified [11]. The study of the different hydration mechanisms of cement and each SCM, as well as the interaction between them must be studied to understand the complete hydration process and, therefore, the behaviour of the final material.

Hydration process is highly exothermic, especially at first stages. Therefore, it has being usually studied by semi adiabatic calorimetry. This technique identify, measuring the amount of heat released, different process involved and the hydration mechanism associated. However, this technique presents some important shortages [11]. For example, there is not a direct correlation with the hydration degree and it cannot estimate the physical properties of the final materials. Chemically, cement hydration implies ion dissolution and the formation of new chemical compounds. Physically, this process transforms the microstructure reducing its porosity. Attending to both chemical and physical factors, some authors are using electrical and ultrasonic pulse measures to study the hydration process of cements. Furthermore, Menendez et al. are using electric measures combining with traditional instrumental techniques [12-15].

In this paper, the evolution of the hydration process of cement based materials manufacture with coal bottom ash and fly ash was studied. Some parameters were monitored to this purpose: weight change, electric resistivity and ultrasonic pulse. Porosity at 28 days of hydration was also measure.
2 EXPERIMENTAL

This work involved the study of mortar’s hydration made of different fly and bottom ashes through the evolutions of its weight, electric resistivity and ultrasonic pulse speed, as well as its porosity at 28 days of curing. First, coal ashes and cement mixtures are described. Then, the techniques used to study the hydration process are explained.

2.1 Characterization of coal ashes

In this study, two Spanish thermal power plants burning different quality of coal were selected. The power plants were named “α” and “β”. Power plant α burns coal from South Africa (90%) and Colombia (10%) while power plant β uses an indigenous Spanish coal whose fly ash contain more organic matter.

FA and BA were supplied from both power plants. Consequently, four types of ashes were examined. The designation of the different ashes is presented in Table 1.

<table>
<thead>
<tr>
<th>Type of ash</th>
<th>Power plant</th>
<th>Designation of ashes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fly Ash</td>
<td>α</td>
<td>FAα</td>
</tr>
<tr>
<td></td>
<td>β</td>
<td>FAβ</td>
</tr>
<tr>
<td>Bottom Ash</td>
<td>α</td>
<td>BAα</td>
</tr>
<tr>
<td></td>
<td>β</td>
<td>BAβ</td>
</tr>
</tbody>
</table>

The main components of ashes were determined by X-ray fluorescence using a Bruker, S8. The Insoluble Residue (RI) was analysed through the sodium carbonate method and the content of Lost on Ignition (LOI) was determined burning the ashes during 1h (950°C); both methods are described in the standard EN – 196 -2:2006. Each determination is the average of three values. The results, expressed as oxides, are listed in Table 2.

<table>
<thead>
<tr>
<th>Type of ash</th>
<th>LOI (%)</th>
<th>SiO₂ (%)</th>
<th>Al₂O₃ (%)</th>
<th>Fe₂O₃ (%)</th>
<th>CaO (%)</th>
<th>Minor elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>FAα</td>
<td>3,63</td>
<td>46,84</td>
<td>26,66</td>
<td>4,72</td>
<td>5,55</td>
<td>5,284</td>
</tr>
<tr>
<td>FAβ</td>
<td>9,14</td>
<td>39,57</td>
<td>24,81</td>
<td>3,43</td>
<td>5,18</td>
<td>4,633</td>
</tr>
<tr>
<td>BAα</td>
<td>1,85</td>
<td>48,12</td>
<td>25,55</td>
<td>5,86</td>
<td>7,07</td>
<td>4,946</td>
</tr>
<tr>
<td>BAβ</td>
<td>1,55</td>
<td>48,34</td>
<td>19,46</td>
<td>8,96</td>
<td>6,44</td>
<td>5,888</td>
</tr>
</tbody>
</table>

In general, the FA and the BA from each power plant have similar compositions, except to calcium which is higher in BAs, as well iron, magnesium and chromium content. Since higher LOI contents might produce durability related problems, it is limited up to 9% in European standard UNE – EN 197 – 1. The relative high LOI content in FAB sample may be explained due to the lower quality coal burned in β power plant. However, the BAβ sample has a similar LOI content that the BAα sample and in any case lower than both FA samples examined.
The microstructure of coals ashes was analysed by scanning electron microscopy (SEM). While FAs are shaped by spherically particles and lower than 30 µm of diameter, BAs are irregular and higher (Figure 1). This difference can be explained by the steep of the process where they are generated [1].

![Figure 1. Micrographs SEM of coal ashes](image)

2.2 Testing methods to study the hydration process

Cement mixtures made of different proportions of coal ashes as cement replacement were designed. Percentages of 0%, 10% and 25% were selected corresponding to cements CEM I, CEM II/A and CEM II/B, respectively. Replacements of 35% were dismissed due to the lower mechanical properties observed in utterly studies [6]. There were cements made of additions 100% of FA and 100% of BA. As 80% of total ashes produced in a power plants corresponds to fly ashes and 20% to bottom ash, two mixtures of additions consisting of 80% of FA plus 20% of BA were also selected (Table 3). All the cement mixtures were prepared with cement type CEM I 42.5.

<table>
<thead>
<tr>
<th>OPC</th>
<th>10FAα</th>
<th>10FBαα</th>
<th>10BAα</th>
<th>10FAβ</th>
<th>10FBαβ</th>
<th>25FAα</th>
<th>25FBαα</th>
<th>25BAα</th>
<th>25FAβ</th>
<th>25FBαβ</th>
<th>25BAβ</th>
</tr>
</thead>
<tbody>
<tr>
<td>CEM I (%)</td>
<td>100</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td>90</td>
<td>75</td>
<td>75</td>
<td>75</td>
<td>75</td>
<td>75</td>
<td>75</td>
</tr>
<tr>
<td>FAα (%)</td>
<td>-</td>
<td>10</td>
<td>8</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>BAα (%)</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>10</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>5</td>
<td>25</td>
<td>-</td>
</tr>
<tr>
<td>FAβ (%)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
<td>8</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>25</td>
</tr>
<tr>
<td>BAβ (%)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>2</td>
<td>10</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>25</td>
</tr>
</tbody>
</table>
Three prismatic specimens of 4x4x16 cm were manufactured with each cement mixture. They were prepared according to the standard UNE-EN 196-1, curing in an oisture chamber (20.0 ±1.0°C y 90% HR) during 24 h. After unmoulding, specimens were placed at these same conditions until testing.

Weight change, electric resistance and ultrasonic pulse were measure at 1, 7, 14, 28 and 90 days of curing. Electric resistivity was measure by the four-point (Wenner) method described in UNE-EN 83988-2. Ultrasonic pulse was measure by the direct transmission method described in UNE 83-308-86. On the other hand, porosity was measure by mercury intrusion at 28 days of curing.

3 RESULTS

The results obtained, both the evolution of hydration parameters and their value at 28 days of curing, are described in this section.

3.1 Evolution of mass, electrical resistivity and ultrasonic pulse speed of mortars

The evolution through the time of weight change, the electrical resistivity and the ultrasonic pulse speed of the mortars studied are detailed below.

![Figure 2. Evolution of weight change of mortars](image)

![Figure 3. Evolution of electrical resistivity of mortars.](image)
A quite linear increase of weight of mortars during time was observed (Figure 2). Regarding to electrical resistivity and ultrasonic pulse speed, there was observed an increase during time, especially at the first days of hydration. As hydration happens, mortar structure densifies, increasing the resistivity of the material to the electric pulse. However, since porosity decreased, ultrasonic pulse reduces the bouncing and increases it speed. The increase of electric resistivity and ultrasonic pulse speed tend to stabilize, especially in the case of ultrasonic pulse speed.

### 3.2 Porosity, electric resistivity and ultrasonic pulse at 28 days of hydration

At 28 days of hydration, porosity, electric resistivity and ultrasonic pulse speed was compared between different mortars formulations. It was observed that the amount of cement replacement was more significant than the use of FA, BA or a mix of them.

Porosity of mortars with 10% of cement replacement was similar to reference mortar CEM I. However, porosity of mortars with 25% of cement replacement was higher, whatever the kind of as or its originating power plant. Regarding to non-destructive techniques, it was observed a decreased of electric resistivity and ultrasonic pulse speed as cement replacement increased. However, at a same cement replacement percentage the differences were virtually negligible.
4 DISCUSSION

4.1 Correlation between the results of non-destructive techniques

Since the tendency observed in both non-destructive techniques were so similar, the correlation between these techniques was analysed. Figure 6 shows the results of electric resistivity and ultrasonic pulse speed of mortars obtained through time. A correlation between these parameters can be observed: as one of them increases the other one also increases, but not at the same speed. Therefore, both techniques can be used interchangeably. Since both techniques are non-destructive and can be used in in-situ structures, it can be used one of them and then predicts the possible results with the other technique.

![Figure 6. Correlation between the results obtained in non-destructive techniques](image)

4.2 Correlation between hydration parameters

There was a tendency between the results obtained in non-destructive techniques and the weight change in cement mortars: as weight change increased, electrical resistivity and ultrasonic pulse speed tended to increased (Figure 7). However, dispersion was observed in these results, especially at first stages of hydration. This is due to both electrical resistivity and ultrasonic pulse speed are not only related to weight change; they are also related to other parameters, which should be studied in more detail.

![Figure 7. Correlation between hydration parameters](image)
5 CONCLUSIONS

- There is an increase of weight, electrical resistivity and ultrasonic pulse speed of cement mortars with different additions during time related to the densification of their structure which means their hydration.
- These non-destructive techniques are useful to compare the hydration process of cement mortars with different additions, both qualitatively and quantitatively,
- There is a correlation between the both non-destructive techniques, which could be useful to evaluate structures built.
- In general, as cement replacement percentage increase, porosity, electric resistivity and ultrasonic pulse speed decrease, at 28 days of hydration. However, at a same cement replacement percentage, non significative differences between mortars with FA, BA and a mix of them were observed in these parameters.

REFERENCES

MEASURING WATER SORPTION ISOTHERM OF MORTAR CONTAINING CHLORIDE

A. Taher, T. Arends, A.J.J. van der Zanden and H.J.H. Brouwers

Department of the Built Environment, Eindhoven University of Technology P.O. Box 513, 5600 MB Eindhoven, the Netherlands – a.taher@tue.nl; t.arends@student.tue.nl; ajjvdzanden@kpnmail.nl; jos.brouwers@tue.nl

Keywords: Mortar, Chloride, Water sorption isotherm, Hygroscopic method.

Abstract
The water sorption isotherm of materials is of great importance to predict their durability. Many different methods are used in the literature to measure this water sorption isotherm. Chloride present in concrete elements/structures e.g. in marine exposure, can change the water sorption of the material. In this paper, the water sorption isotherm of a standard mortar with and without chloride is measured with a hygroscopic method. The chloride penetration into the sample is obtained by the vacuum saturation method. Subsequently the sample is placed in a glass vessel and dried. By injecting a known amount of water into the vessel, the water evaporates and is partly adsorbed by the sample until an equilibrium state is reached. With the known amount of the injected water into the vessel, the humidity inside the vessel and the volume of the vessel, one point of the adsorption isotherm is determined. For measuring the next point, more water is injected into the vessel. More points are measured in the same manner until the sample is saturated and with this the adsorption isotherm is completed. Afterwards, the measurement of the desorption isotherm starts with blowing dry air with a known flow, humidity and duration into the vessel. After reaching equilibrium, the first point of the desorption curve is measured. The next point is measured by blowing more dry air into the vessel. This continues until the sample is completely dry and with it the desorption isotherm is also completed.

1 INTRODUCTION

A sorption isotherm describes the relation between the moisture content in the examined material and the relative humidity of the surrounding environment at equilibrium and constant temperature. Since the sorption isotherm depends upon the history of drying and wetting of a sample, the sorption isotherm contains two curves, one for adsorption and one for desorption. The purpose of this paper is to examine the effect of chloride in mortar on the sorption of water in the material, since the presence of chloride affects the sorption of water. The sorption of water in materials has received a lot of attention in the literature: several methods for obtaining a sorption isotherm are known. The simplest method is by weighing the sample: this can be done manually by a quartz spring [1] or by a balance [2]. Another method for acquiring the sorption isotherm of a material is by means of the manometric measuring
technique, as described by Broom [3]. Van der Zanden and Goossens [4] describe a method for obtaining the sorption isotherm of water in paint films. This method will be applied in the present study on reference mortar sample and mortar sample containing chloride.

2 EXPERIMENT

2.1 Test set-up

A glass vessel with a glass cover is used to measure the sorption isotherm, as shown in Figure 1. The use of rubber rings can disturb the measurements in this set-up, because rubber can absorb or release water [5], and therefore, a Teflon foil ring is placed between the glass vessel and the cover to prevent leakage in and out of the system. The glass vessel contains a humidity sensor and a temperature sensor, which are connected to a data logger. A glass plate is placed in the vessel to capture the added drops of water. The glass vessel is also provided with an inlet and outlet for blowing dry air into the vessel and allowing the air escape from the vessel. An additional opening in the glass cover is used to inject water to the vessel. Furthermore, the glass vessel is placed in a room with a constant temperature and humidity.

![Figure 1: Cross section of the test set-up to measure sorption isotherm of mortar with a hygroscopic method.](image)

2.2 Method

The measurement of the adsorption isotherm starts with placing the mortar sample inside the glass vessel and blowing dry air through the vessel for a certain time in order to dry the sample. The air inlet and outlet are then closed. At this point, it is assumed that the sample is completely dry. This assumption is not entirely true, since it is seen from initial equilibrium that the sample contained a small amount of water. However, this amount is very small, as it causes only little change in the relative humidity at the start.

After initial equilibrium a known amount of water is injected on the glass plate in the vessel using a device with which water can be dosed accurately. The injection opening in the glass cover is closed also with a Teflon plug. The injected water evaporates, which increases the humidity inside the vessel. Consequently, the water content of the sample increases as the sample adsorbs water from the air. After certain time, equilibrium is reached in the vessel. Figure 2 gives a characteristic chart of the humidity in the vessel as a function of time. The chart can be divided into three phases. The first phase is an increase of relative humidity as a result of evaporation of the injected water. The second phase is a decrease of relative
humidity resulting from the adsorption of water by the sample. The final phase is the equilibrium state between the relative humidity inside the vessel and the water in the sample.

![Figure 2: A characteristic chart of the relative humidity in the vessel as a function of time at 21°C (adsorption isotherm).](image)

The total amount of water in the vessel is equal to the known cumulative amount of injected water. The amount of water in the air in the vessel is obtained by calculation from the measurement of equilibrium relative humidity (phase 3). The rest of the total amount of water is adsorbed by the sample. By calculating the amount of water in the sample at certain relative humidity, a single point of the adsorption isotherm is determined.

The measurement of the desorption isotherm starts after reaching an equilibrium relative humidity of approximately 90% in the vessel. One point of the desorption isotherm is calculated by blowing dry air with a known flow and known duration into the vessel. Also in this case, after certain time, equilibrium is reached in the vessel. Figure 3 gives a characteristic chart of the humidity in the vessel as a function of time. This chart can also be divided into three phases; the first phase is the decrease of relative humidity which results from blowing dry air in the vessel. The second phase is the increase of relative humidity resulting from the release of water by the sample. The final phase is the equilibrium state between the water in the air of the vessel and the water in the sample.
3 SAMPLE PREPARATION

In this experiment, mortar samples are examined with a water cement ratio of 0.5 and cement type CEM I 42.5 N. The composition and properties of the mortar are shown in Table 1. The samples are cast in PVC tubes with a diameter of 100 mm. After one day, the mortar is demoulded and cured in water for additional 27 days. Samples of 10 mm thickness are cut from the mortar cylinder and provided with a chloride mass percentage of 2.0%.

Table 1: Composition and properties of the samples

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mortar</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Volume [m³]</td>
<td>Mass [kg/m³]</td>
<td></td>
</tr>
<tr>
<td>CEM I 42,5N</td>
<td>159.47</td>
<td>502.18</td>
<td></td>
</tr>
<tr>
<td>Sand</td>
<td>568.51</td>
<td>1506.54</td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>251.09</td>
<td>251.09</td>
<td></td>
</tr>
<tr>
<td>w/c</td>
<td></td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Porosity [%]</td>
<td></td>
<td>17.0</td>
<td></td>
</tr>
</tbody>
</table>

Chloride is added to the sample by means of the vacuum saturation method, in which four samples are first placed in a vessel and air is removed from the samples by applying vacuum pressure of about 20 mbar for 3 hours. After the air is removed, a sodium chloride solution is poured into the vessel under vacuum. Subsequently, after 45 hours in the solution, the samples are removed from the vessel. One sample is used to determine the sorption isotherm and the other three are used to examine the chloride concentration by titration. Figure 4 shows the chloride concentration profiles measured by titration. It can be seen that the total chloride concentration is approximately 1.0% throughout the samples.
4 RESULTS

For computing the adsorption isotherm, the water content in the sample is determined for various relative humidities inside the vessel as:

\[ m_{\text{sample}} = m_{\text{total}} - m_{\text{air}}, \]  

(1)

where \( m_{\text{total}} \) is the total amount of added water, \( m_{\text{sample}} \) the water content in the sample and \( m_{\text{air}} \) the water content of the air inside the vessel.

With the water content in the air, \( C \), and the water content in saturated air, \( C_{\text{sat}} \), computed with Antione’s law and the ideal gas law as:

\[ C_{\text{sat}} = \frac{M}{R \cdot T} \cdot e^{\frac{23.196}{-46.13 + T}}, \]  

(2)

where \( M \) is the molar mass of water, \( R \) the gas constant and \( T \) the temperature in Kelvin. The relative humidity, \( H \), can be re expressed as:

\[ H = \frac{C}{C_{\text{sat}}} \cdot 100. \]  

(3)

With Eqs. (2) and (3) the amount of water in the air becomes:

\[ m_{\text{air}} = \frac{H \cdot V \cdot M}{R \cdot T \cdot 100} \cdot e^{\frac{23.196}{-46.13 + T}}, \]  

(4)

where \( V \) (m\(^3\)) is the volume of the vessel.

By dividing the water content in the sample by the volume of the sample, the water content, \( w_i \), in the sample at a certain relative humidity is obtained.
To obtain the desorption curve, dry air with known relative humidity is blown in the vessel and air containing water leaves the vessel during a certain period of time. Points on the curve are determined by calculating the amount of water loss during this period. The mass of water leaving the vessel during the drying period is calculated using a mass balance as:

\[ m_{\text{out}} = \int_0^\tau \varphi_v (C_{\text{out}} - C_{\text{in}}) dt, \]  

(5)

where \( \varphi_v \) is the volumetric flow of the dry air and \( C \) the water content in the air, with the subscript indicating whether the water content corresponds to the air entering or leaving the vessel.

With Eq. (3), Eq. (5), \( m_{\text{out}} \) can be rewritten as:

\[ m_{\text{out}} = \frac{C_{\text{sat}} \varphi_v}{100} \int_0^\tau (H_{\text{out}} - H_{\text{in}}) dt. \]  

(6)

It is assumed that the dry air entering the vessel mixes well with the air in the vessel before leaving, meaning that the relative humidity of the air leaving the vessel is equal to the relative humidity of the air in the vessel, which is measured every minute. With this assumption and the relative humidity of the air at equilibrium, the water content in the sample can be determined, which follows from a mass balance.

The adsorption isotherm for a sample with no chlorides and a sample with chlorides are determined as shown in Figure 5.

![Figure 5: The adsorption isotherm of a mortar sample with and without chloride.](image-url)
The equilibrium at the start is reached at 5% relative humidity, which is defined as zero in the curves. The adsorption curve of the sample without chloride consists of 16 points, which are obtained by adding 0.1 ml water to the vessel 15 times and 0.2 ml water once. The adsorption curve of the sample with chloride consists of 19 points, which are obtained by adding 0.1 ml water to the vessel 5 times, 0.2 ml 3 times and 0.3 ml 12 times. Equilibrium state is reached approximately in 3 days after adding water.

From the results obtained thus far, it is however seen that the sorption of the sample containing chloride is significantly larger than the sorption of a sample containing no chloride. The difference between the curves can be explained by the presence of chloride, which seems that it absorbs a large amount of water. In fact, the chloride in mortar changes the type of adsorption isotherm from Linear to BET type II adsorption isotherm.

5 CONCLUSIONS AND DISCUSSION

The durability of a concrete structure is largely governed by deterioration of the steel rebars as a consequence of corrosion, initiated by the presence of chloride. Also, crystallization of salt present in the pores can damage the material. It is therefore very important to accurately describe water and salt transport in cementitious materials. An important water transport property is the sorption of a porous material. The hygral state of a porous material as a consequence of sorption is described with the sorption isotherm. In this work, the adsorption isotherm of mortar is measured with a hygroscopic method. With this technique, the adsorption isotherm of any porous construction material can be measured. The amount of measured points can easily be adjusted, by increasing or decreasing the amount of added water. For example, by adding 0.05 ml of water instead of 0.1 ml, which is used in this work, twice as much measured points can be obtained. With this technique the desorption isotherm can be measured also.

The linear sorption isotherm for mortar containing no chloride, which is measured in this work, is in good agreement with those found in the literature. Tada and Watanabe [6] have predicted a linear sorption isotherm for a mortar containing no chloride. This was in agreement with experimental data, although the experiment showed larger sorption. Estimated data were, however, in better agreement with experimental data obtained by Daian [7]. In this study, a linear adsorption isotherm is found for a mortar sample containing no chloride.

The effect of the presence of chloride in mortar on the sorption of a sample is also regarded in this study. Mortar samples with chloride content are prepared with vacuum saturation. Sodium chloride solution is used to provide the samples with chloride. A homogeneously distributed chloride concentration throughout the sample is obtained by first drying the sample completely before saturating with sodium chloride solution. From the results obtained thus far it is seen that the sorption of the sample containing chloride is significantly larger than the sorption of the sample containing no chloride. Besides larger sorption as a consequence of the presence of sodium chloride, the sorption curve also transitions from a linear curve to a BET Type II curve [8-9]. Since the BET-model is based on the multilayer adsorption on a solid, the presence of chloride can influence the adsorption mechanism. Some chloride can be bound to the surface of the pore walls, which in turn can alter the way and quantity at which water is adsorbed on the surface on
the pore walls. The larger sorption of a sample containing sodium chloride is also seen in the study of Koniorczyk and Wojciechowski [10], where a higher salt concentration led to more water adsorption in mortar. The larger sorption of a sandstone sample containing salt is also found by Franzen and Mirwald [11]. The increased sorption at high relative humidities can also be explained with the results from the study by Rijniers [12]. In this study, it is found that NaCl tends to adsorb a large amount of water at a high relative humidity.

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BACTERIAL CONCRETE: A BIOLOGICAL APPROACH TO REDUCED PERMEABILITY

Alan Richardson (1), Kathryn Coventry (2) and Andrew Scott (3)

(1) Northumbria University, Faculty of Engineering and Environment, Newcastle upon Tyne, UK alan.richardson@northumbria.ac.uk

(2) Northumbria University, Faculty of Engineering and Environment, Newcastle upon Tyne, UK kathryn.coventry@northumbria.ac.uk

(3) Northumbria University, Faculty of Engineering and Environment, Newcastle upon Tyne, UK a.scott@northumbria.ac.uk

Abstract
This paper investigated the effects of Bacillus megaterium (class 1 WHO) ground borne bacteria when introduced into a concrete mix. The range of investigation examined, penetration of water under pressure (permeability) and compressive strength compared against a plain control sample.

Three test batches of concrete were cast; plain concrete, concrete with live bacteria including nutrient broth with urea (NBU) and concrete where the mixing water was replaced with nutrient broth with urea. The purpose of the different batches was to establish the effects of Bacillus megaterium and nutrient broth with urea upon cured concrete. The results were compared to a plain concrete control sample and conclusions were drawn.

The findings established that, bacterial concrete, when compared to plain concrete, provided increased density and lower permeability, however the bacterial concrete compressive strength, when compared to plain concrete, was impaired due to the introduction of NBU into the concrete mix. This was due to the calcium chloride component of the NBU which can act as an accelerator but has the effect of lowering the final compressive strength.

This work contributes to and continues research into applying biological processes to develop more sustainable construction materials.

Key words: Concrete, Bacillus megaterium bacteria, compressive strength, water penetration (permeability), microbial induced calcite precipitation, durability.

Contact author: Alan Richardson
1.0 Introduction

Concrete is the most widely used construction material [1]. Concrete’s inherent properties, of adequate compressive strength, relative durability, workability, fire resistance and the ability to manufacture complex shapes, makes concrete a primary technological solution for designers [2].

However, its use is not without consequence. Due to its physical characteristics it holds a propensity for liquid ingression, thereby reducing its service life and impacting upon the global sustainable development agenda by increasing life cycle costs [1].

In many instances concrete is an inherently porous material, as such it is prone to the ingression of water and other deleterious substances. The ingress of water through the hardened cement paste, via the internal pore/capillary structure, limits the durability of concrete. This paper aims to establish if bacteria, when added to the concrete mix will commence microbial induced calcite precipitation (bio mineralisation), thereby filling the capillaries and pore structure to primarily create a less porous material and secondly to see if the calcite formation creates additional cementation. There was a concern that the Ph level of the concrete may inhibit the performance of the bacteria when used within the cement matrix.

The ingestion of water, acts as a vector for other deleterious substances. Due to the porous nature of concrete is a major contributory factor in the reduction of concrete service life [3,4,5 and 6]. The transfer of water within concrete occurs via three differing mechanisms which are collectively referred to as permeability [4 and 7]. The three differing water transfer mechanisms are displayed in Table 1.

<table>
<thead>
<tr>
<th>Method of Water Transfer</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Absorption</td>
<td>Porous capillary suction driven flow.</td>
</tr>
<tr>
<td>Diffusion</td>
<td>Porous concentration driven flow.</td>
</tr>
<tr>
<td>Permeation</td>
<td>Porous pressure driven flow.</td>
</tr>
</tbody>
</table>

Table 1 – Water transport mechanisms - Source: [4]

The introduction of bacteria which precipitate calcite, thus effectively blocking the pore structure of concrete, promises an ecologic solution to liquid ingress reduction by microbial induced calcite precipitation (MICP) [8]. When MICP occurs in a calcium rich local environment, generation of dense binding calcite cement is feasible [9]. The MICP created must be within voids, capillaries and pores, thus reducing the water ingress pathway and providing a reduction in porosity/permeability.

1.1 Microbial Induced Calcite Precipitation / Bio-mineralisation

Bio-mineralisation is the phenomena of bacteria in nutrient rich environs creating a micro-environment that permits the precipitation of mineral materials [10]. MICP is a form of bio-mineralisation specifically referring to the production of the calcite which is a form of calcium carbonate.

The process is initiated by *Bacillus megaterium* type bacteria which intracellularly hydrolyse urea for nutrients thus beginning the process towards calcite precipitation. The overall chemical reaction is expressed as:

\[
\text{CO(NH}_2\text{)}_2 + \text{2H}_2\text{O} + \text{Ca}^2_- \rightarrow \text{2NH}_4^- + \text{CaCO}_3
\]

[Eq 1] - Microbial Induced Calcite Precipitation

Source: [9] and [14].
However the process is complex with a number of intermediate chemical reactions. The full process of chemical reactions can be found in Figure 1.

The process ends with the production of calcite which precipitates out of solution into a solid state as well as 2 molecules of ammonium and water [11]. The progress of the water is not detailed within the overall reaction, however within the body of research it is shown within the chemical reaction from the bicarbonate reaction stage.

In order to progress the overall reaction four key elements are required and these are calcite precipitating bacteria, urea, water, and calcium. Concrete typically only holds one chemically available element – water. Therefore a source of bacteria, urea and calcium are required within the NBU.

2.0 Concrete composition
2.1 Concrete mix design
Concrete was batched to the mix design as shown in Table 2 using a rotary drum mixer. The cubes were manufactured to BS 1881 : Part 108 : 1983 using 150 mm moulds. Consistency of the six batches was monitored using a slump test as defined within BS EN 12350 – 2: 2000.
With the exception of the plain concrete, the water was replaced with a bacterial suspension or NBU at the same w/c ratio as the plain control mix.

2.2 Bacteria
Bacteria, sometimes referred to as microbiota are single celled simple organisms [9] and are the most diverse and numerous branch of life found on the planet [12]. Physically they are generally invisible to the naked eye, measuring in the range of 0.5 – 5.0 μm [9]. Without a nuclear membrane selected to hold genetic information they are classified as prokaryotic cells. This study focuses upon morphological Bacillus alkaliphile bacteria that have an ability to commence calcite precipitation (bio-mineralisation) within their natural environment.

*Bacillus megaterium* was selected for this study following work by Achal et al [13] which discovered that *Bacillus megaterium* produced the highest output of calcite when compared to other classifications of bacteria, such as *Bacillus Simplex* and *Bacillus Subtilus*. *Sporoscarcina pasteurii* (previously known as *Bacillus pasteurii*) has predominately been used within the concrete matrix, however in one test *Bacillus megaterium* was used [10]. This work will extend the knowledge of *Bacillus megaterium* use.

The *Bacillus megaterium* cultures were stored at -80°C prior to being activated. One colony of *Bacillus megaterium* was inoculated into 100ml sterile nutrient broth containing 20g/l of urea and 20g/l of yeast extract (NBU). The suspension was incubated at 37˚ Celsius overnight and grown to its maximum concentration, which equated to $10^5$ cfu/ml. The culture was then added to 900ml sterile NBU with 50 grams of calcium chloride added within 1 litre of NBU and stored in sealed Duran containers. The process was repeated 10 times to produce 10 litres of bacterial suspension. This provided a food source for the bacteria once it was added to the concrete.

3.0 Test methodology
Twelve 150mm concrete cubes were batched for each concrete type. Nine were used for compressive strength tests and the remaining three for permeability tests. The cubes were stored at 20°C in a curing tank overnight (14 hours) and left to air dry cure during the day (10 hours). This process replicated wet and dry conditions that could be expected in the natural environment.

The test protocol consisted of testing three concrete sample cubes for each concrete batch, at time intervals of 7, 14 & 28 days for compressive strength and at 28 days for porosity, as shown in Tables 3 and 4. The compressive test to determine the compressive strength of the concrete was in accordance with BS EN 12390-3:2002.
There are two population samples of three cubes (total of six) to be compared. The degrees of freedom used, equates to total number of samples minus 2, which is minus one degree of freedom per population sample. This allows for a t-value that is (+/-) 2.776, to determine significance.

A null hypothesis T test was undertaken to evaluate the significance of the compressive strength and permeability test findings. Equation 2 was used to calculate the t values for the significance tests.

\[
t - \text{Value} = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\frac{\sigma_1^2}{N_1} + \frac{\sigma_2^2}{N_2}}}
\]  

[2]

A 95% probability has been used with a p-value of 0.05. The null hypotheses for the test was assumed that when test results were compared and statistically interrogated for batches A&B, B&C and A&C there would be no statistically significant event observed. If the null hypothesis was to be rejected and the alternative hypothesis accepted, a p value would be found to be <0.05, which would indicate a statistically significant difference.

3.1 Permeability test

To determine the relative permeability of the concrete samples, water under 5 bar pressure was applied to the lower surface of the cube sample and the following sequence was followed. The samples were loaded into the porosity testing machine (Porisimeter) as displayed in Figure 2 and secured in place using the screw thread clamp, the water was turned on, and then the valve was opened to increase water pressure to 5 bar at the bottom face of the sample.
The samples were left in porisimeter for 72 hours. After 72 hours the samples were loaded into compressive testing machine with timber length placed at bottom of the sample. The action of applying a load to the cube over a strip of timber, sheared the sample in half when a compressive force was applied. The depth of water penetration was measured with steel rule, to the fully saturated section of cube and the results were recorded.

4.0 Results
4.1 Compressive Strength Test

The results from the compressive strength tests are displayed in Tables 4 - 6. A progressive strength development was observed in all of the batches at all ages. The degree of scatter in the results is reasonably small for concrete and this is confirmed when viewing the standard deviation column.

<table>
<thead>
<tr>
<th>Time Interval</th>
<th>Sample</th>
<th>Max Load (kN)</th>
<th>Cross Sectional Area (Amm²)</th>
<th>Compressive Strength (N/mm²)</th>
<th>Standard Deviation (σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7 Days</td>
<td>A1</td>
<td>366.1</td>
<td>22500</td>
<td>16.27</td>
<td>0.89</td>
</tr>
<tr>
<td></td>
<td>A2</td>
<td>406.2</td>
<td>22500</td>
<td>18.05</td>
<td></td>
</tr>
<tr>
<td></td>
<td>A3</td>
<td>387.3</td>
<td>22500</td>
<td>17.21</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td><strong>17.18</strong></td>
<td></td>
</tr>
<tr>
<td>14 Days</td>
<td>A4</td>
<td>470.8</td>
<td>22500</td>
<td>20.92</td>
<td>0.82</td>
</tr>
<tr>
<td></td>
<td>A5</td>
<td>444.8</td>
<td>22500</td>
<td>19.77</td>
<td></td>
</tr>
<tr>
<td></td>
<td>A6</td>
<td>480.4</td>
<td>22500</td>
<td>21.35</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td><strong>20.68</strong></td>
<td></td>
</tr>
<tr>
<td>28 Days</td>
<td>A7</td>
<td>616.9</td>
<td>22500</td>
<td>27.42</td>
<td>2.68</td>
</tr>
<tr>
<td></td>
<td>A8</td>
<td>633.8</td>
<td>22500</td>
<td>28.17</td>
<td></td>
</tr>
<tr>
<td></td>
<td>A9</td>
<td>728.9</td>
<td>22500</td>
<td>32.40</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td><strong>29.33</strong></td>
<td></td>
</tr>
</tbody>
</table>

Table 4 – Plain – Batch A
## Batch B - Bacterial Suspension

<table>
<thead>
<tr>
<th>Time Interval</th>
<th>Sample</th>
<th>Max Load (kN)</th>
<th>Cross Sectional Area (mm²)</th>
<th>Compressive Strength N/mm² (f = F/A)</th>
<th>Standard Deviation (σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7 Days</td>
<td>B1</td>
<td>378.5</td>
<td>22500</td>
<td>16.82</td>
<td>1.27</td>
</tr>
<tr>
<td></td>
<td>B2</td>
<td>348.1</td>
<td>22500</td>
<td>15.47</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B3</td>
<td>321.2</td>
<td>22500</td>
<td>14.28</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td>15.52</td>
<td></td>
</tr>
<tr>
<td>14 Days</td>
<td>B4</td>
<td>536.8</td>
<td>22500</td>
<td>23.86</td>
<td>2.09</td>
</tr>
<tr>
<td></td>
<td>B5</td>
<td>453.8</td>
<td>22500</td>
<td>20.17</td>
<td></td>
</tr>
<tr>
<td></td>
<td>A6</td>
<td>456.8</td>
<td>22500</td>
<td>20.30</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td>21.44</td>
<td></td>
</tr>
<tr>
<td>28 Days</td>
<td>B7</td>
<td>550.1</td>
<td>22500</td>
<td>24.45</td>
<td>1.63</td>
</tr>
<tr>
<td></td>
<td>B8</td>
<td>565.3</td>
<td>22500</td>
<td>25.12</td>
<td></td>
</tr>
<tr>
<td></td>
<td>B9</td>
<td>495.4</td>
<td>22500</td>
<td>22.02</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td>23.86</td>
<td></td>
</tr>
</tbody>
</table>

Table 5 – Batch B

## Batch C - NBU Concrete

<table>
<thead>
<tr>
<th>Time Interval</th>
<th>Sample</th>
<th>Max Load (kN)</th>
<th>Cross Sectional Area (mm²)</th>
<th>Compressive Strength N/mm² (f = F/A)</th>
<th>Standard Deviation (σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7 Days</td>
<td>C1</td>
<td>299.9</td>
<td>22500</td>
<td>13.33</td>
<td>0.33</td>
</tr>
<tr>
<td></td>
<td>C2</td>
<td>288.3</td>
<td>22500</td>
<td>12.81</td>
<td></td>
</tr>
<tr>
<td></td>
<td>C3</td>
<td>302.3</td>
<td>22500</td>
<td>13.44</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td>13.19</td>
<td></td>
</tr>
<tr>
<td>14 Days</td>
<td>C4</td>
<td>336.8</td>
<td>22500</td>
<td>14.97</td>
<td>0.47</td>
</tr>
<tr>
<td></td>
<td>C5</td>
<td>353.8</td>
<td>22500</td>
<td>15.72</td>
<td></td>
</tr>
<tr>
<td></td>
<td>C6</td>
<td>356</td>
<td>22500</td>
<td>15.82</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td>15.51</td>
<td></td>
</tr>
<tr>
<td>28 Days</td>
<td>C7</td>
<td>379.3</td>
<td>22500</td>
<td>16.86</td>
<td>1.68</td>
</tr>
<tr>
<td></td>
<td>C8</td>
<td>402.2</td>
<td>22500</td>
<td>17.88</td>
<td></td>
</tr>
<tr>
<td></td>
<td>C9</td>
<td>453.3</td>
<td>22500</td>
<td>20.15</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td>18.29</td>
<td></td>
</tr>
</tbody>
</table>

Table 6 – Batch C
The average values from the compressive strength results are displayed in Figure 3.

When the t-Test formula was applied to the data, a statistically significant difference was noticed between the bacterial concrete and the NBU concrete at 7, 14 and 28 days. The overall effect of the NBU when introduced into the concrete matrix was to impede strength development, for a short time the probable production of calcium carbonate within the hardened concrete paste allowed bacterial concrete to overcome this effect. The strength development was impeded by the inclusion of calcium chloride. Calcium chloride is a rapid hardener but can reduce the overall 28 day compressive strength. This is corroborated by Wang et al [14] who found that calcium nitrate can accelerate cement hydration, but they also found that yeast extract delays hydration and decreases the hydration degree if the dosage is higher than 0.85%. However the bacteria has a finite life span within the concrete matrix [1] and the results obtained are suggestive that Bacillus megaterium expires at some point between 14 – 28 days when in the hardened cement paste. This would account for the small gain in compressive strength from the 14 to 28 day time intervals for bacterial concrete, whilst also explaining the increase in performance against NBU samples as the calcium carbonate deposits would remain in place post expiry of the active bacteria. A further test was carried out at 56 days but no significant change was observed due to the action of the bacteria, therefore the results have been omitted from the results section.

4.2 Permeability test
Following 72 hours of water permeability pressure testing at 5 bar, the cubes were split into two halves and the water penetration measured as shown in Table 7.
Table 7 – Saturated water penetration

<table>
<thead>
<tr>
<th>Penetration of Water</th>
<th>Depth of Penetration (mm)</th>
<th>Standard Deviation(σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Batch A (Water)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>A10</td>
<td>145.00</td>
<td></td>
</tr>
<tr>
<td>A11</td>
<td>120.00</td>
<td></td>
</tr>
<tr>
<td>A12</td>
<td>135.00</td>
<td>12.58</td>
</tr>
<tr>
<td>Average</td>
<td>133.33</td>
<td></td>
</tr>
<tr>
<td>Batch B (Bacterial Concrete)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B10</td>
<td>75.00</td>
<td>7.64</td>
</tr>
<tr>
<td>B11</td>
<td>65.00</td>
<td></td>
</tr>
<tr>
<td>B12</td>
<td>80.00</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>73.33</td>
<td></td>
</tr>
<tr>
<td>Batch C (NBU Water Replacement)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C10</td>
<td>104.00</td>
<td>5.03</td>
</tr>
<tr>
<td>C11</td>
<td>100.00</td>
<td></td>
</tr>
<tr>
<td>C12</td>
<td>110.00</td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td>104.67</td>
<td></td>
</tr>
</tbody>
</table>

Bacterial concrete (B) displayed a 55% reduction in water permeability when compared to plain concrete - batch (A). The mean average water penetration for the bacterial and nutrient broth concrete samples was less than that of the plain samples (A). When the t-Test formula was applied to the data, comparing Batch B and C individually against Batch A; a statistically significant result with a p-value of 0.0021 was demonstrated between Batch A and B and a p-value of 0.0215 was demonstrated between Batch A and C. Therefore the experiment undertaken rejects the null hypothesis in both cases, and the alternative hypothesis was accepted for bacterial and nutrient broth concrete.

Bacterial concrete was the most resistant concrete to the penetration of water as displayed in Table 7 and confirmed by the statistical analysis. The results are suggestive of calcium carbonate deposits being formed in the macropores of the hardened concrete paste, thus impeding the progress of water through the concrete mass. The permeability results of this test concur with the findings of Achel et al [13].

Somewhat surprisingly NBU concrete out performed plain samples, this may be in part to the noticed viscosity of the NBU liquid providing a “thicker” fluid within the matrix thereby preventing water progression, it may also be the effect of the calcium chloride in the NBU.. The effects of the NBU may contribute to the performance of the bacterial concrete samples in addition to the bio mineralisation.
5.0 Conclusion
In concluding this research, it is shown that *Bacillus megaterium* through microbial induced calcite precipitation and the choice of suspension medium is the causal factor in the production of a less porous concrete, which aids the materials durability and therefore improves sustainability. However this benefit is offset due to the reduction in compressive strength through the use of NBU as the medium for the bacterial suspension.

This work contributes to and continues research into applying biological processes to develop more sustainable construction materials. Further work is required using calcium lactate as a nutrient broth and this may lessen the compressive strength reduction observed using urea based nutrient broth, however if calcium lactate is used, a different bacterial strain will have to be used, as calcium lactate requires a different metabolic pathway when compared to *Bacillus megaterium*.

6.0 References
DEVELOPMENT AND STANDARDIZATION OF RAPID METHODS FOR ASSESSING THE FLUID PENETRATION RESISTANCE OF CONCRETE

R. Doug Hooton (1), Gita Charmchi (1) and Ester Karkar (2)

(1) Dept. of Civil Engineering, University of Toronto, Toronto - hooton@civ.utoronto.ca; gita.charmchi@mail.utoronto.ca
(2) Lafarge Canada Inc., Dundas, Canada. ester.karkar@lafarge.com

Abstract
The common element in designing durable concrete in aggressive exposures is reducing the rate of ingress by fluids, such as chloride and sulfate solutions. While some tests for measuring chloride diffusion, permeability and rates of absorption have been standardized, due to the extended time required for their completion, they are often only suitable for prequalification of concrete mixtures. For acceptance during construction, rapid index tests are needed. While the ASTM C1202 resistance to chloride penetration test has served this purpose for many years, it can be replaced by even more rapid and less costly test methods such as bulk resistivity and surface resistivity. This contribution will discuss these issues, review alternative test methods, and discuss adoption of test methods in standard specifications.

1. INTRODUCTION

1.1 General
The durability of concrete is greatly influenced by its resistance to fluid penetration. Fluids and aggressive ions can penetrate by a combination of diffusion, permeability, capillary suction and by wick action, with the relative importance of each mechanism determined by boundary conditions. While test methods for measuring each of these mechanisms have been developed, they are often time consuming and expensive to perform. Therefore, limits on specific standard penetration resistance properties (eg. Bulk chloride diffusion in Nordtest NT Build 443 (ASTM C1556), water sorptivity in ASTM C1585), or vapor transport (ASTM E96) may be useful for prequalification of concrete mixtures, and limits on fluid penetration resistance (although not necessarily using standard test methods) have been specified in many high-profile, long-service life structures (such as the Confederation Bridge in Canada, the Tsing Ma Bridge in Hong Kong, and the Oresund Bridge between Denmark and Sweden, to name a few).
However, for quality control / quality assurance during construction, standardized rapid index tests that relate to fluid penetration resistance are needed. As well, due to inherent test variability, appropriate, statistically-based test limits for such tests need to be established.

2. RAPID TEST METHODS

There are limitations to practical use of rigorous permeability and diffusion-based testing beyond prequalification of concrete mixtures, and there is an important role for rapid index tests for chloride penetrability for quality assurance during construction. As a result, for acceptance and quality assurance purposes, rapid permeability-index tests, such as the ASTM C1202 “coulomb” test, the Nordtest NT Build 492 (also AASHTO TP-64) Rapid Chloride Migration Test, surface resistivity test (Wenner probe, AASHTO TP095) or a bulk resistivity test are needed. Due to its relative simplicity and its initial standardization as AASHTO T277 more than 30 years ago, the ASTM C1202 test has become widely used both in North America and globally as a permeability index test.

2.1 ASTM C1202 Test

This widely used, but often criticized test method evolved from a FHWA study by Whiting [1], as AASHTO T277, also ASTM C1202. In this test, a saturated concrete disk is sandwiched between two cells (one filled with 0.3M NaOH and the other with 0.5M NaCl solution). A 60V DC potential is applied to electrodes in the two cells. The current is monitored and integrated over a 6-hour period to obtain the charge passed in coulombs. The current flow is related to the volume and connectivity of the saturated capillary pore system. Fundamentally, it is simply a conductivity or inverse-resistivity test and in spite of it often being referred to as the Rapid Chloride Permeability Test, it does not measure the “permeability” or “chloride diffusion” of concrete. Numerous researchers have pointed this out [2,3,4]. Because current flow is also affected by pore fluid conductivity, admixtures that result in large changes in conductivity influence the test results (eg. calcium nitrate corrosion inhibitors). This interference is noted in the standard.

Others have criticized it since high current flows will result in heating of the sample and solutions during the 6 h test, raising the measured conductivity. However, others have found that C1202 test data, if corrected for temperature, do relate to salt ponding data [5,6].

In 2004, the Canadian CSA A23.1 concrete standard [7] adopted ASTM C1202 rapid chloride penetration index limits for prequalification of concrete mixtures to meet (a) C-1 exposure conditions (concrete exposed to freezing in a saturated condition with de-icer salts: 35 MPa, air-entrained, 0.40 w/cm max.) of 1500 coulombs at 56 days, and (b) C-XL exposure (similar to C-1 but where extended service life is required, 50MPa, air-entrained, 0.40 w/cm max.) of 1000 coulombs at 56 days. The CSA A23.1 standard also includes a maximum single value limit as well as average value coulomb limit when this test is used for acceptance during construction (eg. For the C-1 exposure, a single test value is allowed to be up to 1750 coulombs as long as the average value remains below 1500 coulombs at 56 days). This allows for test variability and is similar to the statistical approach typically used for strength acceptance.

As an interim measure to try and shorten the test procedure, in 2012, a bulk electrical conductivity test, using the same sample preparation and equipment as C1202, was standardized as ASTM C1760 [8]. In this test, both sides of the concrete specimen are
exposed to 1N NaCl solution, 60V DC is applied, and the current after 1 minute is measured and the bulk conductivity is calculated. This test can also be used to calculate the bulk resistivity. In addition to being faster, other advantages of this test relative to C1202 are that with the short period of voltage application there is no temperature rise, and highway and test agencies can use the same equipment they already have. However, C1760 is considered to be an interim method since bulk resistivity test methods can be conducted more simply and without the time-consuming sample preparation.

2.2 Rapid Migration Test

This non-steady-state migration test, developed by Tang and Nilsson [9] and adopted as NT Build 492 involves measurement of the depth of chloride ingress under an applied DC potential (the voltage and time of test are determined from an initial current measurement). The depth of penetration is measured by splitting the specimens open after test and spraying with AgNO₃ solution. Above about 0.07N chloride concentration, the AgNO₃ will convert to AgCl₂ and turn white in color. The non-chloride affected areas turn dark brown after a few minutes exposure to light. A non-steady-state diffusion coefficient can then be calculated from the depth of penetration together with knowledge of the magnitude and period of the applied potential. The test is of similar rapidity to the ASTM C1202 procedure and its advantages are that (a) results are not influenced by pore fluid conductivity, (b) the depth of chloride penetration is measured directly, and (c) that the calculated diffusion coefficient can be used in service-life prediction models. However, the validity of the equation used to determine the diffusion coefficient has been questioned [10,11].

2.3 Wenner Probe Surface Resistivity Test

In this test, four equally-spaced electrical probes are pressed against a moist concrete surface. The two outer probes apply a low-frequency alternating current while the voltage drop between the two inner probes is measured. Use of Wenner probes has recently become popular with several US state highway agencies due to their simplicity and commercial availability. Standards include Florida DOT FM 5-578, 2004 [12] and AASHTO TP095 [13]. ASTM is also balloting a similar procedure. However, as detailed by Gowers and Millard, 1999 [14], either the spacing between the probes needs to be adjusted based on the size, geometry, thickness of the concrete specimen being tested, the location of the probe array from the edges of concrete specimen, and the thickness of concrete cover in reinforced concrete elements, or corrections have to be applied to the values obtained. Recorded resistivity values will vary if an inappropriate probe spacing is used and also if the concrete surface is not fully saturated. The Wenner probe can be modified using clip-on wires to each of the four probes attached to two stainless steel plate electrodes to obtain bulk resistivity Spragg et al 2011 [15]. It has been found that the Wenner probe results are influenced by the solutions used to saturate the concrete specimen since the electrical conductivity of the near-surface pore fluid can be altered substantially [16].

2.4 Bulk Resistivity Test

In bulk resistivity tests, high impedance AC voltage is applied over the whole cross-section of a saturated specimen using plate electrodes and conductive gel, and the current passing through the pore system is measured. DC power is not used due to polarization resistance that builds up at the electrodes, however, Monfore [17] overcame this by cycling between two DC
voltages. The advantage of this method is that the entire cross-section is tested and the test can be applied to saturated concrete test cylinders or cubes prior to testing for compressive strength. Portable test devices are now commercially available.

Three different devices were used to measure bulk resistivity: The Resipod from Proceq, the RCON from Giatec, and the Merlin from Germann Instruments. Each of these devices use different wave forms and AC current frequencies, so to allow use of all these devices, an equipment-specific standard test cannot be developed. Therefore, the draft ASTM test method currently being developed will not specify the details of the equipment, but each device will have to meet accuracy requirements when calibrated using standard resistances. Results obtained from each device can differ by as much as 4% as shown in Table 1 for two verification cell settings.

The verification cell used for the data in Table 1 is intended for calibration of the Germann Merlin device. The 100 mm diameter by 200 mm long cylindrical cell was used since it can be fitted into the cells of each of the commercial test devices to be evaluated as if it was a concrete sample.

Table 1: Comparison of bulk resistivity results from two verification standards using three commercial devices and either water or conductive gel as coupling fluid

<table>
<thead>
<tr>
<th>Verification Cell Resistivity ohm-m</th>
<th>RCON (water) ohm-m</th>
<th>RCON (gel) ohm-m</th>
<th>Resipod (water) ohm-m</th>
<th>Resipod (gel) ohm-m</th>
<th>Merlin (water) ohm-m</th>
<th>Merlin (gel) ohm-m</th>
</tr>
</thead>
<tbody>
<tr>
<td>Setting 3</td>
<td>39.27</td>
<td>39.6</td>
<td>39.9</td>
<td>41</td>
<td>40.33</td>
<td>39.8</td>
</tr>
<tr>
<td>Setting 4</td>
<td>392.7</td>
<td>396.4</td>
<td>393.0</td>
<td>396</td>
<td>393.3</td>
<td>392.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>% Difference from Verification Value</th>
<th>Setting 3</th>
<th>Setting 4</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>+0.90</td>
<td>+1.63</td>
</tr>
<tr>
<td></td>
<td>+0.95</td>
<td>+0.09</td>
</tr>
<tr>
<td></td>
<td>+4.41</td>
<td>+0.84</td>
</tr>
<tr>
<td></td>
<td>+4.41</td>
<td>+0.20</td>
</tr>
<tr>
<td></td>
<td>+2.71</td>
<td>+0.16</td>
</tr>
<tr>
<td></td>
<td>+1.35</td>
<td>+0.02</td>
</tr>
</tbody>
</table>

It has eight resistor settings, where the setting ‘0’ corresponds to no resistivity and the setting ‘7’ corresponds to an open channel. The remaining settings, ‘1’ through ‘6’ cover a range of 0.3927 to 39,270 ohm-m, increasing by a power of 10 with each consecutive setting. The settings included in Table 1 are those that were within the range to be picked up by all three commercially available bulk resistivity meters: The RCON, Resipod and Merlin. The percentage difference values in Table 1 compare the average resistivity readings (the average of between 2 to 5 replicate measurements) from each device with respect to the standard reading as indicated by each verification cell setting.

3. TESTING AND RESULTS

Nine concrete mixes (mix proportions shown in Table 2) were tested after 35 and 56 days of moist curing at 23°C. In Canada, GU portland cement is equivalent to ASTM Type I or EN CEM I, MS is moderate sulfate resistant portland cement, HS is highly sulfate resistant...
Portland cement, and GUb-30F is a blended cement with 30% Class F fly ash. Ground granulated slag was then used in some cases to replace 30, 35 or 50% of these cements. Efficiency factors were not used in any w/cm calculations. The ASTM C1202 coulomb test, NT492 non-steady state diffusion test, and bulk resistivity test data (but only using the Merlin device from Germann Instruments) are provided in Table 3.

Table 2: Concrete mix proportions

<table>
<thead>
<tr>
<th>Mix Proportions (kg/m³)</th>
<th>w/cm</th>
<th>0.5</th>
<th>0.5</th>
<th>0.45</th>
<th>0.4</th>
<th>0.5</th>
<th>0.45</th>
<th>0.4</th>
<th>0.5</th>
<th>0.4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cement Type</td>
<td></td>
<td>GU</td>
<td>GU</td>
<td>GU</td>
<td>GU</td>
<td>GUb-30F</td>
<td>HS</td>
<td>HS</td>
<td>MS</td>
<td>MS</td>
</tr>
<tr>
<td>% Slag</td>
<td></td>
<td>35%</td>
<td>0%</td>
<td>50%</td>
<td>50%</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
<td>50%</td>
</tr>
<tr>
<td>Cement</td>
<td></td>
<td>215</td>
<td>330</td>
<td>183</td>
<td>206</td>
<td>330</td>
<td>367</td>
<td>413</td>
<td>330</td>
<td>206</td>
</tr>
<tr>
<td>Slag</td>
<td></td>
<td>116</td>
<td>0</td>
<td>183</td>
<td>206</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>206</td>
</tr>
<tr>
<td>Coarse Aggregate</td>
<td></td>
<td>963</td>
<td>963</td>
<td>963</td>
<td>963</td>
<td>963</td>
<td>963</td>
<td>963</td>
<td>963</td>
<td>963</td>
</tr>
<tr>
<td>Fine Aggregate</td>
<td></td>
<td>931</td>
<td>939</td>
<td>894</td>
<td>854</td>
<td>939</td>
<td>908</td>
<td>869</td>
<td>939</td>
<td>854</td>
</tr>
<tr>
<td>Water</td>
<td></td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165</td>
<td>165</td>
</tr>
</tbody>
</table>

Table 3: ASTM C1202, Nordtest NT492, and bulk Merlin resistivity values

<table>
<thead>
<tr>
<th>Mixture</th>
<th>ASTM C1202</th>
<th>Nordtest</th>
<th>NT492</th>
<th>Bulk Resistivity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Migration</td>
<td>10-12 (m²/s)</td>
<td></td>
<td>(ohm-m)</td>
</tr>
<tr>
<td></td>
<td>35 Days</td>
<td>56 Days</td>
<td>35 Days</td>
<td>56 Days</td>
</tr>
<tr>
<td>0.5/GU/35S</td>
<td>994</td>
<td>877</td>
<td>5.7</td>
<td>4.9</td>
</tr>
<tr>
<td>0.5/GU</td>
<td>2456</td>
<td>2205</td>
<td>13.6</td>
<td>11.1</td>
</tr>
<tr>
<td>0.45/GU/50S</td>
<td>726</td>
<td>581</td>
<td>4.8</td>
<td>3.7</td>
</tr>
<tr>
<td>0.4/GU/50S</td>
<td>784</td>
<td>632</td>
<td>3.2</td>
<td>3</td>
</tr>
<tr>
<td>0.5/GUb-30F</td>
<td>975</td>
<td>607</td>
<td>7.2</td>
<td>4.6</td>
</tr>
<tr>
<td>0.4/HS</td>
<td>3360</td>
<td>3029</td>
<td>16.8</td>
<td>14.1</td>
</tr>
<tr>
<td>0.4/HS</td>
<td>3137</td>
<td>2680</td>
<td>12.7</td>
<td>10.8</td>
</tr>
<tr>
<td>0.5/MS</td>
<td>3178</td>
<td>3012</td>
<td>18.7</td>
<td>21.2</td>
</tr>
<tr>
<td>0.4/MS/50S</td>
<td>808</td>
<td>775</td>
<td>6.2</td>
<td>3.1</td>
</tr>
</tbody>
</table>

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The relationship between the initial bulk electrical resistivity and total ASTM C1202 charge values is presented in Figure 1, and with the NT492 migration coefficient in Figure 2.

![Figure 1: ASTM C1202 charge passed vs bulk resistivity values.](image1)

![Figure 2: Relation between NT492 migration coefficient and bulk resistivity values.](image2)

### 4. CONCLUSIONS

As shown in the figures good relationships can be established between results from ASTM C1202, bulk resistivity, and migration coefficients obtained using Nordtest NT492. While the ASTM C1202 test has served its purpose as a relatively rapid index test for fluid penetration resistance, it could be reliably replaced by very rapid and inexpensive bulk resistivity tests. ASTM has moved in this direction with the adoption of C1760 in 2012. As well, standardization of both surface (Wenner probe) and bulk resistivity test methods is in progress. Although not as rapid, the Nordtest NT492 non-steady state migration results are not influenced by pore fluid conductivity, and diffusion values can be calculated from the
chloride penetration values and used in service life prediction models. However, resistivity values can also be converted into diffusion values using, the Nernst-Einstein equation.

ACKNOWLEDGEMENTS

The support of the Cement Association of Canada and NSERC are gratefully acknowledged. Use of resistivity equipment by Germann Instruments Inc., Proceq Inc. and Giatec Scientific Inc. is also greatly appreciated.

REFERENCES

ASSESSMENT OF CHLORIDE INGRESS IN BLENDED CEMENT WITH COMBINED ADDITION OF CALCINED CLAYS AND LIMESTONE

Antoni(1), M, Henocq(1), P, Kazemi-Kamyab, H(1), Favier, A(1), Martirena, F(2), Scrivener, K(1)

(1) EPFL-STI-IMX LMC, Station12, CH-1015 Lausanne, Switzerland,
(2) CIDEM-UCLV, Universidad Las Villas, Santa Clara, Cuba

Abstract:
Substitution with pozzolanic materials such as calcined clays, even of average purity, is known to improve concrete properties. However above a certain threshold commonly accepted to be of about 30% of substitution, these materials reduce the mechanical properties, especially at early age. In this contribution the combined addition of calcined clays and limestone is investigated. Previous results have indeed shown that there is a synergetic effect with formation of carbo-aluminates phases in this ternary system and that 45% of substitution by 30% of metakaolin and 15% of limestone powder gives better mechanical properties at 7 and 28 days than the 100% OPC reference. The porosity is indeed globally refined.

In this contribution, the resistance to chloride ingress has been studied by the STADIUM migration test on mortars. It has been set up for one pure metakaolin and for natural calcined clay, in binary blend with pure metakaolin alone and in ternary blend with combined addition of 30% calcined clays and 15% limestone.

Both blended cement, with and without further substitution of cement by limestone powder have demonstrated to significantly reduce the diffusion coefficient of chloride ions. It is attributed to the refinement and increased tortuosity of the pore structure.

1 INTRODUCTION

Substitution with pozzolanic materials such as calcined clays, even of average purity, is known to improve concrete properties [1, 2]. In a previous study, Antoni et al [3] have shown that 45% of cement substitution by 30% of metakaolin and 15% of limestone powder gave better mechanical properties at 7 and 28 days than the 100% OPC reference. In this case, the porosity was globally refined although the total porous volume was slightly increasing. It could be demonstrated that these very promising mechanical properties were related to the synergetic effect in this ternary system by formation of carbo-AFm phases [4-6], besides the commonly accepted pozzolanic reaction. In this contribution, the resistance to chloride ingress of such a blend has been studied by the migration test derived from ASTM C1202 -97 standard test procedure [7, 8]. It has been set up for one pure metakaolin and for natural calcined clay, in binary blend with calcined clay alone and in ternary blend with combined addition of 30% calcined clays and 15% limestone.

The properties of these blended mixtures were compared to an ordinary Portland cement mix.

2 EXPERIMENTAL PROCEDURE

Mortars cylinders, 10 cm diameter, with a water-to-binder ratio of 0.5 were casted. For reference, mortars made with a CEM I 32.5 cement (labelled here “OPC”) containing around 3% CaCO3 as well as a blend containing 30% of a pure metakaolin were prepared. A ternary blend with combined addition of a pure metakaolin (30% of the cement mass content) and limestone (15% of the cement mass content, adjusted for cement original limestone content) was used. These mixtures had their composition adjusted with gypsum for adequate sulfatation, following the procedure described elsewhere [3] and labelled MK30 and MKB45 respectively. The chemical composition of materials used in this study has been
characterized by X-ray Fluorescence (XRF) (APC Solutions, Denges, Switzerland) and are given in Table 1. The samples were cut at the desired thickness, prepared and tested after 28 days of curing in an atmosphere with over 90% RH.

Table 1. Chemical composition of raw materials

<table>
<thead>
<tr>
<th>Oxides</th>
<th>OPC</th>
<th>MK30</th>
<th>MKB45</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>19.8</td>
<td>29.3</td>
<td>26.3</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>4.6</td>
<td>17.5</td>
<td>16.8</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>3.5</td>
<td>2.3</td>
<td>2.1</td>
</tr>
<tr>
<td>CaO</td>
<td>64.7</td>
<td>45.8</td>
<td>44.6</td>
</tr>
<tr>
<td>MgO</td>
<td>1.4</td>
<td>1.0</td>
<td>0.8</td>
</tr>
<tr>
<td>SO₃</td>
<td>2.8</td>
<td>2.7</td>
<td>2.3</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.1</td>
<td>0.2</td>
<td>0.1</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.7</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>H₂O</td>
<td>0.1</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>CO₂*</td>
<td>1.4</td>
<td>1.0</td>
<td>7.2</td>
</tr>
</tbody>
</table>

(*): obtained from TGA analysis

The resistance to chloride ingress is mainly dependent on the transport properties of the material which is considered. A migration test derived from ASTM C1202-97 standard test procedure has been used for estimating the diffusion coefficient of ions in the case of cement-based materials [7-10]. In this test, a fully saturated cylindrical sample (100 mm diameter and 30 mm thickness) is placed between an upstream cell containing both 0.5 M sodium chloride and 0.3 M sodium hydroxide and a downstream cell with only 0.3 M sodium hydroxide. Basically, a 10 V DC voltage is applied; the current as well as the voltage between the two sides of the tested sample are daily measured during 14 days (Figure 1). In addition, chloride concentration has been regularly monitored in downstream bath. Porosity value according to norm ASTM 642 and pore solution composition by mechanical extraction were also obtained on other replica samples to characterize the sample prior to the migration test.

![Figure 1. Schematic representation of the migration test (SIMCO)](image)
With the current and voltage evolution, the porosity value and the pore solution composition, finite element model STADIUM®[11] allows the determination of the tortuosity $\tau$ and, consequently, the diffusion coefficient of chloride ions $D_{Cl^-}$. The numerical extrapolation of the tortuosity, which is the parameter related to the refinement of the porous structure, is based on the expression of the total current as a function of the sum of the ionic flows [8], described here in equation 1:

$$I = SF \sum_{i=1}^{N} z_i J_i$$  \[Eq 1\]

Where $S$ is the surface of the sample [m$^2$], $z_i$ is the valence number of the species $i$, $F$ is the Faraday constant [C/mol] and $J_i$ are the ionic fluxes [mol/m$^2$/s], given by Eq 2 below [8, 11]:

$$J_i = -\phi D_i \nabla c_i - \phi \frac{D_i z_i F}{RT} c_i \nabla \psi - \phi D_i c_i \frac{\nabla \ln \gamma_i}{c_i}$$  \[Eq 2\]

Where $\phi$ is the water accessible porosity [m$^3$/m$^3$], $c_i$ is the molar concentration [mmol/l], $D_i$ is the diffusion coefficient [m$^2$/s] of the species $i$, $\psi$ is the electrodiffusion potential [V], $R$ is the ideal gas constant, $T$ is the temperature [°K], and $\gamma_i$ is the activity coefficient of the species $i$ [-].

The chloride ingress, characterized by the Nernst-Planck equation, is influenced by the reactions of the chloride ions with the cementitious matrix. The chemical reactions between chloride ions and AFm phases to form Friedel’s salt are taken into account according to an ionic exchange mechanism [Eq 3]. In this present work, the Stadium model not including any carboaluminates hydrates, the hypothesis that carboaluminates have the same chloride binding capacity than monosulfoaluminate hydrates has been taken, and form Friedel’s salt by ionic exchange [12], according to the following relationship [Eq 4]:

$$SO_4^{2-} - AFm + Cl^- \rightarrow Cl^- - AFm \text{(Friedel’s salt)} + SO_4^{2-}$$  \[Eq 3\]

$$CO_3^{2-} - AFm + Cl^- \rightarrow Cl^- - AFm \text{(Friedel’s salt)} + CO_3^{2-}$$  \[Eq 4\]

The first layer (2.5 mm thickness) of the samples after exposition to upstream bath was milled on 3 cm diameter perpendicularly to the migration direction. X-Ray Diffraction (XRD) measurements were carried out on the resulting powder using CuKα source ($\lambda$=1.54 Å).

3 RESULTS

3.1. Experiments

Current values monitored during 14 day of test are given in the Erreur ! Source du renvoi introuvable. below. Initial current value is much higher for OPC than for both other blended cements. While there is strong current decrease during 6 days and later on an increase for OPC, currents regularly but very slowly decrease in both MK30 and MKB45 mortars. Figure 2 show significantly distinct behaviours between OPC and the blended mixtures. For OPC; the applied voltage was 10V while 30V were applied to both MK30 and MKB45 blends in order to adjust initial currents within a window between 10 to 70 mA. The lower current observed in the blended mixes denote a higher sample resistivity and lower ionic flow.
Figure 2. Current evolution during migration test a) for OPC b) for the three different samples.

Figure 3 shows the evolution of chloride downstream concentrations during the migration test for the different mixtures. The first chloride ions detected coincide for each sample with the increase of the current observed in Figure 2. In the case of the PC and the MK30 samples, current and chloride concentration started to increase after about four days of testing. Note also that the increase of the chloride concentration was higher for OPC than for MK30, while no chloride ions were found in the downstream cell for the MKB45 mortars, in accordance with their current curves showing a continuous decrease.
Figure 3. Chloride downstream concentrations monitored during the migration test and quantified by chromatography.

On figure 4 & 5, the XRD diffractograms are given respectively before and after the migration test for the face exposed to the upstream bath. Before exposure, OPC’s phase assemblage contains ettringite and a large poorly crystallized amount of AFm, probably a mixture of monosulfoaluminate, hydroxyl-AFm and hemicarboaluminate. In MK30, ettringite, strätlingite and Ms are present. In MKB45, the large amount of hemicarboaluminate is visible, as well as ettringite and traces of strätlingite. After exposure, OPC contains only ettringite and Friedel’s salt, all the AFm were converted. In MK30, the phase assemblage remains relatively stable, with only traces of chlorocarboaluminates. In MKB45 a clear shift of the hemicarboaluminate peak can be observed toward Cl rich AFm phases. Hydrocalumite and Friedel’s salt seem to be coexisting. The hypothesis taken in Stadium seems therefore to be correct in a first approximation. Also, ettringite and to a lesser extent strätlingite do not seem to be destabilized by the chloride ions.
3.2. Analysis

The current evolution of each mixture has been analysed with Stadium©. Figures 5 and 6 show the modelling of the current measurements for both blended mixtures in comparison with the OPC mix. The extrapolation of the current curves provided the tortuosity of the tested sample. The tortuosity is related to the diffusion coefficient as follows [8]:

$$\tau = \frac{D_i}{D_0}$$

Where $D_i$ is the diffusion coefficient in free water of the species $i$.

The chloride diffusion coefficient have been estimated and are given in Table 2 with the other characteristics values regarding the transport properties of the cementitious mixtures. The initial phase assemblage was determined by Rietveld refinement [13]. The simulations fit reasonably well to the current evolution over time. The chloride diffusion coefficient values have been computed and are of $3.01 \times 10^{-11}$ m$^2$/s for OPC, $0.94 \times 10^{-11}$ m$^2$/s for MK30, and $0.29 \times 10^{-11}$ m$^2$/s for MKB45 respectively. These results mean that the porous structure of the blended mixtures is much more refined than the OPC one, and that the combined addition with limestone brings additional refinement in comparison with calcined clays addition only.

Figure 4. XRD patterns of the three samples taken from the surface exposed to the upstream bath before (a) and after (b) the migration test

Figure 5. Simulations of the current evolution during the migration test for OPC
The various characteristics of the material properties are summarized in Table 2. These properties show that the refinement of the microstructure is not dependent on the total porous volume. Indeed, the porosity of OPC was lower than blended mixtures while the tortuosity, related to the refinement of the porous network, was much higher for OPC. That indicates the remarkable effect of the calcined clays as cement replacement in concrete. The use of calcined provided a significant enhancement of the transport properties of the blended materials.

<table>
<thead>
<tr>
<th>Porosity (%)</th>
<th>Initial pore solution composition [mmol/L]</th>
<th>Inverse Tortuosity $[10^{-3}]$</th>
<th>$D_{Cl^-}$ $[10^{-11}$ m$^2$/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>PC</td>
<td>17%</td>
<td>Na$^+$ 108.9, K$^+$ 485.4, Ca$^{2+}$ 1.4, Al(OH)$_4^-$ 0.2, SO$_4^{2-}$ 1.9, OH$^-$ 38.4</td>
<td>3.01</td>
</tr>
<tr>
<td>MK30</td>
<td>19%</td>
<td>Na$^+$ 36.2, K$^+$ 42.5, Ca$^{2+}$ 0.5, Al(OH)$_4^-$ 0.2, SO$_4^{2-}$ 1.2, OH$^-$ 74.7</td>
<td>12.1, 0.94</td>
</tr>
<tr>
<td>MKB45</td>
<td>23%</td>
<td>Na$^+$ 41.5, K$^+$ 49.9, Ca$^{2+}$ 1, Al(OH)$_4^-$ 2.2, SO$_4^{2-}$ 1, OH$^-$ 83.9</td>
<td>3.7, 0.29</td>
</tr>
</tbody>
</table>

4 CONCLUSION

In this contribution, the resistance to chloride ingress has been studied by the STADIUM migration test. Blends containing 30% calcined clays, with and without 15% further substitution of cement by limestone powder have demonstrated to reduce by a factor three and by one order of magnitude respectively the coefficient diffusion of chloride ion in comparison with OPC.

The chloride chemical binding has been confirmed by XRD and Friedel salt and hydrocalumite were observed, but their formation does not seem to explain the reduction of the chloride diffusion, since OPC shows the highest Friedel’s salt formation.

These results are therefore attributed to the more tortuous porosity obtained with the calcined clays, with and without limestone. On-going research are carried out to better understand the role of chloride binding and pore structure; first results on ponding experiments, indicate very promising chloride resistance properties of blends containing high amount of calcined clays confirming these migration results [13].
5 ACKNOWLEDGEMENTS

The Fonds National Suisse is acknowledged for funding this work, part of the FNS Project “production of activated clays for low cost building materials in developing countries”. SIMCO is thanked for supplying the STADIUM cells as well as the access to the STADIUM Lab software.

6 REFERENCES

CHLORIDE PENETRATION DEPENDENCY ON FREEZE-THAW CYCLES

Miguel Ferreira (1), Markku Leivo (1), Hannele Kuosa (1), Lasse Makkonen (1) and David Lange (2)

(1) VTT Technical Research Centre of Finland, Espoo, Finland – miguel.ferreira@vtt.fi, markku.leivo@vtt.fi, hannele.kuosa@vtt.fi, lasse.makonen@vtt.fi

(2) University of Illinois, Illinois, USA – dlange@illinois.edu

Abstract
Concrete research is focusing ever more on the combined effect of degradation mechanisms on the durability of reinforced concrete structures. While most of the research has been directed at combined carbonation and chloride penetration, recently some attention has been given to emulate combined degradation conditions, especially those existing in Nordic countries (frost attack/chloride penetration), with regards to the harsh environmental conditions. Frost attack of concrete effects the chloride penetration by reducing the concrete cover, and more importantly, by changing the characteristics of the surface and internal concrete due to cracking. Recent research has shown there to be a synergetic effect, but no steps have been taken to characterize and comprehend the mechanisms involved, and to develop a procedure to make it possible to transfer this knowledge to the industry and into practise.

As part of an ongoing research progress addressing this need, series of tests were undertaken to ascertain what influence freeze-thaw cycles would have on the transport of chlorides into concrete. In this paper the, the preliminary result of this research project are presented.

1 INTRODUCTION

In Finland, reinforced concrete structures (RCS) have to perform in rather difficult conditions due to extremely harsh winters, resulting in unique combinations of degradations mechanisms. Traditionally, from a research perspective, freeze-thaw and carbonation have been considered as the predominant degradation mechanisms, with chloride penetration being relegated to a secondary status. However, in light of recent research in Finland [1-3] and internationally, more attention is now being drawn to other degradations mechanism such as chloride penetration and alkali-aggregate reactions and the combination of these with freeze-thaw. Much research has been dedicated to describing the phenomena of freeze-thaw, with
many significant contributions by [4-19]. Despite all the advances in this research, little attention has been given to the interaction between freeze-thaw and chloride ingress. Freeze-thaw reduces the concrete cover due to scaling (in the presence of salts), and by changing the characteristics of both surface and internal concrete due to cracking [17]. As a result, it has been shown [1] that frost attack affects chloride penetration, but research has noted that this phenomenon is not yet understood.

Recent research has shown there to be a synergetic effect between freeze-thaw and chloride penetration, but no steps have been taken to characterize and comprehend the mechanisms involved, and to develop a procedure to make it possible to transfer this knowledge to the industry and into practice [1-3, 19]. For this reason, a research project was undertaken to study the influence that freeze-thaw cycles have on chloride ingress. This paper describes the preliminary result of the ongoing research progress.

2 METHODOLOGY

The purpose of the testing was to ascertain the influence of freeze-thaw cycles on chloride penetration into concrete. For this purpose, two distinct concrete s were subject to three different freeze-thaw test cycles and two ponding conditions. In the following the concrete and the test setups are described.

2.1 Concrete mix design and specimen conditioning

Two concrete mixes were prepared with a 0.42 and 0.55 w/b ratios (B42 and B55, respectively). A CEM I 42.5 N-SR3 was used to minimise the possibility of chloride binding. A plasticizer (VB-Parmix) was used in the B42 mix, and an air entrainment agent (Ilma-Parmix) for both mixes. Details of the concrete mixtures and their workability are presented in Table 1, and the characteristics of the air entrainment measured on hardened concrete in Table 2.

Table 1: Concrete compositions and workability

<table>
<thead>
<tr>
<th>Series</th>
<th>w/b</th>
<th>Water (l/m³)</th>
<th>Binder (kg/m³)</th>
<th>Aggregate – Total (kg/m³) &amp; fractions (%)</th>
<th>Slump (mm)</th>
<th>Air (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Total</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>&lt;0.125</td>
<td>&lt;0.250</td>
<td>&lt;4.0</td>
</tr>
<tr>
<td>B42</td>
<td>0.42</td>
<td>175</td>
<td>420</td>
<td>1695</td>
<td>5.8</td>
<td>12.1</td>
</tr>
<tr>
<td>B55</td>
<td>0.55</td>
<td>195</td>
<td>355</td>
<td>1716</td>
<td>4.3</td>
<td>9.7</td>
</tr>
</tbody>
</table>

Table 2: Characteristics of the air pores of hardened concrete.

<table>
<thead>
<tr>
<th>Series</th>
<th>Protective AP 0.02-0.80 mm (%)</th>
<th>Compaction AP &gt; 0.800 mm (%)</th>
<th>Total AP ≈ (%)</th>
<th>Specific surface for protective AP (mm²/mm³)</th>
<th>Spacing factor for protective AP (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B42</td>
<td>2.9</td>
<td>1.2</td>
<td>4.1</td>
<td>33</td>
<td>0.21</td>
</tr>
<tr>
<td>B55</td>
<td>5.1</td>
<td>0.8</td>
<td>5.9</td>
<td>21</td>
<td>0.24</td>
</tr>
</tbody>
</table>

The concrete batches were produce in a vertical axis mixer with a 200 litre capacity. Cubic specimens 150 mm in dimension were cast in moulds and compacted using a vibrating table.
24 hours after casting the specimens were removed from the moulds and permanently stored in a climate chamber at RH≥95% at 20 ± 2 ºC until testing. From the results in 2, it can be concluded that very good air entrainment was achieved in both concretes mixes, as shown by the low spacing factor and the specific surface.

Concrete samples were kept in the climate chamber for approximately 8 months to minimise the influence of microstructure changes due to continuous hydration on the test results. After this period, the specimens were stored in a climate chamber at RH≥65% for an additional month. Ten days prior to testing, the concrete specimens were prepared according to the procedure defined in the CEN/TS 12390-9:2006 [20], except with regards to the age of the specimens. A test specimen constitutes a concrete prism with 150x150x50 mm. After a three day period where test specimens were ponded with a layer of deionised water of 3mm, the water was replaced with a 3% by weight of NaCl solution.

2.2 Test setup

Three different freeze-thaw testing cycles were defined to assess influence of freeze-thaw cycles on the transport of chlorides in concrete. These have one day duration, and follow the reference test procedure curve for freeze-thaw scaling, varying only in the minimum temperature reached: -5, -10 and -20°C. The rate of freezing was kept as close as possible to the reference test [20] (see Figure 1).

![Figure 1: Temperature of the solution on the freeze-thaw specimens in the three different freeze-thaw cycles.](image)

The total number of freeze-thaw cycles is 120 for the 1 day test cycle, although currently only the results up to 70 days are available for analysis. The choice of different lower (negative) limit temperature in the cycles, resulting in different durations of time at negative temperatures, was to promote different freezing-thawing behaviour of the brine solution in the pore structure of the concrete. As the temperature decreases, and the longer a certain low temperature is maintained, a greater volume of the pore structure is frozen [3]. In addition, two ponding tests were carried out at +20°C and +5°C, where no freeze-thaw occurs.
During testing, scaled material mass, specimen mass variation and fundamental frequency were measured periodically. In addition, at certain intervals specimens were removed and the chloride profiles determined.

3  RESULTS

3.1  Water uptake

In Figures 2 and 3 the water uptake as a function of time (i.e. freeze-thaw cycles) is presented for concrete B55 and B42, respectively. The water uptake values were not adjusted for the weight of scaled material as this was less than 0.7% of the amount of water up taken.

![Figure 2: Water uptake as a function of the number of freeze-thaw cycles for concrete B55.](image)

![Figure 3: Water uptake as a function of the number of freeze-thaw cycles for concrete B42.](image)

The average value of relative dynamic modulus (RDM), measured using fundamental frequency, after 70 freeze-thaw cycles for all three freeze-thaw tests, was 99.5%. These values
indicate that there is practically no internal damage taking place in the concrete. This was expected given the quality of the air entrainment achieved (see Table 2).

Figure 4: Chloride profiles after ponding at +5°C (a) and at +20°C (b).

Figure 5: Chloride profiles after freeze-thaw testing at -5/+20°C (a), -10/+20°C (b), and -20/+20°C (c). Comparison of chloride profiles for B42 after 70 freeze-thaw cycles (d).
3.2 Chloride profiles
In Figures 4 and 5 the chloride profiles are presented for both concretes, and after 28 and 70 cycles (i.e. days), subject to surface ponding and freeze-thaw tests, respectively. Figure 5(d) presents a comparison of chloride profiles for the B42 concrete from different freeze-thaw tests after 70 cycles.

4 ANALYSIS
A theoretical analysis of the test setup identifies as the main transport mechanisms contributing to the ingress of water and chlorides during the course of the test capillary action, diffusion (both water vapour and chloride in the solution) and micro ice lens pumping (as a result of freeze-thaw loading) [3, 14, 17].

4.1 Water uptake
The initial internal moisture condition of the test specimens conditions which transport mechanisms are predominant at any given moment of the test. After a month in which samples were kept at 65% relative humidity, the samples where water ponded for 3 days prior to testing. As a result, a large capillary uptake of water occurs without chlorides. Measurements performed on the same concrete, but at a younger age show that after three days, the B55 concrete had taken up approximately 97% of its capacity due to capillary action, whereas the B42 concrete had taken up approximately 84%. With time, a slight reduction in the volume of water taken up, and an increase in the time necessary for this uptake to occur, is observed. Therefore, at the time of testing, it is can assume that the concrete still has a capacity to up take water due to capillary action, but limited due to the preliminary 3 day ponding with water.

The results for water uptake (see Figures 2 and 3) show that the water uptake is higher for C55 concrete than for C42. This expected based on the characteristic of the concrete composition (i.e. w/b ratio) and the results of capillary uptake.

Samples subject to freeze-thaw cycles show a larger uptake of water when compared to the samples subject only to ponding. This suggests that during freezing, the formation of micro ice lenses might be assisting water uptake. Furthermore, no clear difference is noticed in the uptake of water between the freeze-thaw tests at different temperatures, suggesting that the intensity (i.e. minimum negative temperature) of the freeze-thaw cycle does not have a significant influence on the uptake of water.

4.2 Chloride profiles
Some chloride profiles show a reduction of the concentration near the surface, commonly referred to as the convection zone. These areas were not considered in the analysis.

An analysis of the ponding chloride profiles (Figure 4) reveals the differences expected between a B55 and a B42 concrete, i.e., deeper chloride profile for B55. When looking at the influence of ponding temperature, a clear difference is seen after 70 days, but not at 28 days (see Figure 5(d) for B42 concrete).

The profiles from the different freeze-thaw tests show that the influence of the minimum negative temperature reached during the test is small or negligible. In addition, these profiles also show small or negligible differences when compared to the ponding profiles. This is an unexpected finding as the water uptake in Figures 2 and 3 is clearly larger for the concretes with freeze-thaw cycles than with just ponding. It is thought that this water uptake is mainly
salt free as it occurs due the ice lens pumping effect, and could possibly dilute the concentration of the chloride ions solution, affecting the profiles accordingly. However, the profiles show no such reduction in chloride concentration.

5 CONCLUSIONS

The experiments, designed to mimic real world conditions, allow for interaction of multiple transport mechanism. They are complex experiment because they combine capillary water uptake with diffusion at the same time, and overlaying these with the action of freeze-thaw. The results provide an insight into possible interaction therefore it is important to observe the outcome of verified behaviour. It can be difficult to extract a full explanation without additional experiments; however, based on the limited tests and samples tested, the following conclusions can be drawn:

- The water uptake for B52 is greater than B42, as would be expected due to the pore structure of these concretes;
- Concrete samples subject to freeze-thaw cycles have a greater uptake of water than subject to pure ponding, suggesting that micro ice lens pumping is active;
- No clear difference is noticed in the uptake of water by the intensity of the freeze-thaw cycle (low negative temperature);
- The depth and volume of B55 profiles is greater than B42 profiles, as would be expected due to the pore structure of these concretes;
- The effect of the freeze-thaw cycles on the chloride profile is small or negligible, which is surprising considering the increased water uptake that the same concrete presents. This could suggest that diffusion is occurring in saturated microstructure unaffected by the action of freeze-thaw, i.e. diffusion dominates, or, that the interaction between competing transport mechanism is complex, with no pure mechanism explaining consistently the findings, but their current interaction results in profiles identical to those of pure ponding.

ACKNOWLEDGEMENTS

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REFERENCES


COMBINING GEOGRAPHICAL INFORMATION SYSTEMS AND MULTI-SCALE MODELLING TO PREDICT SERVICE LIFE

Ivan S. Cole (1) and Wayne D. Ganther (2)

(1) CSIRO Division of Materials Science and Engineering, Clayton, Victoria, Australia, – Ivan.Cole@csiro.au
(2) CSIRO Division of Materials Science and Engineering, Highett, Victoria, Australia, – Wayne.Ganther@csiro.au

Abstract

Service life prediction (SLP) allows materials selection tailored to actual usage and environmental conditions and can guide maintenance and infrastructure planning. To be effective SLP must be done on a country or continental scale while incorporating local effects and building design features. Combining geographic information systems (GIS) with multi-scale models (MSM) of material degradation can yield effective SLP. The GIS systems provide an easy means for the user to input specific data about the location and the building and receive life estimates. They are an effective geo-sensitive data base of geographic and climatic data and they facilitate particular spatially sensitive calculations. The MSM integrates models from $10^{-3}$ m to $10^{-6}$ m to simulate the processes that control corrosion. Processes include, production, transport and deposition of pollutants onto components, the surface conditions at components, wetting or moisture content of the building material, the degradation of the building material. The MSM runs on a three hourly basis and calculates each factor and the resulting degradation each 3 hours. Degradation on different facades or internal spaces of a building can be calculated separately for buildings across continental domains. The system is used to predict the life of building components across Australia.

1 INTRODUCTION

Service life predictions are usually carried out on a component-by-component basis and must incorporate variations in component design, material type, usage and environment and thus large databases of component life must be developed. The end-use of the service life prediction may also necessitate different levels of detail in these databases (1). For example SLP of known products in known environments does not need a knowledge of the effect of component design on life while such knowledge is absolutely needed if SLP is to be used to select from a range of designs or materials.
There are a number of ways to generate such databases of SLP including data mining of maintenance records, inspection programs, expert opinion surveys and the development of validated models. This paper will concentrate on model development. Models based on the actual physical processes of material degradation can take into account local and usage factors that may influence these degradation processes and so can be a very accurate method of predicting the life of individual components. Such process models have been developed to predict the life of concrete (2-6) and metal products(7-9). Akiyama et al’s (6) model is particularly interesting as it combines a model of airborne salinity, corrosion initiation (based on Fickian transport of chlorides), oxide growth and concrete cracking and as such is one of the few concrete lifetime models to consider explicitly geographic variations in chloride level. In atmospheric corrosion a number of processes models have been developed (7-9) to predict metal corrosion. One of the leading models is Graedel’s ‘Gildes’ (Gas, inter-face, liquid, deposition layer, electro regime and solid) model, which systematically looked at the chemical interactions across a range of interfaces. However, Graedel (7) applied the model to relatively simple gaseous and interface regimes, and therefore a greater understanding of the external environment was required. The holistic model of corrosion of Cole et al. (10-17) aims to incorporate a deep understanding of local environmental and usage factors.

The holistic model of Cole et al (10) combines processes that control atmospheric corrosion on a range of scales (see Figure 1), from macro through meso to local, micro, micron and lastly electrochemical (10). Macro refers to gross meteorological conditions (polar, subtropical etc.), “meso” refers to regions with dimensions up to 100 km², “local” is in the immediate vicinity of a building, while “micro” refers to the absolute proximity of a material surface. “Surface response” refers to largely physical responses of a surface such as deposition and retention of pollutants or condensation and evaporation. “Micron” refers to interactions within the metal/oxide/electrolyte interfaces and electrochemical refers to the charge transfer and redox processes at the same interface.

For SLP it is essential to model how design and the local environment interact and thus rather than just model the performance of an exposure plate which is subject to a uniform environment it is essential to model a real building and take into consideration how the buildings features alter such effects as aerosol deposition, cleaning of surfaces by rain washing, solar heating etc. The framework of Cole et al. (10-17) does this across a range of dimensions by integrating geographic, meteorological, building information and corrosion models. This paper will outline the role of role of geographic information systems and multi-scale modelling in facilitating this integration.
2 SALT PRODUCTION

In Australia, the main atmospheric pollutant is marine aerosols which are produced both in the open ocean and by breaking surf [10]. In the open ocean, aerosols are produced by white-caps of ocean waves. Whitecap production varies systematically with latitude and season, being at a maximum at low latitudes in July and at a maximum at high latitudes in December, and low all year round in tropical seas. Experimental studies support this observation [16, 17]. Surf aerosol production depends only on local wind speed and fetch [18].

This dependence on aerosol production on local factors requires integration of the multi-scale framework with the geographical information system. Aerosol production is calculated for over 15000 points around the Australian coast at spacing of 5 km. The calculation is undertaken within the MSM by analytical formulae derived from the work of McKay et al. (18) but requires the GIS system to supply wind speeds, fetch distances and a coast type definition to the MSM. The wind speeds and the fetch is provided for the 8 principal compass directions. The GIS system defines in the coast type as adjacent to open ocean, or an open or closed bay. Fetch is the distance of open ocean in a particular direction from a point on the coast, (see Figure 2). This Figure displays the southern section of Spencer Gulf in South Australia which opens onto the Southern Ocean. At each coastal square the Fetch is determined in the eight cardinal directions by measuring the length of a vector along each direction across the water body to the nearest land. For example from the selected square (No 11,106) the distance to the nearest land in the easterly direction is 128Km, in excess of 300 Km in the southerly and only 22 Km in the northern direction. A wave will only build to its maximum height if it is the Fetch is in excess of 300Km and thus waves from the south will reach their maximum height while thus from the east and north will be moderately and severely reduced in height respectively. The reduction in wave height will lead to a similar reduction in surf aerosol production.
3 SALT TRANSPORT

Factors controlling aerosol transport are also outlined in Cole et al. [10]. Aerosol residence times and thus transport distances are controlled by convection, gravity and aerosol scavenging by cloud drops, raindrops and physical objects on the ground (trees, buildings etc.). Thus, marine aerosol transport is likely to be favoured by dry climates with low rainfall and low ground coverage, while it will be restricted by humid and high rainfall climates with forest cover. Aerosols produced by surf tend to be coarse (5–20 µm) and those produced from whitecaps are generally smaller (0.5–3 µm). Thus, surf-produced aerosol rapidly deposits (due to gravity), while ocean-produced aerosol may be transported considerable distances.

In a similar manner to salt production the GIS system must be linked to the multi-scale model to allow the calculation or aerosol transport between the coastal sources and the location being modelled. This is schematically shown in Figure 3. For the modelled point all the coastal points that could contribute to the airborne salinity at the modelled point are selected. A direction line (transept) from a coastal point to the modelled point is drawn and the terrain, landform and climate along this transept and the compass direction of the transept are defined. These details, along with wind frequency information at the modelled point are passed to the MSM. For each positive wind direction the MSM looks at the transepts in this directions and then calculates the fraction of aerosol produced at the coastal point that are transported to the modelled point taking into account transept characteristics and sums up across all wind directions to give the total airborne salinity. The criticality of accurate terrain data is illustrated by Figure 4 which shows the effect of ground roughness on the salt concentration for surf-generated salt. The wind speeds are 2.5 and 5.0 m/s. The output concentrations are at a height of 2.0 m above ground level. The ground roughness has a huge effect on the surf-generated salt concentration at this height. Table 1 gives some typical roughness values of various terrain types (from AS/NZ 1170.2:2002). A salinity map of Australia (Figure 5) can then be constructed by modelling a grid of points.
Figure 3 Schematic diagram of Calculation of Airborne salinity at an Inland Location

Table 1 Typical roughness values

<table>
<thead>
<tr>
<th>Roughness (m)</th>
<th>Terrain</th>
<th>Roughness (m)</th>
<th>Terrain</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>City buildings (10–30 m)</td>
<td>0.06</td>
<td>Level wooded country, suburban buildings</td>
</tr>
<tr>
<td>1</td>
<td>Forests</td>
<td>0.02</td>
<td>Isolated Trees, long grass</td>
</tr>
<tr>
<td>0.8</td>
<td>High-density metropolitan</td>
<td>0.008</td>
<td>Uncut grass, airfields</td>
</tr>
<tr>
<td>0.4</td>
<td>Centre of small towns</td>
<td></td>
<td>Cut grass</td>
</tr>
</tbody>
</table>

Figure 4 Variation of surf-generated aerosol as a function of wind speed and ground roughness
4 SALT DEPOSITION

Marine aerosol deposition is primarily controlled by wind turbulence, and deposition onto an object is a function of turbulence intensity, wind speed, object shape and, to a lesser extent, aerosol size [11]. For marine aerosols with sizes of 0.1 to 10 µm, the deposition efficiency (as a function of size) is relatively constant, while the efficiency increases rapidly for aerosol sizes above 20 µm, so that the efficiency of 100 µm aerosol is roughly four times that of the 20 µm aerosol. The size and shape of object is also important. The deposition on an exposed plate (at 45°) is likely to be more than 1.5 times that on an equivalently exposed salt candle. For complex forms such as dwellings, deposition efficiency will vary across a structure, with deposition being highest at the edges of the structure where turbulence is highest.

5 WETTING AND SURFACE TEMPERATURE

Models have been derived to estimate the surface temperature of an exposed surface considering undercooling to the night sky and daytime solar heating and convection effects [16]. Further work [13] predicts the RH at the metal surface and thus when hygroscopic salts (NaCl, MgCl) on the surface will wet. The evaporation of such moisture films has been modelled [14] so a complete picture of moisture cycles has been developed. The role of wind and rain in removing deposited salts from the surface is also modelled [16, 17].

6 CORROSION MODELS

To estimate the rate of corrosion the “state” of a surface is calculated on a 3 hourly basis. Four states are defined, State 1 – Dry, State 2 – Wet From Rain, State 3 – Wet From Wetting of Hygroscopic Salts, State 4 – Drying. A corrosion rate depending on pollutant levels is determined for each state from dose function tests in accelerated test chambers and then the annual corrosion is determined by summing the three hour corrosion rates. The A map of the estimated rate of corrosion of exposed zinc is presented in Figure 6. The predictions of the model are compared with measured one year data for 40 sites around Australia. The fit is quite high (R-sq= 0.82). The model has been extended to predict the lifetime of metal
components on diverse parts of a building façade and thus having diverse degrees of exposure to wind, rain and airborne pollutants.

![Figure 6. Map of corrosion rate of zinc (g/m².year)](image)

7 CONCLUSIONS

This example has highlighted how process models can be linked to a geographic information system to predict accurate service life estimates of different metallic components at different positions of a building at locations throughout Australia. Both the GIS system and the process models are required. The GIS system is necessary to allow the user to easily input spatially important specification (location etc), calculate spatially dependent parameters and in particular those that depend on vectors (Fetch etc) and to simply represent data. On the other hand the multi-scale model is critical to assess and bring to give the range of factors that control atmospheric corrosion.

REFERENCES


MULTI-OBJECTIVE DECISION-MAKING SUPPORTING SYSTEM OF MAINTENANCE STRATEGIES FOR DETERIORATING REINFORCED CONCRETE BUILDINGS

Chien-Kuo, Chiu

Department of Construction Engineering, National Taiwan University of Science and Technology, Taipei, Taiwan. ckchiu@mail.ntust.edu.tw

Abstract
This work presents an integrated system that can help engineers search for the optimal maintenance strategy for deteriorating RC buildings via multi-objective optimization. This system applies Particle Swarm Optimization (PSO) and the Pareto optimal solution to achieve the optimization of the multiple objectives of minimal LCCs (economy), minimal failure probability of the building (safety), minimal spalling probability of concrete cover (serviceability), maximum rationality, and minimal maintenance times. Additionally, to enhance computing efficiency in optimization, probabilistic effect assessment models for setting repair and retrofitting strategies are proposed. The proposed system has four main modules: (1) Deterioration analysis; (2) Seismic performance assessment; (3) Setting maintenance strategies; and (4) Multi-objective optimization. These four modules are integrated into the 「Multi-objective Decision-making Support System for Maintenance Strategies for Deteriorating Reinforced Concrete Buildings」 (MDMS-RCB). Finally, a case study is conducted to demonstrate application of the proposed system.

1 INTRODUCTION
Reinforced concrete (RC) structures or buildings deteriorate over time when exposed to corrosive environments. When the service-life in terms of the structural safety or serviceability of a deteriorating RC building does not meet the original target, lifecycle maintenance strategies should be implemented. Once the cause of corrosion has been identified, structural members, beams, or columns with reinforcing steel components corroded by carbonation or chloride ingress can be repaired or retrofitted. Appropriate repair or retrofitting to maintain the required performance or extending the specified service period for a building is essential in 「Sustainable Development」 or 「Sustainable Construction」. A decision-making support system is needed that can help engineers comprehend the benefits of maintenance strategies. The proposed system has four main modules: (1) Deterioration analysis—mechanical analysis of deteriorating RC beams and columns; (2) Seismic performance assessment - seismic performance assessment of a deteriorating RC building; (3) Setting maintenance strategies - determining maintenance strategies for a deteriorating RC building; and (4) Multi-objective optimization - appropriate maintenance strategies are suggested using Particle Swarm Optimization (PSO). These four modules are integrated into
the 「Multi-objective Decision-making Support System for Maintenance Strategies for Deteriorating Reinforced Concrete Buildings」 (MDMS-RCB) (Fig. 1). Finally, a case study is conducted to demonstrate application of the proposed system.

Figure 1. Proposed system in this work.

2 SEISMIC RISK EVALUATION FOR DETERIORATING RC BUILDINGS

2.1 Deterioration induced by chloride ingress

Generally, the corrosion of reinforcing steel due to chloride ingress is divided and modeled into three stages (initiation stage, propagation stage, and deterioration stage), based on previous studies [1]. The initiation stage is defined as the time period required for corrosion to take place. According to “Recommendations for Durability Design” proposed by AIJ [2], we assume that the surface chloride concentration of concrete does not increase over time and the chloride diffusion coefficient of concrete is time-independent in this work.

Based on the results of previous studies [3], we assume that corrosion is initiated when the chloride concentration on the surface of the reinforcing steel attains a critical threshold value, which is a uniformly distributed random variable in the range of 1.0 – 1.2 kg/m³. In addition, the critical threshold value can be set on the basis of concrete, steel and site conditions. In this work, we take into account the uncertainties pertaining to the apparent diffusion coefficient, concrete cover, and surface chloride concentration induced by environmental or construction conditions; the probability of the initiation of corrosion in the outermost reinforcing steel layer is calculated using Monte Carlo simulation (MCS). In the simulation, corrosion is assumed to have occurred when the probability of corrosion initiation exceeds 0.1 at 90% confidence level (failure probability: 0.1); this condition is consistent with the prediction of corrosion initiation specified in ASTM C876 (1991). In the probabilistic analysis, the apparent diffusion coefficient, concrete cover, and surface chloride concentration are treated as lognormal random variables, as shown in Table 1. The propagation stage is defined as the period from corrosion initiation to cracking initiation on the concrete surface. This stage is followed by the deterioration stage, during which the deterioration process progresses and the
structural capacity of the members is degraded significantly. Based on a previously developed model [4], the rate of corrosion in these two stages is assumed identical, and the corrosion quantity (expressed in terms of the weight loss) is treated as a lognormal random variable with a mean value, $\Delta W_{\text{avg}}$, a coefficient of variation of 0.5.

Table 1. Statistics of deterioration parameters.

<table>
<thead>
<tr>
<th>Uncertainty</th>
<th>Distribution</th>
<th>C.O.V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface chloride content ($C_{\text{o}}$)</td>
<td>Lognormal</td>
<td>10%</td>
</tr>
<tr>
<td>Depth of concrete cover ($x$)</td>
<td>Lognormal</td>
<td>20%</td>
</tr>
<tr>
<td>Apparent diffusion coefficient ($D_{\text{p}}$)</td>
<td>Lognormal</td>
<td>30%</td>
</tr>
<tr>
<td>Critical threshold of chloride concentration ($C_{\text{limit}}$)</td>
<td>Uniform</td>
<td>1.0 – 1.2 kg/m$^3$</td>
</tr>
<tr>
<td>Weight loss ($\Delta W_{\text{avg}}$)</td>
<td>Lognormal</td>
<td>50%</td>
</tr>
</tbody>
</table>

2.2 Degradation of structural capacity of corroded RC columns

The reduction in the shear and flexural capacity of an RC column due to corrosion is taken into account in the investigation of safety performance under earthquake conditions. About doing the analysis in structural capacity of corroded RC columns, instead of the reduction in the area of the reinforcing steel due to corrosion, the degradation in the material properties, i.e. yield stress and modulus of elasticity of steel, and ultimate bond stress between steel and concrete, are adopted to do the analysis in structural capacity following Lee et al [5]. In addition to the flexural capacity of RC members caused by the yielding of main bars, it can also be calculated on the basis of bonding failure between the main bars and concrete after evaluating the ultimate bond stress corresponding to the given corrosion level of the reinforcing steel [6]. The flexural capacity of RC members caused by the yielding of the main bars corresponding to any corrosion level, $M_{\text{yu}}$, can also be estimated based on the past research [6]. Then, comparing these two flexural capacity ($M_{\text{yu}}$ and $M_{\text{bu}}$), and either the yielding or bonding failure, could be determined; the result is required to estimate the structural capacity curve of the member. The shear capacity of an RC column in the case of bonding failure or stirrup yielding induced by corrosion, can be estimated by the truss-arch theory associated with material properties of corroded reinforcing steels.

2.3 Safety performance and failure probability

After generating the structural capacity curve of each column within a building, the structural capacity curve of each floor can be calculated using the simplified pushover analysis [7]. Then, the structural capacity curve of a building can be acquired using the structural capacity curve of each floor and the distribution coefficient of each floor for lateral forces, i.e., the first mode of natural vibration. In the structural capacity curve of a building, the x-axis represents roof displacement and the y-axis represents base shear force. Moreover, this work utilizes the capacity spectrum in ATC-40 [8] to identify ultimate deformation capacity, $\delta_u$, yielding strength, $F_y$, and the elastic fundamental period, $T_y$, of the single degree of freedom (SDOF) system for a building. This work presents the proposed method for evaluating seismic structural damage. The model developed by Park et al. [9], the most widely used model in literature, is a linear combination of maximum deformation response and hysteretic energy. The damage index of this model is expressed as
where \( D_{P&A} \) is the damage index—an empirical measure of damage \((D_{P&A} \geq 1.0\) indicates total damage or collapse); \( \delta_M \) is maximum response deformation; \( dE \) is incremental absorbed hysteretic energy (excluding potential energy); and \( \beta \) is the coefficient for cyclical loading effects (as a function of structural parameters, it can be set at 0.05 for general RC buildings).

The limiting values of \( D_{P&A} \) for each damage state may differ among buildings or structure type and damage assessments [12]. Based on the evaluation model of seismic structural damage and hazard curve of response spectral accelerations, the cumulative distribution function (CDF) of the damage index can be estimated when we assume structural properties are deterministic values, \( i.e., \delta_y, \delta_u, \) and \( F_y \). According to limiting values of \( D_{P&A} \) for each damage state, the occurrence probability of each damage state can then be assessed. Moreover, this work defines the occurrence probability of total damage or collapse as the failure probability of a building, which can be regarded as its safety performance.

### 2.4 Serviceability Performance and Spalling Probability of Concrete Cover

The serviceability performance of RC structures is affected by spalling, which is caused by the formation of a crack with a width greater than 0.5 – 1.0 mm [10]. The extent of corrosion that leads to spalling can be estimated using Eq. (2) and Eq. (3) [11].

\[
\begin{align*}
\delta_c &= 0.141 \exp\left(\frac{1.078 x}{d_{bo}}\right) \times c_w \\
\Delta W_{avg} &= \frac{4 \times w_c}{\rho_s d_{bo}}
\end{align*}
\]

where \( w_c, c_w, \) and \( \rho_s \) are the corrosion of steel (\text{mg/mm}^2), crack width (mm), density of steel (7.85 \text{mg/mm}^3), respectively.

Therefore, when the extent of corrosion on the outermost reinforcing steel component in an RC member exceeds the crack limit concerning spalling occurrence, spalling can be assumed to occur on the surface of the concrete. As described in Section 2.1, MCS is used in the evaluation of spalling probability of a corroded member; then, the annual spalling probability of a building, \( P_s \), is expressed in terms of the weighted average computed on the basis of the surface area exposed in the air of corroded members and their spalling probabilities. Additionally, it is reasonable to assume that chloride ingress results in a high spalling probability, which subsequently leads to high repair costs in the maintenance. Therefore, the spalling probability of a building, \( P_s \), is used to define the deterioration degree of a building and calculate repair costs in this work.

### 2.5 Estimation Model of Seismic Risk

According to ATC-13 (1985) and Hazus-99 (FEMA 1997), the damage ratio, \( C_D \), defined by Eq. (4), is applied to describe the degree of damage caused by an earthquake. That is, the damage ratio is repair or recovery cost, \( C_L \), normalized by the initial construction cost of a building, \( C_I \). Table 2 shows the relationship between the damage ratio and damage state. Based on the damage ratio and occurrence probability for each specified damage state, this work applies Eq. (5) to derive annual seismic risk, \( C_{Risk} \).

\[
C_D = \frac{C_L}{C_I} \quad (4)
\]
\[ C_{\text{Risk}} = \sum_{i=0}^{4} (C_D^i \times P_D^i) \] (5)

where \( i \) is the damage state (i=0 – 4; 0: non-damage; 1: slight damage; 2: moderate damage; 3: serious damage; 4: total damage or collapse); \( C_D^i \) is the damage ratio of \( i \) damage state; \( P_D^i \) is the occurrence probability of \( i \) damage state.

Table 3. Relationship between the damage ratio and damage state.

<table>
<thead>
<tr>
<th>Damage state</th>
<th>Damage ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Non-damage</td>
</tr>
<tr>
<td>1</td>
<td>Slight</td>
</tr>
<tr>
<td>2</td>
<td>Moderate</td>
</tr>
<tr>
<td>3</td>
<td>Serious</td>
</tr>
<tr>
<td>4</td>
<td>Total damage or collapse</td>
</tr>
</tbody>
</table>

When a building is totally damaged or collapsed by an earthquake, non-monetary losses of reconstruction and social responsibility (e.g., loss of life) should be included in recovery cost.

3 PROBABILISTIC EFFECT ASSESSMENT MODELS AND COST ESTIMATION FOR REPAIR AND RETROFIT

3.1 Cost Evaluation of Repair and Retrofitting Strategies

To enhance the accuracy in estimating the LCC of a structure, a cost evaluation method based on assessments of practical repair and retrofitting technologies is needed. The unit prices of repair and retrofitting technologies, and the information on the expected extent of the repair or retrofit should be considered in the estimation of repair and retrofitting costs. In this work, deterioration degree defined by spalling probability is adopted as an index representing the expected extent of maintenance activities. Thus, we reasonably assume repair and retrofitting technologies in each maintenance strategy change when the deterioration degree changes. The unit prices of repair and retrofitting technologies are obtained from previous studies [12] and from some local repair and retrofitting companies. Thus, the cost of each repair and retrofitting strategy can be calculated depending on the degree of deterioration and unit prices of these technologies [12]. As no domestic studies of seismic retrofitting costs of RC buildings exist, this work refers to data from the Building Research Institute of Japan and evaluates these data using Eq. (6):

\[ C_{\text{Ret}} = \frac{C_I}{5} \left( \frac{R_u}{R_i} + 4 \right) \] (6)

where \( C_{\text{Ret}} \) is the seismic retrofitting cost; \( C_I \) is the initial construction cost; \( R_i \) is the response spectral acceleration; \( R_u \) is the response spectral acceleration specified in the seismic design code.

3.2 Estimation Model of Life-cycle Cost

In addition to seismic risk, construction cost, retrofitting cost, and repair cost must be considered. Therefore, the LCC, \( C_T \), is derived by Eq. (7). Seismic risk can be calculated using the estimation method in Section 2.5. Moreover, to enhance computing efficiency for
optimization, the seismic risk curve can be generated using the probabilistic effect assessment models for repair and retrofitting in Section 3.1 [12].

\[ C_r = C_i + \sum C_{Rep} \times \frac{1}{(1+k)^{t_{Rep}}} + \sum C_{Ret} \times \frac{1}{(1+k)^{t_{Ret}}} + \sum C_{Rsk} \times \frac{1}{(1+k)^{t_{Rsk}}} \]  

(7)

where \( C_{Rep} \) is the repair cost; \( t_{Rep} \) is the time of seismic retrofit; \( t_{Ret} \) is the time of the maintenance time; \( k \) is the discount rate.

### 3.3 Maintenance Strategy based on the Decision and Building Deterioration

The maintenance strategy based on a decision by the owner or user and building deterioration can be determined by considering operational loss or the residual value of a structure. However, as the residual value of a structure is unlikely to be evaluated, this work uses residual life to represent an owner’s or user’s decision. Take a building with a specified service life of 100 years as an example. If the building’s age is 30 years, its residual life is 70 years, and the owner or user should prefer a permanent maintenance strategy to a temporary or prolonged maintenance strategy. In this work, residual life, the safety performance of the structure, and its apparent deterioration are evaluated when considering a maintenance strategy [12].

### 4 MULTI-OBJECTIVE OPTIMIZATION USING PSO

The multi-objective PSO, proposed by Lin et al. [12], was developed from single-objective PSO, such that traditional PSO can handle multi-objective optimization problems, with its ultimate objective of identifying the non-dominated solution set on the Pareto front. In order to spread Pareto front completely and avoid excessively similar solutions, the minimum number of dominated solutions is in the colonial optimal memory; thus, particles can search a region with few solutions. The maintenance strategy of 6 separate maintenance tasks at most means that this particle solves in a 12-dimensional search space. Figure 2 shows the composition of particles. The maintenance strategy is “8-V, 9-IV, 14-III, 25-II, and 34-I.” The first column of particles is the maintenance scheme; the second column is the maintenance interval time. The total line number is the maximum maintenance times. Each maintenance strategy can evaluate the fitness values of the five indices: economy (LCC); safety (average failure probability within service life); serviceability (average spalling probability within service life); rationality [12]; and maintenance times. The initial maintenance strategy particles are generated randomly, and maintenance strategies are in a uniform distribution of 0 – 7. Additionally, the maintenance interval time is in a uniform distribution of 0 – 10.

Figure 2. Maintenance strategy particles in this work.
5 CASE STUDY

The applicability of the proposed system, MDMS-RCS, is demonstrated by its identification of the optimal maintenance strategy for a four-story RC school building in Taipei, Northern Taiwan (for the frame structure). Additionally, we assume the surface chloride content on components is 1.5 kg/m$^3$ and 3.0 kg/m$^3$ to discuss the relationship between maintenance strategies and environmental conditions. Using deterioration analysis and seismic performance assessment, the time-dependent degradation in yielding base shear force and ductility of the first floor are assessed.

The LCCs for a specified service life of 100 years are 11.1 and 21.4 times initial construction cost (20,000 new Taiwanese dollars per the unit floor area), respectively. When LCC minimization is the objective to optimize in maintenance strategies, the optimal maintenance strategy reduces LCCs to 1.9 and 4.7 times initial construction cost, respectively.

When multi-objective optimization is applied to search for the optimal maintenance strategy, PSO and the Pareto optimal solution are used to generate the Pareto front. To select useful information for the owner or user from the Pareto front, this work sets a limiting value for LCCs of 1.1 times the minimal LCCs. Additionally, for the researched Pareto optimal solutions, referring to the best and worst fitness values of each objective, the fitness values of the LCCs (economy), failure probability of the building (safety), spalling probability (serviceability), rationality, and maintenance times of each maintenance strategy, are scored in the range of 0.0 – 1.0. For the maintenance strategy with highest total scores (Fig. 3) (LCCN: the score in LCCs; FP: the score in failure probability; SP: the score in spalling probability; TAV: the score in rationality value; MT: the score in maintenance times), when surface chloride content is 1.5 kg/m$^3$, repair and retrofitting strategies are mostly the Type-III Strategy; however, they are mostly the Type-V Strategy when surface chloride content is 3.0 kg/m$^3$. That is, when surface chloride content is relatively high, the outermost corroded reinforcing steel of members must be replaced, and chloride ions on members must be removed. However, according to simulation results, spalling probability of concrete cover is not likely to be markedly reduced under any maintenance strategy, as the selected repair technology has no improvement on finishing material. Therefore, this system will offer different finishing materials in the future for engineers to choose, thereby establishing a more effective maintenance strategy.

![Figure 3. Scores of the maintenance strategy corresponding to the maximum total scores.](image-url)
6 CONCLUSIONS

This work presents an integrated system, MDMS-RCB, that helps engineers search for the optimal maintenance strategy for deteriorating RC buildings via multi-objective optimization. This work applies PSO and the Pareto optimal solution to achieve the optimization of the multiple objectives of minimal LCCs (economy), minimal failure probability of the building (safety), minimal spalling probability (serviceability), maximum rationality, and minimal maintenance times. Additionally, to enhance computing efficiency in optimization, probabilistic effect assessment models for setting repair and retrofitting strategies are proposed. According to case study results, the optimal maintenance strategy with a definite and effective improvement can be sought. Put simply, the proposed system can be used to obtain the optimal lifecycle maintenance strategy, reducing seismic risk with respect to structural safety and serviceability of an RC building effectively, particularly in Taiwan or Japan.

REFERENCE

SUSTAINING A HISTORIC HIGH-RISE STRUCTURE

Nina A. Mahjoub P.E.(1), Megan Stringer P.E.(2) and Bill Tremayne S.E.(3)

(1) Holmes Culley, San Francisco CA – nmahjoub@holmesculley.com
(2) Holmes Culley, San Francisco CA – mstringer@holmesculley.com
(3) Holmes Culley, San Francisco CA – billt@holmesculley.com

Abstract
The most sustainable building is one that is already built. The safer building in an unforgiving seismic environment is one that has been retrofitted. Embracing both ideas, one of the tallest seismic retrofit in North America was undertaken in the heart of San Francisco. The Pacific Telephone & Telegraph Company headquarters opened in 1926, becoming the tallest building in the city upon completion. The building was an achievement of architecture and construction and remains an emblem of the Art-Deco movement. The building’s current owner decided to embark on a challenging endeavour of reviving the historic structure: from preservation of the historic fabric to creation of open flexible workspace, infusing state of the art technology and sustainability to all its aspects, including a voluntary full seismic structural upgrade.

Careful planning and collaboration between design and construction teams allowed for a smarter, less invasive, and sustainable renovation of this landmark structure.

The next generation must acknowledge the environmental impact of adapting these buildings to fit current cities’ demand, while keeping their historic identity. With advancing structural strategies, historic buildings no longer need to be viewed as obsolete, but as strong beacons of possibility in the marriage of preservation, innovation, and structural sustainability.

1 INTRODUCTION

Situated in the heart of Downtown San Francisco, the Pacific Telephone & Telegraph Company headquarters opened in 1925, reaching 435 feet and becoming the tallest building in the city upon completion. The building, now known as 140 New Montgomery (140NM), still stands as an icon of design and a reminiscence of the power of the latest technology of the time.

The building’s current owner since 2008, Wilson Meany, a real estate developer, decided to embark on a challenging endeavour of reviving the historic structure. While it will continue to host offices, the building will now introduce state of the art technology in all aspects, including a voluntary structural system upgrade, while maintaining the Architect, Timothy Pflueger’s, original intent. In addition to preserving the building’s historic features, Wilson Meany wanted to create a healthy, sustainable space for their tenants and targeted LEED Gold for the project.
This paper outlines the design goals of this upgrade: from the preservation of the historic fabric to the creation of open flexible office space, all while providing a safe and sustainable structure in San Francisco’s unforgiving seismic environment. It also discusses the strengthening scheme evaluated and challenges faced during the design. It presents details on the analysis method of the seismic retrofit which utilized a performance based design. This method presents the engineer with the capability to look past conservative building codes and determine in a more precise way the capacity of the existing building system. Moreover, this approach allows the engineer to better understand how the new and existing system behave together during a seismic event, and therefore provides a smart, more sustainable, and less obstructive solution while maintaining the historic fabric of the building.

Lastly, the paper discusses the environmental benefits of retrofitting versus rebuilding and how the sustainability objectives of the project shaped the design.

2 THE HISTORIC BUILDING

140NM is a 1925 neo-gothic structure with 26 stories, a four story tower above the 27th Floor and two basement levels, designed by Timothy Pflueger and Frank Miller. When completed, 140NM became the tallest building in the city, at 435ft, until its height was matched by the neighbouring Russ Building two years later.

The historic building housed the headquarters of the Pacific Telephone and Telegraph Company providing space for its 2000 employees. The building was known nationally and internationally in the business and design communities, with visiting VIPs such as Winston Churchill. In 1929, Churchill made one of the first trans-Atlantic phone calls from 140NM.

The building is classified by the City of San Francisco as a Category I Historic Building and is eligible to register for the National Register of Historic Places. Some of its historic features are the six acres of terra cotta façade constructed by the Gladding McBean Company and eight terra cotta eagles perched atop the tower. The entrance houses an ornate and dramatic lobby with detailed bronze doors, marble walls, and a hand-painted plaster ceiling by Mark Goodman, and leads to a marble staircase. presents the façade and lobby entrance.

Since completion, the building has remained relatively untouched, with a façade renovation in the eighties and parapet bracing installed just prior to the 1989 Loma Prieta earthquake. Comparing the building then to the building now, one can quickly notice that the historic fabric of the structure has stayed intact throughout the decades (see .)

After housing solely one company for over 80 years, the building was sold in 2008 to real estate developer, Wilson Meany. It was the refined character of the historic building that would set the tone for the project and the vision of its new and proud owner.

3 PROJECT VISION AND GOALS

An article in the San Francisco Newsweek dating back to 1925 described 140NM as “the new building generation, a monument to western progress and foresight.” Eight decades later, Wilson Meany set to continue this vision and honor its original inception as a modern communication hub and a centre of innovation. This building was going to continue housing the technology of tomorrow by attracting creative entrepreneurs and companies and more importantly by providing them with state-of-the-art technology infrastructure and flexible workspace – all within the historic high-rise. To achieve their vision, Wilson Meany engaged a
design team in 2011 who would spend the next few years following the guiding principles that
would restore and reinvigorate this iconic structure.

Some of the major work undertaken in this renovation have included the historic lobby
rehabilitation, elevator modernization (destination-controlled), and entirely new mechanical,
electric, and plumbing systems designed with tenant controllability. The majority of the
windows were replaced with high performance glazing ones and will remain operable to
promote natural ventilation. Some exterior restoration of the historic terra cotta and historic
glazed brick façade was undertaken, which included repointing of joints and
patching/repainting individual units as necessary.

But one of the most important components of 140NM’s refurbishment was the full
seismic retrofit. This retrofit was carefully designed and implemented to improve the
building’s safety and performance in the harsh seismic environment of San Francisco, while
preserving the building’s iconic historic fabric, including the exterior façade and grand lobby.

![Image: The Existing Building]

**Figure 1: The Existing Building**

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### 4 SEISMIC UPGRADE CRITERIA

#### 4.1 Existing Building Structural System

The existing building has an L-shape floor plan, measuring about 150 feet along New
Montgomery (the North-South direction) and about 140 feet along Minna Street (the East-
West direction). Typical floor to floor height is about 13ft. The structural system is made of a
grid of steel beams encased in concrete for fireproofing, connected to steel columns encased
in concrete at the interior floors and brick encased at the perimeter. The infill beams are
reinforced concrete and the floor consists of a 4 to 6” thick concrete reinforced slab. The
building steps throughout its height at the 19th, 23rd and Roof Level. The existing foundation
consists of concrete spread footings at each column and perimeter concrete walls.

The existing lateral structural system consists of steel beams and columns, classified as
“Partially Restrained Moment Frames”. The beams and girders, typically around the
perimeter, are connected to the columns via riveted clip-angle connections and, in some
instances, large gusset plates denoted on the existing drawings as wind braces.

#### 4.2 Performance Based Engineering

The requirement to assess and enhance the seismic safety of 140NM stemmed from two
main drivers: owner desired improvement and code-required triggers per the San Francisco
Building Code (SFBC). Given the building’s historic classification, the following code of reference was used: 2010 *California Historical Building Code* (CHBC), as permitted by the 2010 *San Francisco Building Code*. For the evaluation of the building’s seismic performance and retrofit design, ASCE 41-06, *Seismic Rehabilitation of Existing Buildings* (ASCE 41) was primarily used. ASCE 41 uses a Performance Based Engineering (PBE) approach. While traditional code prescribed approaches are often conservative, especially for existing buildings, PBE provides the framework to capture the inherent strength of the original structure.

Once seismic hazard and performance objectives were defined, a comprehensive finite element model of the building was developed. The analysis simulated how the building would perform during a large earthquake and how a seismic strengthening scheme could be most effectively designed to reduce damage and mitigate collapse hazards. This provided Wilson Meany with a better understanding of the value and risk associated with the building. The design team was able to develop a smarter and more efficient system, taking full advantage of the inherent strength of the existing building materials and overall retrofitted system.

### 4.3 Rehabilitation Objective & Design Criteria

In the case of 140NM, the global seismic rehabilitation objective for the building was to achieve the Life Safety Performance Level at 75 percent of the BSE-1 seismic hazard. This corresponds to a return period of 310 years with a 15% probability of exceedance in 50 years. This performance objective was consistent with the intent and requirements of the CHBC, which requires enforcing agencies to accept reasonable equivalent strategies to the regular code and permits the seismic forces used to evaluate the structure to be limited to 75 percent of those prescribed for new construction.

### 4.4 Analysis Procedure & Earthquake Ground Motions

In the vicinity of the project site, strong shaking can be expected from moderate to large earthquake. In particular, a moment magnitude 7.9 rupture on the nearby San Francisco Peninsula segment of the San Andreas Fault controls the seismic hazard adopted for this project. The Geotechnical Engineer, Treadwell & Rollo, developed the suite of earthquake time histories used for this project. Those earthquakes ranged from a magnitude of 6.9 to 7.9 with duration of 25 to 90 seconds.

For this project a Nonlinear Dynamic Procedure (NDP) was adopted for the analysis of 140NM.

### 4.5 Finite Element Model of the Existing Building

The analytical model was prepared using geometry obtained from a comprehensive set of existing drawings. Grades and strengths of materials used in the model were obtained from material testing. Testing included strengths of the steel (at column, beam, plate, angles, rivets, and reinforcing) and concrete. Being able to use expected strengths versus more conservative code prescribed strengths provided an increase in strength of, on average, 10%.

The model included the foundation, concrete floors, built-up steel columns, and the steel beams with the contribution of their concrete encasement. The partially restrained (PR) moment frame connections were explicitly modelled to evaluate their strength and flexibility. The contribution of the relatively stiff existing infill brick façade was also accounted for. The stiffer response resulted in the exterior frames attracting a greater proportion of the lateral load that
would be assumed from the bare steel PR frame alone, which consequently resulted in more severe demands on the existing steel columns.

After running the model of the existing building (pre-retrofit), it was concluded that the existing building performed poorly with excessive damage to the masonry façades and column failures due to high axial demands.

5 THE STRENGTHENING SCHEME

In order to select a seismic strengthening scheme, a number of criteria were established by which to measure overall performance: Life Safety, the proposed architectural layout of the renovated space, maximizing leasable square footage, minimizing impact on the existing historic building elements, constructability, LEED Gold objective, and overall project cost.

The brick and terracotta cladding system is inherently brittle. Therefore, the seismic strengthening system needed to be sufficiently stiff to limit the potential for damage to this façade under the specified seismic hazard.

5.1 New Seismic Load Resisting System (SLRS)

Based on the evaluation of a number of schemes with respect to the previously discussed selection criteria, a strengthening scheme utilizing new reinforced concrete core walls with structural steel outrigger trusses was chosen. Other considered schemes included propped steel plate shear walls, internal steel braced frames with buckling restrained braces (BRBs), and perimeter shotcrete overlay walls. The proposed scheme had the less impact on the existing building, the most efficient and innovative structurally, and cost effective.

The new reinforced concrete shear walls, with coupling beams, were located around the new stair and service cores extending from the basement level foundation to the underside of Level 27. The walls over the typical floors were connected to the existing adjacent steel columns to effectively provide a composite column, which strengthened the existing deficient elements. These new “core” elements provided not only a suitable location for new lateral load resisting elements, but also a functional separation between the tenant spaces and service areas.

The core walls were stiffened with the two-story deep outrigger trusses located below the 19th and 8th floors. They were located as high as practicable in the building without adversely impacting the useable floor space as the building façade steps at the 19th floor. The outrigger truss frame included one bay of diagonal Buckling Restrained Braces (BRB) which form a ductile fuse to absorb and dissipate the seismic energy in the form of heat. From Level 19 and below, new steel “super columns” were provided at the perimeter to transfer the load from each outrigger truss down to the foundation.

At the West and South wing facades, new reinforced concrete overlay walls were added. Those perimeter overlay walls were punched to maintain the existing window openings.

Error! Reference source not found. illustrates the new seismic load resisting system as implemented in the analytical model.

5.2 Combined Model

The new SLRS and existing structure, each described in the preceding sections, were combined in a single analytical model. Figure 2 illustrates the form of the combined model. This step of the analysis was crucial to verify the deformation compatibility of the two systems and ensuring they work effectively together during a large seismic event.
5.3 New Structural Lateral Resisting System

Upon running and analysing the new structural scheme only, it was concluded that the new SLRS was capable of resisting 100% of the seismic demands associated with the specified seismic hazard. Story drifts were generally low; less than 1.2% of the total height, except at the upper levels. Drifts at the North-west corner were also slightly higher.

5.4 Combined Systems

The combined model showed that the new SLRS added significant strength, stiffness, and ductility to the existing building while maintaining deformation compatibility with the existing lateral force resisting elements. This allowed the rehabilitated building to achieve Life Safety at the specified seismic hazard.

Story drifts were generally significantly less than 1% with larger drifts at the corners and towards the top of the building (above the upper outrigger trusses) with maximum drifts less than 1.5%. Additional strengthening of some existing components were identified, including reinforcement of the existing columns. The low drifts will provide added protection to the relatively brittle façade elements.

![Figure 2: New SLRS & Combined Building Model](image)

6 SUSTAINIBILITY

Sustainable design, in the context of the built environment, is one that meets the needs of the present without compromising the ability of future generations to meet their own needs. The “triple bottom line”, a phrase coined in the mid-1990s by John Elkington, is the model for evaluating sustainability that includes environmental, economic, and social performance measures. While many are starting to be concerned about environmental impacts of the built environment, a creative, effective, and truly sustainable design should consider all three.

6.1 Benefits of a Retrofitted Building

It is often said that the most sustainable building is the one already built. As the annual replacement rate of buildings has historically been around 2%, reusing and greening the existing building stock offers one of the greatest opportunities for reducing the environmental impacts of buildings. Reusing existing buildings requires creative problem solving, evaluation of the best use of the existing structure, and integrated design practices. It has dramatically lower impacts on the environment by requiring not only less new material and use of non-renewable
resources, but reducing the amount of waste produced and energy required for demolition and the rebuilding.

The retrofit scheme used for 140 NM allowed for the reuse of 95% of the existing structure. This has a great impact on the sustainability of the project and was achieved because of the owner’s objectives and through utilizing PBE. Preserving the building had more than just environmental benefits. 140NM is a historic building and preserving it is essential to maintaining the city’s heritage. Historic buildings are typically structurally robust and can be upgraded to meet current standards with typically minimal impacts to the environment. The significant reuse of the existing structure paired with the minimal addition of new materials to bring the existing building to Life Safety level provided not only a sustainable solution but a smart and necessary one.

6.2 LCA Analysis

Life-cycle assessment (LCA) is a technique used to account for all of the environmental impacts associated with a product or process over its life cycle with the goal of evaluating and reducing those impacts. LCA offers a powerful tool for evaluating the environmental impacts of buildings and strategically reducing them, considering all the building systems and all stages of a building’s life cycle. It enables comparison between different building component and system selections and offers a comprehensive way to evaluate and reduce the overall environmental impacts.

Looking at the retrofit from an LCA standpoint, the impacts from the retrofit versus construction of a new building were compared. Rough material quantities were estimated for a new building with equivalent floor area. Athena’s Impact Estimator was used for the LCA analysis. From the LCA, it can be seen that the retrofit has a little over a quarter of the environmental impact of constructing an equivalent new building, per Figure 3. The most common metric of an LCA is Global Warming Potential. This is a measure of the potential to increase in temperature the earth’s atmosphere and oceans.

![Figure 3: Comparison of Global Warming Potential by Assembly Groups](image)

6.3 LEED Certification

One of the more common ways to quantify sustainability is through the Leadership in Energy and Environmental Design (LEED) rating system developed by the U.S. Green Building Council (USGBC). The USGBC’s mission statement is “to transform the way buildings and communities are designed, built and operated, enabling an environmentally and socially
responsible, healthy, and prosperous environment that improves the quality of life.” 140NM achieved LEED Gold. The buildings received the maximum amount of credits available for building reuse including an Innovation in Design and Regional Priority credit.

CONCLUSION

Reviving 140NM entailed designing building system upgrades and insertions as elegant and contemporary elements that would complement the building’s historic features. It required delivering a robust, smart, integrated and nimble infrastructure and required a flexible architectural layout that could adapt over time. All those goals were achieved when the building reopened at the end of 2013 and houses now already more than a thousand people. Moreover, the seismic upgrade is expected to have increased the building’s lifespan by another fifty to a hundred years, allowing many more to experience the historic structure.

Future generations must acknowledge the significance of keeping these landmark buildings close to their original design while lending them a strong, efficient, and sustainable system. It must also recognize that with advancing structural analysis, historic buildings no longer need to be viewed as archaic, but rather as successful marriage of preservation and innovation.

REFERENCES

EFFECT OF CEMENT CONTENT ON CONCRETE DURABILITY

Amnon Katz (1), Arnon Bentur (2), R. Wasserman (3)

(1) Faculty of Civil and Environmental Engineering, Technion-Israel Institute of Technology, Haifa, Israel, akatz@technion.ac.il
(2) Faculty of Civil and Environmental Engineering, Technion-Israel Institute of Technology, Haifa, Israel, akatz@technion.ac.il
(3) National Building Research Institute, Technion-Israel Institute of Technology, Haifa, Israel, wasserman@technion.ac.il

Abstract

Requirement for minimum cement content exists in many international standards. This requirement varies according to environmental exposure where higher cement contents are required in environments that are more aggressive.

This work reports the study of the properties of concrete at varying water to cement (w/c) ratios of 0.45 to 0.70 and varying cement contents in each of them. Reducing cement contents was achieved by lowering the water demand while maintaining good workability with the aid of water reducing agents of different types at varying dosages. The properties of interest were those related to durability (carbonation, chlorides ingress, porosity etc.) in addition, of course, to strength.

It was found that strength, carbonation and shrinkage were related to w/c and independent of cement content. Capillary absorption and chloride ingress were reduced with reduction in w/c but also with reduction in cement content at a given w/c. These observations challenge the prevailing opinion presented in the standards regarding the requirement for minimum cement content.

1 INTRODUCTION

The cement industry accounts for a large share of the carbon footprint of the construction industry. About 0.8-1.0 ton CO₂ is emitted while producing 1 ton of cement clinker; therefore, the motivation for reducing cement content in concrete is obvious. A common approach towards reducing clinker contents is by using blended cements in which part of the clinker is replaced by pozzolanic materials [1-5]. Nonetheless, many standards set a minimum on the allowed cement content in concrete as function of the aggressiveness of the environment. For
example, the European standard EN 206 [6] requires a minimum cement content of 280 kg/m$^3$ for concrete exposed to carbonation in area with moderate humidity, and 340 kg/m$^3$ in areas exposed to wet/dry cycles of salty water. These requirements are in addition to a maximum water to cement (w/c) ratio of 0.55 and 0.40, respectively. On the other hand, in the USA the only requirement is for a maximum w/c ratio (ACI 318 [7]).

The requirement for a minimum cement content stems, probably, from the era when workability was achieved by changes in water content and the minimum set was intended to assure that the w/c ratio will not be too high. At present, modern admixtures technology allows better control of the water requirement for achieving a desired workability, up to a reduction of ~40% in the water demand and accordingly, a reduction in the cement contents.

The work present examines systematically the effect of cement content on the properties of concrete, mainly with respect to durability, while preserving the strength requirements.

2 EXPERIMENTAL PROGRAM

Different types of water reducing admixtures (WRA) were used to reduce the water demand for a constant workability: none (N), lignosulfonate (R700), sulfonated melamine (R2000) and polycarboxylate (G51). Concrete was produced in four w/c's: 0.45, 0.52, 0.60 and 0.70. Using the admixtures allowed producing the concrete at a constant workability (~150mm slump) at three water contents of ~205, ~180 and ~160 kg/m$^3$. Table 1 presents the water and cement contents of the mixes and the specific admixture used to obtain them. The specimens were tested as follows: Compressive strength was tested on 100mm cubes at various ages (only 28days is presented here); accelerated carbonation of 90 days old specimens exposed to 5% CO$_2$ at 50% R.H and 30°C; chlorides penetration according to ASTM C1202; capillary absorption according to EN 772-11; and shrinkage of 70x70x280mm prisms exposed to 50%RH/20°C.

Table 1: Water and cement contents of the mixes.

<table>
<thead>
<tr>
<th>Component</th>
<th>w/c=0.45</th>
<th>w/c=0.52</th>
<th>w/c=0.60</th>
<th>w/c=0.70</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water content, kg/m$^3$</td>
<td>202</td>
<td>186</td>
<td>167</td>
<td>201</td>
</tr>
<tr>
<td>Cement content, kg/m$^3$</td>
<td>450</td>
<td>413</td>
<td>372</td>
<td>386</td>
</tr>
<tr>
<td>Admixture type</td>
<td>R700</td>
<td>R2000</td>
<td>G51</td>
<td>N</td>
</tr>
</tbody>
</table>

3 RESULTS AND DISCUSSION

The relative strength at different w/c ratios is presented in Figure 1, calculated against the mix containing the maximum cement content in each of the w/c tested. Variation of ±10% was noted as the water (and cement) content were reduced without a noticeable trend. Such variation is reasonable between different mixes and it is hard to draw a concrete conclusion if any effect indeed exists.
Carbonation depth was measured at various ages and the rate of carbonation was determined vs. square root of time. Figure 2 presents the results from the accelerated carbonation test. A significant reduction in carbonation rate is seen as \( w/c \) reduces, from \( \sim 4 \) mm/\( \sqrt{\text{day}} \) for \( w/c \) of 0.70 to \( \sim 1 \) mm/\( \sqrt{\text{day}} \) for \( w/c \) of 0.45. It was found, however, that the penetration rate was not affected by reducing the cement content despite its reduction by \( \sim 20\% \).

Figure 1: Relative strength at different \( w/c \) ratios for mixes with different water contents.

Figure 2: Carbonation rate of the different mixes.

The results for the effect of cement content on volume changes of hardened concrete showed mixed trends (Figure 3). Reducing \( w/c \) or the cement content is known to reduce the shrinkage [8]; however, while the effect of lowering \( w/c \) was evident, the effect of cement
contents was not clear as can be seen in Figure 3. Another study on the effect of WRA on shrinkage of cement paste showed that the shrinkage increases significantly when WRA is used [9]. Since reducing the cement contents was obtained by using different types of admixtures at various dosages, it resulted in contradicting influences that annul each other.

Figure 3: Shrinkage results for mix with w/c=0.45 and varying cement contents.

Chlorides penetration tests showed significant reduction as w/c reduces or cement content reduces. The effect of reducing w/c is well known but the significant reduced penetration while reducing cement content is remarkable. Chlorides penetration of 1100-2300 coulombs was obtained at water to cement ratio of 0.45, which is a reasonable range for this low w/c. However, values as low as 1550 and 1640 were obtained at relatively high w/c of 0.52 and 0.60 at the lower cement contents tested. Similar results were observed for capillary absorption. Increasing cement content for a given w/c significantly increased the rate of capillary absorption.

It appears that two opposing mechanisms act simultaneously when lowering the cement content, with respect to durability: i. reduced permeability due to the reduced volume of the permeable phase (i.e. the cement paste); ii. increased binding capabilities of the paste. Penetration of CO\textsubscript{2} or chloride ions into the concrete is slowed due to binding of the species to the portlandite or to the aluminate phases [10], respectively. The amount of portlandite in the hardened cement paste is considerably high [8], thus, increasing the paste volume in the concrete increases simultaneously the permeability and the binding capacity, probably at the same rate. Binding capacity of chloride to the aluminate phases is limited thus the dominant mechanism is the increased permeability as paste volume increases, leading to significant increase in chloride penetration as paste volume increased.
SUMMARY AND CONCLUSIONS

The effect of cement content on concrete properties, strength and durability was studied by preparing and testing concretes at varying water to cement ratios (0.45-0.70) at different cement contents. The following trends were observed:

- **Strength** - The cement content hardly influences the compressive strength of the concrete tested in this study. The effect was in a range of ±10% without a clear trend. The major parameter influencing the compressive strength was the water to cement ratio.

- **Carbonation** - Similar effect was identified for carbonation rate. Increasing the volume of cement paste leads to more porous medium but at the same time to generation of more portlandite, which binds the CO$_2$ penetrated, leading to no effect of cement content.

- **Chlorides penetration** - significant increase in chloride penetration was identified as cement content increased at a constant w/c. Increasing the relative volume of the porous phase with only a limited binding capacity of chlorides leads to this finding.

- **Shrinkage** – generally, increased cement contents leads to increased shrinkage in concrete. However, water reducing agents that are used to reduce the cement content affect the capillary pressure in the pores, leading to adverse effect on the total shrinkage.

- **Capillary absorption** – increases as cement content increases, supporting the assumption that penetration increases as cement content increases.

These results indicate that reducing the cement content does not affect the mechanical properties of concrete and improves, or does not affect its durability. These findings challenge the prevailing opinion presented in the standards.
ACKNOWLEDGEMENTS

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