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Determination of hygrothermal properties of cementitious mortar: The effect of partial replacement of cement by incinerated sewage sludge ash

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Abstract

Two sewage sludge ashes were used as substitutes for cement and their effect on the hygrothermal properties of mortar was examined. Different cement to ash ratios and two ash pre-treatment methods (water washing and grinding) were in focus. The impact of cement replacement by sewage sludge ashes on thermal conductivity, sorption isotherms, water vapour permeability and carbonation was described with standard cement-based mortar as the reference material. Measurement results showed that thermal conductivity decreased by 15% when 30% of the cement was replaced by sewage sludge ash. Water vapour permeability increased as the cement to ash ratio was reduced. Sorption was tested by two methods; although differences in the sorption isotherms of mortars were reported when a climatic chamber method was applied, no differences, or only minor differences, were observed with the desiccator method. Measurements revealed that cement-based mortar possessed a higher content of carbonate than cement-ash-based mortar, and it thus appears that the carbonation rate was higher when the cement content was high.

Keywords

Introduction

Sewage is the collection of discharge from domestic, medical, commercial and industrial establishments with added rain water. Sewage sludge has been used in agriculture as a fertilizer, due to its high content of phosphorus. More recently, the tendency has been to incinerate sewage sludge (1). The organic matter is combusted and by-products such as CO_2, other trace gases and water vapour are produced. However, the process of waste disposal by incineration is not complete since a non-negligible amount of residue of incinerated Sewage Sludge Ash (SSA) remains afterwards. It is estimated that the global

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production of SSA is approximately 1.7 million tons annually, mainly from the USA, the EU and Japan, and this quantity is increasing (2,3). Most of the SSA has been landfilled (4–7), even though there have been some exploration of recycling and beneficial resource recovery (2,4,6,8–17). These investigations included the potential use of SSA as a supplementary material in blended cements (2,18), mortar (11,12,19), blocks (14,20) and bricks (21,22). The role of the SSA in mortar can either be as a cement substitute (similar to coal fly ash), as a binder when it possesses some degree of pozzolanic activity, or it may replace sand and thus act as a filler. Such replacement would not only preserve natural resources, but would also significantly contribute to a reduction of CO_2 emissions, and to increased energy conservation in the course of cement production and waste handling (23–25). These benefits will be substantial, as the cement industry makes 3.4 billion tons of cement annually, contributes 5 % of all CO_2-emissions caused by human activity (25) and accounts for 2 % of global energy consumption (23).

The present study was performed in the "ZeroWaste" development area at the Department of Civil Engineering of the Technical University of Denmark (DTU) (26), which was intended to develop new construction materials based on partial replacement of ordinary material constituents with secondary resources such as recycled materials. Characterization of two ashes and their effect on selected physical properties of mortar were described in a previous part of the study (27).. The aim of the present work is to comprehensively assess the effect of cement replacement by two specific SSAs at different replacement ratios and with two ash pre-treatment methods on the heat and moisture transport properties of mortar.

Materials and Methods

Material samples

Mortar mixture
SSAs from two different locations were used in this study as a partial replacement material for cement. One type of SSA originated from a wastewater treatment plant in "Avedøre", while the other was from "Lynetten". Both of these plants are located near Copenhagen, Denmark. The plants treat wastewater from 17 municipalities that together serve approx. 1 375 000 inhabitants.

As a reference material (REF), cement-based mortar samples (being mixtures of water, cement and sand) were prepared according to the standard (28) with the water-cement ratio kept constant at 1:2 for all mixtures. The same recipe was used for cement-ash-based mortars in which the cement content was partly replaced by the specific ashes, with mixtures and substitutions as shown in Table 1. After casting and curing for 24 hours in a moist atmosphere, the samples were demoulded and the mortar monoliths were cured for 28 days in water at a constant temperature of 20 ± 1 °C. The samples were then stored and dried in an oven at 45 °C.

Pre-treatment of ashes

Two different pre-treatment methods were applied to the ashes: grinding or water washing. As reported in (27,29), a simple ash pre-treatment can improve the compressive strength of the mortar.

Water washing of the SSA consisted of 3-washing cycles in distilled water. In a 1L-plastic bottle, 100 g of ash was mixed with 500 ml of distilled water and agitated for 1min. Afterwards, the bottle was placed in a vertical position until the SSA settled. When settled, water was decanted and the bottle was refilled with a new dose of distilled water. This procedure was repeated 3 times. During the last cycle, water and the SSA were decanted through a filter paper (11 μm). The wet ash was then dried in an oven at 105 °C for at least 24 h, and after cooling it was ready for use.

The other pre-treatment method was ash grinding. In this case, ash was ground for 30 seconds to achieve a finer particle size, using a Vibrating Cup Mill Pulverisette 9. After grinding, the ash was dried in the oven at 105 °C, and after cooling it was used in the mortar production.

Table 1   A matrix of mortar mixtures used in the study. The designations of the samples derive from their origin (Avedøre, AVE, or Lynetten, LYN), their substitution ratio (10 or 30) and the pre-treatment applied (Grinding, G, or Water-washing, W).

Name	Substitution ration [%]	Ash origin	Pre-treatment
REF	0	X	X
AVE 10G	10	AVE	G
AVE 10W	10	AVE	W
LYN 10G	10	LYN	G
LYN 10W	10	LYN	W
AVE 30G	30	AVE	G
AVE 30W	30	AVE	W
LYN 30G	30	LYN	G
LYN 30W	30	LYN	W

Experimental methods

Drying

The samples were dried either in an electrical oven or in a desiccator with silica gel. The samples used for sorption tests in the climatic chambers were exposed to different environments in order to determine their dry mass under various conditions. Prior to the sorption experiment, the samples were dried in an oven at 45 °C. Approximately 1.5 years after the sorption experiment, the samples were again dried, but now in various environments:

- in a sealed desiccator with silica gel at 20 °C,
- in a ventilated oven with silica gel inside at 45 °C,
- in a ventilated oven at 105 °C, and
- in a ventilated climatic chamber with a silica gel bag inside at 20 °C (in contrast to the desiccator method, CO_2 access was not limited).

Samples were not exposed initially to more than 45 °C, to avoid possible structural changes due to their exposure to elevated temperatures and to ensure that the material was exposed to conditions relevant for typical central-European indoor climates. Mass changes were captured by regular weighing using a Sartorius Research R 300 S microbalance with an accuracy of ±0.02 mg.

Thermal conductivity and volumetric heat capacity

The thermal conductivity and volumetric heat capacity were determined under laboratory conditions at an average temperature of 24 °C using a heat transfer analyser, ISOMET 2104. Measurements were performed with a surface probe with a disc sensor. The specific heat capacity c_p [J/(kg K)] was calculated by dividing the measured volumetric heat capacity C_p [J/(m³ K)] by the measured density ρ [kg/m³]. Dry and conditioned samples of dimensions 30 x 30 x 3 cm were examined at 5 surface points. The measuring accuracy was according to the producer: 10 % of the reading for thermal conductivity in the range 0.7 – 6.0 W/(m·K) and 15 % of the reading for volumetric heat capacity - around $1 \cdot 10^3$ J/(m³K).

Sorption and moisture fixation

The hygroscopic sorption properties were measured in accordance with the standard (30). Moisture storage in the hygroscopic range was determined by exposing the samples to various levels of relative humidity (RH). Once equilibrium had been reached, moisture content was determined by weighing. The standard specifies two alternative methods that can be used, i.e. the desiccator method and the climatic chamber method. In this study, both methods were applied and were compared. For both of the sorption tests, samples were crushed into pieces of a few mm (up to 5 mm) in maximum size and placed in glass cups (three cups per sample) or in inert textile bags (three bags per sample). All samples were dried in an oven at 45 °C prior to the experiment (RH in the oven was approximately 10 %). RH levels set in the climatic chamber were the following: 11 %, 30 %, 50 %, 70 %, 85 %, 93 % and 96 %; and in the desiccators it was: 33 %, 58 %, 75 %, 86 % and 94 %. In

the climatic chamber, only the adsorption isotherm was determined, so the RH level increased once equilibrium had been reached. In contrast, the samples in the glass desiccators were exposed to a high level of RH (100 %) first and RH levels were then progressively reduced in order to determine the desorption isotherms. The samples from the desiccators were then dried in an oven (45 °C) and subjected to moisture uptake in an adsorption process.

Water vapour permeability

The water vapour permeability was determined experimentally using the cup method to create a relative humidity gradient in such a way that a steady one dimensional transport across the samples was attained, as described in the standard (31). In dry cup tests, silica gel was used as a desiccant to achieve low RH close to 0 %, while Potassium nitrate (KNO_3) was used to generate a high relative humidity of 93 % $\pm$ 0.66 % RH in the wet cups. All the assemblies were placed in a temperature and humidity controlled chamber and exposed to constant conditions of temperature 23 $\pm$ 1 °C and relative humidity 53 $\pm$ 3 % RH. By regular weighing, the rate of water vapour transmission was determined. Based on the results determined from the first measurements (on 3-months old samples), the dry cup test was repeated a year after the first dry cup tests. The material parameters were expressed in terms of the water vapour permeance W [kg/(s Pa)] (Eq. 1) and water vapour permeability δ [kg/(m s Pa)] (Eq. 2). The resistance of the air layer was not considered since the air layer resistance is negligible compared to the resistance of the mortar (W = x10^{-12} kg/(s Pa) (air) and x10^{-10} kg/(s Pa) (mortar), δ = x10^{-10} kg/(m s Pa) (air) and x10^{-12} kg/(m s Pa) (mortar), respectively).

$$W = \frac{G}{A \cdot \Delta p_v} \quad (1)$$
$$\delta = W \cdot d \quad (2)$$

 - where G [kg/s] is the moisture mass change rate, A [m²] is the transmission area, Δp_v [Pa] is the vapour pressure difference over the sample and d [m] is the thickness of the sample.

Carbonation test

Dry mortar samples were ground into "flour-like" powder and immersed in a hydrochloric acid (HCl) solution. Calcium carbonate $CaCO_3$, a product of carbonation, reacted with hydrochloric acid to form calcium chloride ($CaCl_2$), carbon dioxide (CO_2) and water. The concentration of CO_2 was measured and based on that the content of $CaCO_3$ was calculated.

Table 2   Characterization of samples used in the carbonation test

Name	Description
New I	Freshly cast samples, cured for 28 days in water, then dried and tested
New II	The same batch as New I but 1 month older, dried in an oven at 45 °C

New III	The same batch as New I, dried in an oven at 45 °C, exposed to 50 % RH for 8 weeks before carbonation test took place

Results

Drying methods

The dry mass of the mortar specimens presented in Table 3 show that the most effective drying took place at the beginning of the experiment, immediately after the samples had been taken out of the water curing bath. None of the other methods achieved a lower dry mass compared to the initial drying.

Comparing the amount of moisture released in the given condition, cement-based mortar (REF) had the lowest ability to release the moisture compared to the mortar samples containing 70 % cement and 30 % SSA. The ability to release the moisture content increased with the ash to cement ratio. No effect of ash type or ash pre-treatment was observed.

Table 3   Average weight difference in [%] of three mortar specimens based on different drying methods compared to the initial drying in the oven at 45 °C(*), SD – standard deviation

Material	Oven 45 °C * [g]	Desiccator + silica gel [%]	SD [%]	Oven 45 °C + silica gel [%]	SD [%]	Oven 105 °C [%]	SD [%]	Climatic chamber + silica gel 4 % RH [%]	SD [%]
REF	17.5	4.6	0.29	4.2	0.01	3.0	0.25	3.2	0.32
AVE 30G	19.1	3.0	0.28	3.1	0.23	2.3	0.34	2.1	0.19
AVE 30W	21.2	3.0	0.05	3.2	0.05	2.5	0.09	2.4	0.18
LYN 30G	19.3	3.0	0.18	3.2	0.16	2.2	0.14	1.8	0.25

*Weight of mortar samples immediately after initial drying in an oven at 45 °C (drying performed immediately after 28-days curing in a water bath)

Thermal conductivity and specific heat capacity

Figure 1 shows the relation between the porosity of the mortar and its thermal conductivity. Mortar porosity was clearly influenced by ash content. A coarser ash texture than the fine cement resulted in a higher porosity in the mortar containing ash compared to the REF. The grain size distributions of cement and ashes, their chemical composition and their ability to hydrate, all have significant effects on mortar porosity. These findings were reported in (27).
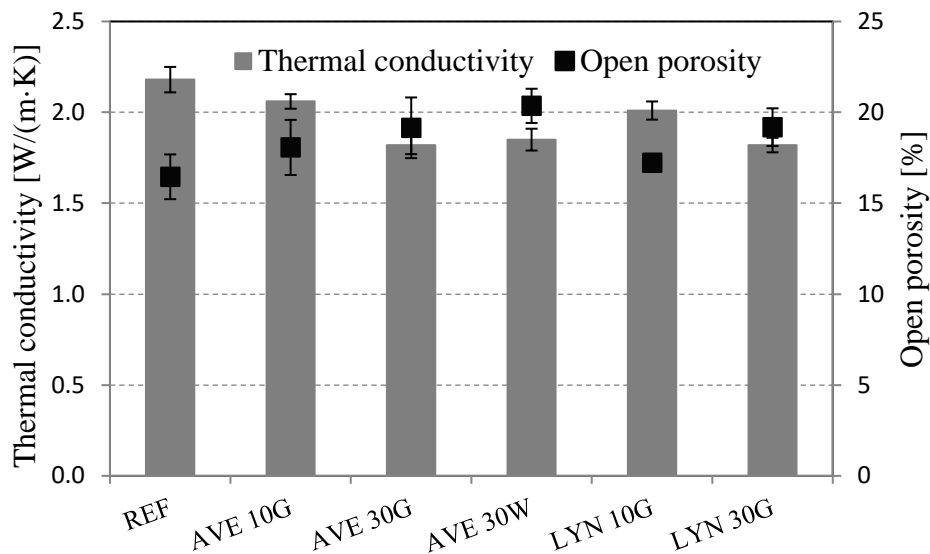

Figure 1   Average values and standard deviations of three duplicates for thermal conductivity and porosity, graphical relation

The thermal conductivity and specific heat capacity results are shown in Table 4. As expected, thermal conductivity increased with decreasing porosity as the insulating ability of air is greater than that of the solid constituents of mortar. Specifically, the reference material with 0 % ash has the highest ability to conduct heat compared to the mortar containing ash. Replacement of 30 % of the cement by ash led to an approximately 15 % decrease in the thermal conductivity, which may be an additional advantage for cement substitution. No significant differences in terms of these measurements were observed between the two ashes or between their pre-treatment methods.

Table 4   Average values of three duplicates for thermal conductivity λ and specific heat capacity c_p measured by the surface probe of a heat transfer analyser, SD – standard deviation

Material	λ [W/m·K]	SD	Diff [%]	C_p [J/kg·K]
REF	2.18	0.07	---	821
AVE 10G	2.06	0.04	-5.5	822
AVE 30G	1.82	0.05	-16.5	806
AVE 30W	1.85	0.06	-15.1	808
LYN 10G	2.01	0.05	-7.8	813
LYN 30G	1.82	0.04	-16.5	814

Sorption and analyses of moisture fixation

Equilibrium moisture content for specific mortar compositions was measured by two experimental setups: in a climatic chamber and in glass desiccators. Results obtained in tests are presented in this subsection.
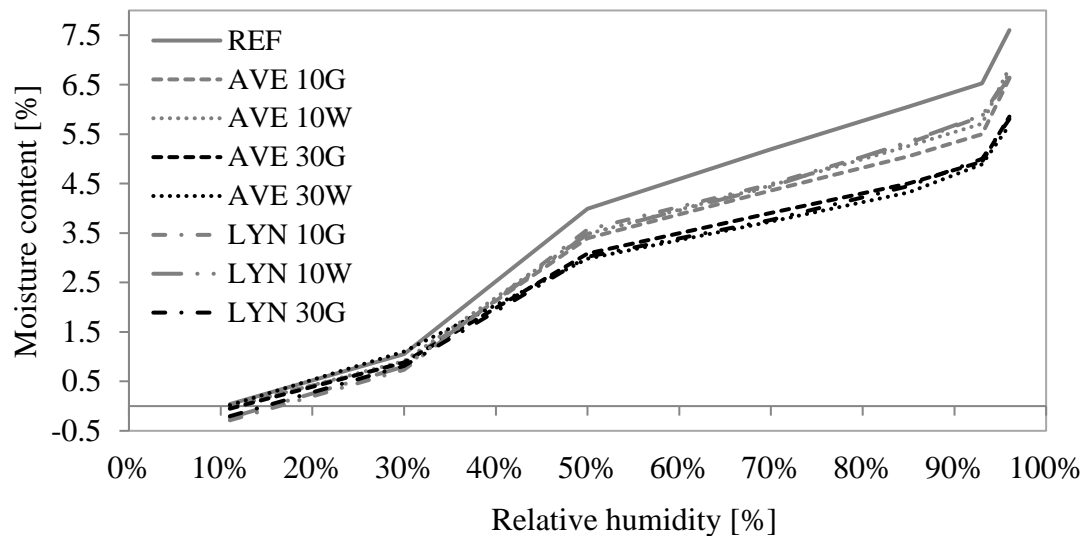

Figure 2   Sorption isotherms of the mortar samples performed in the climatic chamber based on average values of three duplicates

In Figure 2, exposure to low RH in the adsorption test in the climatic chamber, i.e. at 11 % RH, shows that the moisture content of the mortar remained unchanged or even slightly

decreased in all the samples compared to its state after initial drying in an oven at 45 °C. This indicates that the moisture content in the samples initially after drying was higher than or equal to the equilibrium moisture content at the first RH-value imposed in the climatic chamber during the sorption test, i.e. at 11% RH. The reference material, REF, without ash content is clearly characterized by having the highest moisture content at all RH levels compared to the specimens containing 10 % or 30 % of ash. This difference became more pronounced with increasing RH level. In general, for increasing ash to cement ratio, the moisture content became lower. No clear effects of the type of ash or the ash pre-treatment were found.
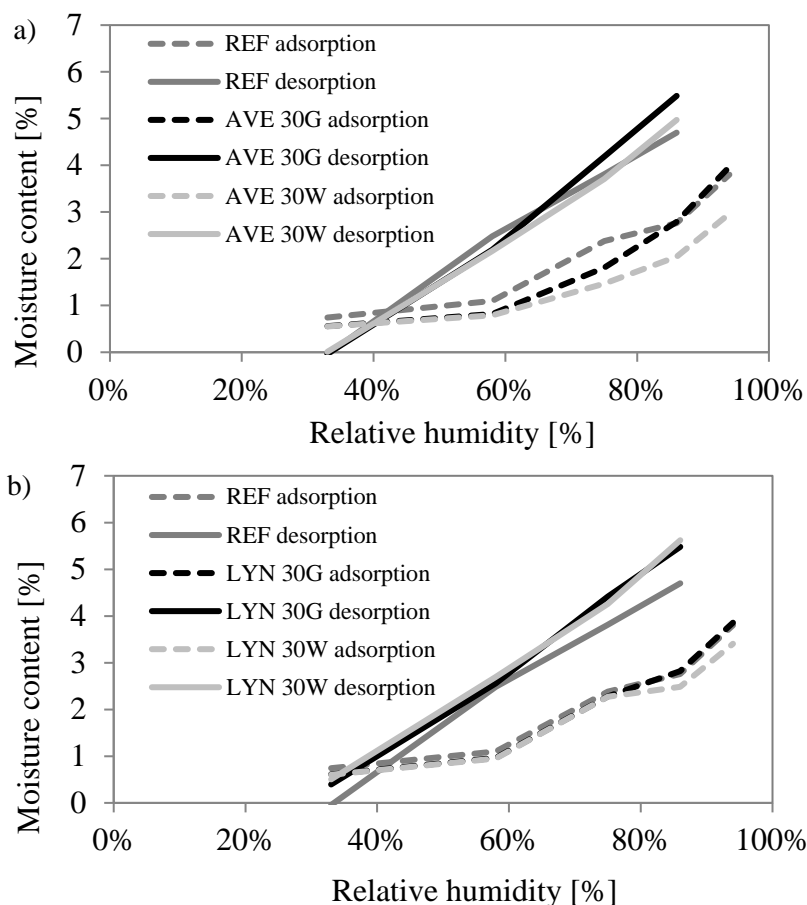

Figure 3   Sorption hysteresis of the mortar samples performed in the desiccators a) comparison of cement-based mortar (REF) and mortar with 30 % cement replacement by Avedøre ash (AVE); b) comparison of cement-based mortar (REF) and mortar with 30 % cement replacement by Lynetten ash (LYN).

Figure 3 shows the adsorption and desorption isotherms of hardened mortars, comparing REF mortar with mortar containing Avedøre or Lynetten ash as a partial cement replacement.. Comparing the different mortar compositions, the graphs show that the sorption or desorption curves coincide with each other over the greater part of the relative

humidity , although some differences can be observed at high humidity. Only small differences or no difference at all were found between the different ash pre-treatments. However, a slightly higher sorption ability is evident for mortar with AVE30G, which reached the level of cement-based mortar at RH levels above 80 %.

Water vapour permeability

Measurements of the water vapour permeability are shown in Fig. 4. Comparing the different mortar compositions, the water vapour permeability increased with the ash to cement ratio. Comparing pre-treatment methods, the results show that using ground ash with a finer ash structure kept the open porosity lower, so the permeability remained lower.

An analysis of results from the dry cup tests and its repetition one year later showed a significant decrease in the water vapour permeability with the age of the cementitious material. This may be due to the microstructural evolution of the cementitious material (e.g. porosity and tortuosity) resulting from the processes of hydration and carbonation.
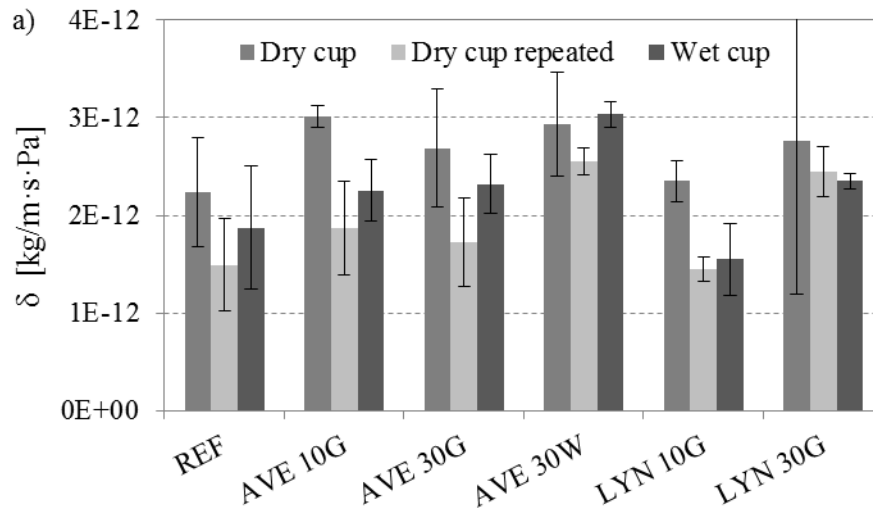
b)	Difference [%]		
	Dry cup	Dry cup repeated	Wet cup
REF	0.0	0.0	0.0
AVE 10G	34.6	25.7	20.3
AVE 30G	20.3	15.8	23.8
AVE 30W	31.3	70.9	62.0
LYN 10G	5.0	-2.8	-17.4
LYN 30G	23.5	64.2	25.4

Figure 4   a) Average values of three duplicates for water vapour permeability δ [kg/(m·s·Pa)] of the mortar samples and standard deviation (SD), b) percentage difference of samples containing ash compared to cement-based mortar (REF)

Carbonation test

Figure 5 shows the calcium carbonate content in newly-cast mortars that were prepared for the purpose of carbonation test and had never been used in other tests. The observations show that the initial calcium carbonate content in 'New I' varied between 12.2 – 13.1 % and thus its level was similar for all mortar samples regardless of their ash content.
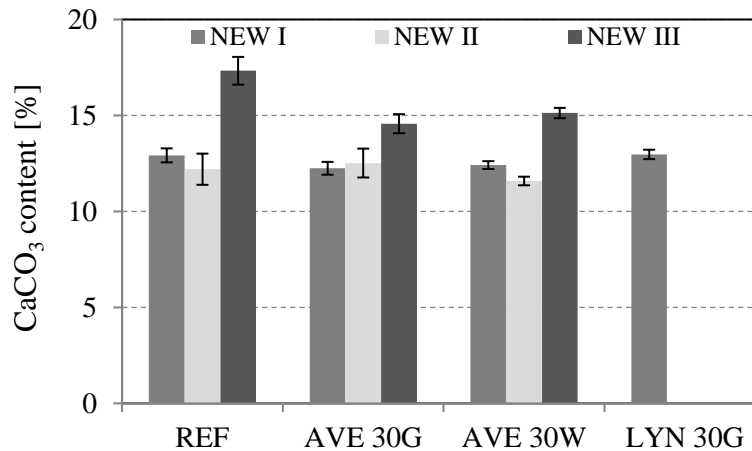
Figure 5   Results of the carbonation test shown as $CaCO_3$ content in mortar samples.

The content of calcium oxide (CaO) naturally occurring in cement and in the SSAs may potentially form $CaCO_3$ in reaction with CO_2 (Eq. 3), resulting in similar amounts of calcium carbonate ($CaCO_3$) content in mortar with and without ash (27). Comparison of 'New I' and 'New II' shows that drying in the oven slightly affected carbonate content. While exposure to 50 % RH for 8 weeks had a significant impact on mortar carbonation, the calcium carbonate content increased by 2.1 – 2.5 % steps in samples containing 30 % of Avedøre ash and by 5.1 % steps in cement-based mortar samples (REF). Separate carbonation tests indicate that this reaction takes place quite fast.
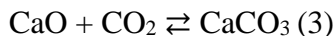
$$CaO + CO_2 \rightleftarrows CaCO_3 \text{ (3)}$$
    (reversible at high temperature above 825 °C) (32)

Discussion

The dry weight of samples is a central parameter for the determination of hygrothermal properties. Without knowledge of the dry mass it is impossible to define many parameters such as porosity, density, the moisture content, water vapour permeability, water retention, etc. However, in the case of cementitious materials, the dry state is not clearly defined. Cementitious materials naturally contain water that is either bonded in the structure in the form of a C-S-H gel and portlandite $Ca(OH)_2$, or which is physically present in the pores (33), so depending on the drying method, different amounts of water can be expelled. Some studies, e.g. (34,35), refer to the drying temperature set to 105 °C. Such an elevated temperature may however cause structural change to the material. For the purpose of the current study with its focus on the interaction of mortar with indoor climate, the drying temperature was initially set to 45 °C. Based on the observations, the most effective drying was achieved at the beginning of the experimental testing, just after the samples had been cured. Furthermore, the higher the temperature applied, the better drying results were observed.

Determination of basic material parameters, such as porosity, is an integral part of the material characterization and should not be omitted when reporting on the hygrothermal performance. The present study reports only on open porosity in relation to the thermal conductivity of mortar samples. More detailed results can be found in (27). The open porosity affects the following hygrothermal properties most: a) The thermal conductivity, which decreases as the open porosity increases. Pores filled with air serve as thermal insulation, and thus decrease the overall thermal conductivity of the mortar. In addition, the thermal conductivity of SSA has been observed to be lower than that of pure cement paste (36). b) Similar observations were made for the water vapour permeability, which was clearly higher for mortars with high ash content. Moreover, it may be concluded that grinding the ash before casting the mortar resulted in lower open porosity of the mortar, which have led to a significant decrease in the permeability of the material compared to the water-wash pre-treatment.

The adsorption and desorption isotherms are required to predict the hygroscopic water content in cementitious materials under changing climatic conditions. It has been shown that hysteresis between the adsorption and desorption isotherms of cementitious materials is significant (37), and so it was for the mortars analysed in the present paper. Sorption thermodynamics and the particular pore structure of cementitious materials determine the sorption behaviour. Since the pore structure changes during the hydration of cement (38), during the drying-process (39), and during chemical aging (40), it could be expected that it also affects the sorption properties. Determination of sorption isotherms that was performed in climatic chambers showed that the amount of moisture adsorbed was higher for the mortar samples with low porosity. The water vapour was adsorbed by the cement constituent and reacted with the CO_2 supplied to the climate chamber, together with moist air, to produce calcium carbonate, $CaCO_3$. Carbonation is a natural process that occurs in moist air with access to CO_2, and the highest carbonation rate occurs at 50 – 80 % RH (41). This reaction may result in a greater increase in mass change when comparing the two sorption test methods applied, i.e. the climatic chamber method (Fig. 2) and the glass desiccators (Fig. 3). For example, cement-based mortar reached 6.5 % mass gain due to carbonation, compared to dry mass, when measured with the climatic chamber method, and only 4 % mass gain when measured in a desiccator. The results obtained in the sorption tests performed in the climatic chambers corroborate the results on cement and ash sorption isotherms reported in (27). The sorption isotherm of cement was located significantly above the sorption isotherms of the ashes, so larger mass gains for cement were observed compared to that of any type of ash, regardless of which pre-treatment method was applied. This indicates that sorption ability may be affected by the potential reactivity of cement/ash with water, which is obviously higher for the cement.

In contrast, the sorption isotherms obtained in the tests in the glass desiccators show a slightly different adsorption ability, one that was dependent on the percentage of cement replacement. Isotherms obtained from desiccators (when the desorption test was performed before the adsorption test) coincided with each other independently of the mortar composition. Similar results were observed in (42) when cement-based mortar was

compared with mortar where a proportion of the cement (30 %) had been replaced with blast furnace slag. It was reported that the adsorption/desorption isotherms of samples with the same age showed only minor differences between these materials. However, it is not easy to assess the hygroscopic properties of cementitious materials, as in addition to the capillary effect between condensation and evaporation in the pores, according to the Kelvin equation, the chemical water uptake, the drying process, and chemical aging may affect the observed values.

Summarizing the results achieved by different sorption methods, the observed differences between the isotherms may have been caused by changes in the material structure, which occur as a result of the chemical aging and carbonation that take place in parallel with the weight gain by water vapour adsorption. Even though cement and ash show different sorption activity (27), replacement of cement by 30 % of SSA did not lead to any significant effect on the sorption ability of the final product, i.e. on the mortar. The aging of the material and its reactivity with adsorbed water and the carbon dioxide present in the climatic chamber caused differences to be observed between samples containing 0 % and 30 % of ash. The climatic chamber method was chosen in order to simulate most credibly the indoor environment, where CO_2 is present. It seems that experimentally this is the weakness of using the climatic chamber method for the determination of the sorption properties of cementitious materials. Detailed examination of the carbonation process in the material sample is difficult due to the various factors that affect carbonation rate and the methods used to examine it. The climate chamber method with open CO_2 access is therefore not recommended for assessing cementitious materials if it is required that the measurements should be unaffected by the CO_2 concentration, as was also recommended in (43,44).

An effect of the structural changes was also shown in the repeated permeability test in which the dry cup water vapour permeability test was repeated after 1 year. Except for the first dry cup test when samples had been newly cast and dried, the samples were stored for a year in a test chamber with a normal indoor environment and thus came into contact with moisture and CO_2. Comparing the first and the second dry cup test taken 1 year later, the measured permeability decreased for all the samples tested. This is in accordance with the theory of Pham and Prince (45) that small pores are clogged by calcium carbonate while the distribution of mesopores increases simultaneously.

In general, it can be concluded that partial replacement of cement by SSA has the effect on hygrothermal properties of mortar especially those properties directly related to mortar structure (porosity) and at higher cement replacement ratios (30 %). Cement replacement by up to 10 % can be a good compromise of slightly changed mortar properties and beneficial contribution to CO_2 reduction related to decreased cement consumption.

Conclusions

- The most effective drying was achieved with new samples of mortar prior to all experiments.
- Lower thermal conductivity and higher vapour permeability may be a result of higher mortar porosity due to the larger grain size of SSA compared to cement.
- The thermal conductivity of mortar was reduced by 15 % when 30 % of the cement was replaced by SSA.
- Sorption was influenced by the test method used. Differences between cement-based and cement-ash-based mortar could be seen when samples were exposed to air containing CO_2.
- The calcium carbonate content was shown to be higher for cement-based mortar compared to cement-ash-based mortar.
- Carbonation of mortar and its consequences must be considered not only in the process of data processing but already in the experimental procedure and time planning. The age of the samples may influence the parameters of cementitious materials considerably.

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