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Techniques and Optimum Size of Inclusions

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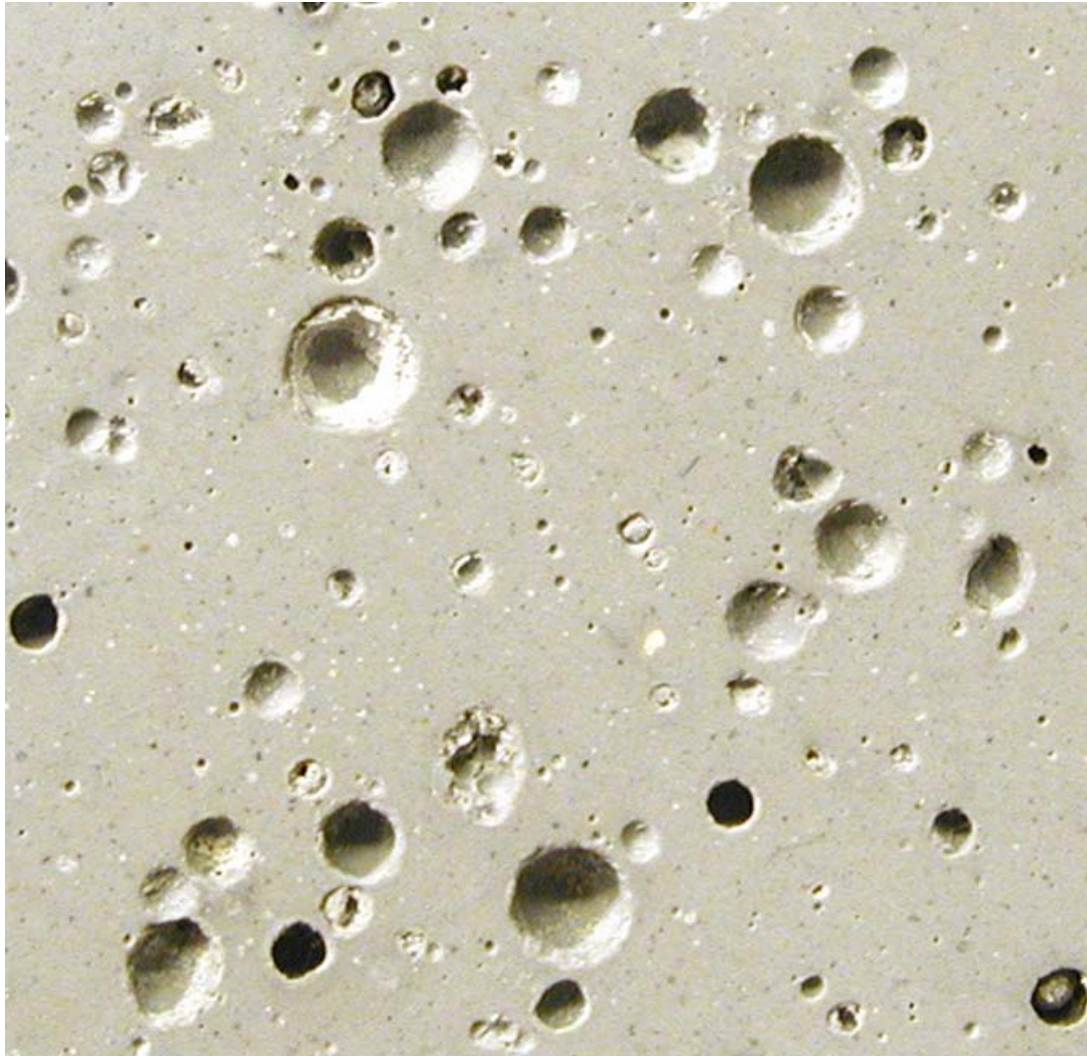
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Water entrainment in concrete

- techniques and optimum size of inclusions



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Introduction

This report gives a survey of different techniques for incorporation of designed, water-filled cavities in concrete: Water entrainment. Also an estimate of the optimum size of the water inclusions is given.

Water entrainment can be used to avoid self-desiccation and self-desiccation shrinkage during hydration [1,26]. What is needed is some sort of container which retains the shape of the water when mixed into the concrete. The container may function based on several different physical or chemical principles. Cells and gels are examples of containers found in nature. A cell membrane provides a boundary to water, whereas a polymer network incorporates water in its intersticious space with its affinity due to interaction energy and polymer entropy. Such containers allow water to be stored as an entity.

In relation to concrete the water encapsulation may be accomplished either before or after start of mixing. If encapsulated water is added to the concrete as particles they must be strong enough to withstand the mixing process. If the encapsulation takes place after start of mixing it must be finished before the setting. After the setting the entrained water must be accessible for the cement hydration, i.e. it is not allowed to be confined or bound. This means the water should have an activity close to 1.

Despite these partly conflicting demands several techniques may, potentially, be used. Water absorbing materials is a group of candidates for this purpose [27]. In the following some of these are presented. Special emphasis has been put on super absorbent polymers since these seem to be the most promising way to entrain water in concrete.

Methods for water entrainment in concrete

SUPER ABSORBENT POLYMERS

A super absorbent polymer, SAP, can be defined as a polymeric material which exhibits the ability to absorb a significant amount of liquid from the surroundings and to retain the liquid within its structure without dissolving [2,28], see Figure 1. SAPs are principally used for absorbing water and aqueous solutions. This type of SAP is also referred to as hydrogel. With the present polymer types the theoretical maximum water absorption is approx. 5000 times their own weight. This level has been reached by Japanese researchers with highly pure raw materials. With normal raw materials a water absorption of 2000 times can routinely be reached [9]. However, in dilute salt solutions such as urine, the absorbency of commercially produced SAPs is around 50 g/g.



Figure 1. Superabsorbent polymers are swellable substances which can absorb many times their own weight of liquids by forming a gel. The absorbed liquid is not released even under moderate pressure [28]. The picture shows a dry, collapsed and a swollen suspension polymerized SAP particle.

Most SAP are cross-linked polyelectrolytes. Because of their ionic nature and interconnected structure, they absorb large quantities of water and other aqueous solutions without dissolving, see Figure 2. Natural hydrogels can be produced from starch, however, these hydrogels may be rapidly decomposed by bacteria and fungi and they are for that reason of limited use. The commercial important synthetic polymers are covalently cross-linked polyacrylamides/polyacrylates [3]. These synthetic polymers have superior gel strength compared to starch based polymers [4]. Hydrogels are important elements in many biological systems, e.g. slug mucin. The synthetic hydrogels have found a widespread use as a high-tech material e.g. for contact lenses, breast implants, fire fighting, drug delivery, in baby diapers and as soil conditioner. Today's world production exceeds 500.000 tons per year of which about 85% is used for baby diapers [25].

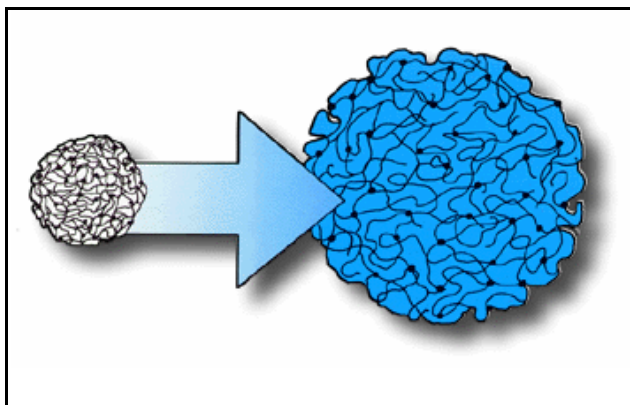


Figure 2. The cross-linking of the polymer chains prevents dissolving and retains the shape of the superabsorbent polymers during water uptake [5].

Polymer gels exist in two distinct phases, swollen and collapsed. The phase transition is a result of a competitive balance between repulsive forces that act to expand the polymer network and attractive forces that act to shrink the network. Several mechanisms are involved in this. One major effect is due to water solvation of the polymer chains.

Another major effect is due to that the macromolecular matrix of a hydrogel is typically a polyelectrolyte, i.e. a polymer with ionizable groups that can dissociate in solution, leaving ions of one sign bound to the chain and counterions in solution. For this reason, a high concentration of ions exists inside the gel leading to a water flow into the gel due to osmosis. Another factor contributing to increases in swelling is the interaction and repulsion of charges along the polymer chain. Intramolecular repulsion between charged segments will stretch out the macroion from a coiled to a more rod-like structure, and causes the macroions to arrange themselves so that they are as far from each other as is possible. Elastic free energy opposes swelling of the gel by a retractive force [6,7,8].

The volume transition of SAP occurs either continuously or discontinuously in response to chemical and physical stimuli such as temperature, solvent composition, pH, electric field and light. Especially the salinity of the exposure solution has importance for the swelling of hydrogels. The added salt changes the inter- and intramolecular interactions of the polyelectrolytes due to shielding of charges on the polymer chain. Furthermore, as the concentration of ions outside the gel increases, the osmotic pressure inside the gel will decrease, and the hydrogel de-swells [7]. Multivalent ions in the exposure solution are able to neutralize several charges inside the gel. The concentration of multivalent counterions inside the gel will, thus, be less than monovalent ions; this reduces the osmotic pressure and the degree of swelling [7]. Divalent cations such as Ca^{2+} or Mg^{2+} inhibit cross-linked polyacrylamide-polyacrylate absorbency very significantly. This is believed to be due to formation of ionic bridges between carboxyl groups in the gel, restricting polymer expansion [10,11]. In calcium rich solutions SAP containing sulfonate functional groups, rather than carboxylic acid groups, are preferred for this reason [3].

Information about the molecular nature of water within the hydrogel network has been obtained with several techniques including Nuclear Magnetic Resonance and Differential Scanning Calorimetry [2,12]. The water may be (a) polarized around charged groups, (b) oriented around hydrogen bonding groups or other dipoles, (c) structured in "ice-like" configurations around hydrophobic groups, and/or (d) imbibed in large pores as "normal" bulk water. These types of water may be summarized as "bound water" (type a and b), "interface water" (type c), and "bulk water" (type d). For a specific hydrogel it was found that about 0.2 g/g was bound water, 0.1 g/g interface water and the rest could be considered bulk water [12,13]. Essentially all the water inside a SAP can be considered bulk water.

The key properties of superabsorbent polymer are the swelling capacity and the elastic modulus of the swollen gel. Both these properties depend on the cross-link density of the network: Elastic modulus increases and the swelling capacity decreases with increasing cross-link density. Generally, the higher the water content of the gel, the poorer the mechanical properties of the gel become. The cross-link density of superabsorbent polymer used in diapers is about 0.05 mole of cross-linker per liter of dry polymer [3]. Approaches can be taken to minimize problems due to poor mechanical properties, the simplest of these approaches consists of forming the hydrogel over a strong polymeric mesh [2]. Other important properties include the extent of polymerization and completeness of cross-linking. The amount of free residual monomer is a measure of the extent of monomer conversion in the polymerization reaction, and the extractable polymer content indicates the completeness of cross-linking [3]. Residual monomer and unbound polymer and other extraneous materials can be extracted from the gel network if they may cause problems in the environment where the hydrogel is going to be used [2].

Commercially produced crosslinked acrylamide / acrylic acid copolymers cost from around 7 DM/kg or about DKK 2 per litre of absorbed water, assuming the water-uptake to be 14 kg/kg. This corresponds to about DKK 50 per m^3 concrete. Specially designed SAP are more expensive. The value for the water-uptake has been measured on specially produced spherical SAP particles in synthetic pore fluid, see Appendix A.

DIATOMACEOUS EARTH

Diatomaceous earth originates from the skeletons of a prehistoric single celled algae, diatoms, which synthesized shells for themselves out of silica. When the diatoms died the shells settled on the bottom of sea or lake beds and fossilized. The amorphous silica (opaline) content in diatomites ranges from 25 to 100 per cent. The diatom particles have an average size from 5-20 microns in diameter and, generally, exhibit either radial symmetry or line symmetry. The open porosity of the diatoms depends on the type, but may be around 50-70% leading to a water absorption of about 1 kg/kg, see Figure 3 [15,16,17].

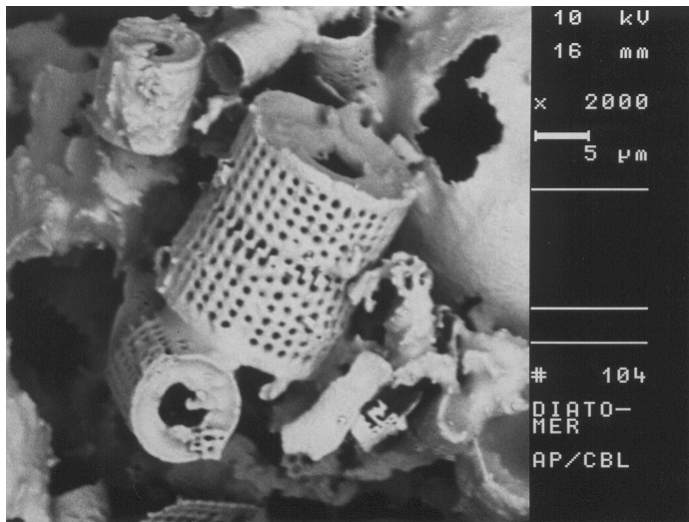


Figure 3. Scanning electron micrograph of pennate (i.e. line symmetric) diatoms. The open porosity of these particles has been measured to 68% [16].

Diatomaceous earth has multiple uses within e.g. filtration, drilling mud thickener, chromatography, as catalyst carrier and insecticide. Diatomaceous earth has been tested in relation to concrete technology. Diatoms are highly reactive towards lime owing to their high content of amorphous silica and high specific surface area. However, the use of diatoms in pozzolanic cements is hampered by their particle shape which has an unfavorable effect on the paste consistency and causes the water demand of cement to increase [18,19]. Diatomaceous earth has previously been suggested as a source of curing water [29].

Several aspects make it difficult to use diatomaceous earth for water encapsulation in concrete. As mentioned above diatomaceous earth has a very negative influence on paste consistency. A further complication may arise from cement and silica fume entering the cavity of the diatom particles during mixing of the concrete and, thus, off-setting the water entraining effect. In addition, the use of diatomaceous earth is unpractical since it has to be saturated before it is mixed into the concrete.

The price of diatomaceous earth varies considerably depending on the type and purity. A representative price for the cheaper types may be around \$0.4 per pound or about DKK 10 per litre of absorbed water [20].

LECA

Leca (Light Expanded Clay Aggregate) are ceramic particles with a dense shell surrounding a porous core. They are produced from plastic clay which during heating expands due to development of air from organic material in the clay. A number of other natural and artificially produced products have a physical structure similar to Leca, e.g. perlite and pumice.

The total porosity of Leca particles may be as high as 90%, however a large part of the pores of Leca particles are closed pores, and for that reason not useful for water encapsulation. The available amount of

entrained water may for example be 0.15 kg per kg of dry lightweight aggregate [30]. The price of leca is DKK 230 per m³, or about DKK 0.46 per kg dry aggregate. This corresponds to DKK 3 per litre of entrained water assuming the above value for water saturation.

The difficulties in using diatomaceous earth for water encapsulation in concrete as mentioned above also apply to Leca. In addition it may take a long time to water saturate the Leca particles, e.g. several days [30]. The technique of using lightweight aggregate particles for water entrainment has been known for some years [31].

MICROENCAPSULATION

Microencapsulation is a technique by which parcels of a gas, liquid, or solid are packaged inside tiny spheres of a second material. The capsules may be as small as 1 micron [21]. Many different capsule wall materials have been developed as well as techniques by which a substance might be encapsulated. The process may consist of dispersing fine droplets of the active material in an aqueous medium. The droplets are subsequently encapsulated by a polymer formed by a production technique called interfacial surface polycondensation, see Figure 4.

The core material is released from the microcapsule e.g. due to mechanical rupture of the capsule wall, dissolution of the wall, melting of the wall or by diffusion through the wall. Microencapsulation is used in many different products, examples are: Inks for carbon-free copy paper, chemicals for multi-component adhesives, taste masking of drugs, and pesticides. In relation to cementing materials microencapsulation of water has been previously used. By this method it was possible to prepare a dry mix of water-filled wax capsules and fast-setting grouting cement. The capsules were crushed and the mixture hardened when an anchor element in a one-step operation was inserted into the mixture. These micro-capsules consisted of 33% shell and had a size of 1-2 mm [22].

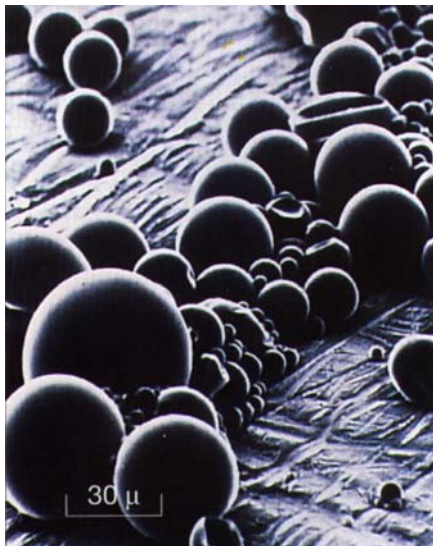


Figure 4. Scanning electron micrograph of a microencapsulated pesticide [23].

It has not been possible to obtain prices on microencapsulated products. However, it is believed to be a relatively expensive as well as delicate technique for encapsulation of water in concrete. The capsule wall has to be both chemically and physically strong enough to remain intact until setting. Thereafter it should allow the release of water. The water release may be based on diffusion through the capsule wall, but in that case it will not be possible to store the water-filled capsules with the cement.

EMULSIFIED WATER

A colloid is a dispersion of small particles of one material in another. If both materials are liquid the colloid is termed an emulsion. Many examples of emulsions both natural and artificial can be given, one being milk which is a dispersion of fats in water. An emulsion needs to be stabilized by an emulsifying agent which forms a protective film around the dispersed phase. In the case of milk this emulsifier is a protein, casein.

Within concrete technology a similar technique is used to improve the frost resistance of concrete. This is done by introducing 10-250 μm spherical air-voids into the fresh concrete which is enabled by an air-entraining agent. The air-entraining agent produces a foam by lowering the surface tension of the liquid and forming a stabilizing film in the liquid-air interface [24].

It may be possible to form water inclusions in a similar way, i.e. an emulsion of water in water. The emulsifying agent will have to be rather special since an interface between two different phases does not exist in that case.

It has not been possible to find out whether a technique like the one described exists.

Estimated optimum size of inclusions for water entrainment

In the considerations below focus is made on water entrainment with SAP particles. However, many points may be equally valid for water inclusions made by other techniques.

The size of the entrained SAP particles affects their performance in concrete. Several properties may be influenced by this such as:

- rheology of the fresh paste, i.e. mixing and placing,
- mechanical stability of the SAP particles during mixing, and
- leaching of extraneous substances from the SAP.

Most important, however, the ability of the SAP particles to reduce self-desiccation in concrete is also influenced by the particle size - the very reason why SAP may be added to concrete. To prevent self-desiccation a certain optimum size of the SAP particles is believed to exist.

If the SAP particles are too small the formed cavities may be partly filled with hydration products, thus offsetting their effect. Computer simulations suggest that hydration products may grow up to 50 μm into water from a free cement paste surface. The hydration products precipitated outside the original cement paste will be dominated by crystalline compounds such as calcium hydroxide and C_3AH_6 . In addition the porosity of the precipitated hydration products will increase rapidly from the original cement paste surface [32,33]. For this reason, the offsetting effect from precipitated hydration products may be limited to a few micro meter. SAP inclusions smaller than about 10 μm may for this reason be of very limited value with respect to reducing self-desiccation.

On the other hand, if the inclusions are too large they may not be able to supply every part of the cement paste with water during hydration. The maximum distance for the water transport in the paste is not known. A rough guide is given by measurements of chemical shrinkage [34]. From this, the maximum distance is believed to be less than 1 mm. Some further information is given by comparison with air-entrainment. Air-entrainment is used to improve the frost resistance of concrete. Typically, the pores needed to provide adequate frost resistance are 10-250 μm in diameter and the average maximum distance from any point in the paste to the nearest void is about 150 μm [24]. However, freezing is a fast process relative to hydration and the water transport to avoid self-desiccation is mainly required at an early age where the permeability of the paste is relatively high. On the other hand the pastes which exhibit self-desiccation have a very low permeability due to addition of silica fume and a low w/c-ratio. Computer simulations indicate that the water diffusion in a cement paste is effectively limited to distances of the order of 100 to 200 μm when the capillary pore space is depercolated [35]. Summing up, 150 μm may be considered an estimate for the maximum distance for water transport in these cement pastes.

Based on this value the maximum size of the water inclusions can be estimated. This is done below. In the calculations it is assumed that:

- all inclusions have the same size,
- the inclusions are cubic and periodically spaced in the concrete matrix, and
- water entrainment corresponding to an increase in the w/c of 0.05 is required [1].

A cement paste with w/c=0.3 and 20% silica fume addition is considered. Such a paste will have the following composition:

	Weight (g)	Density (g/cm ³)	Volume (cm ³)
Water	300	1.0	300
Cement	1000	3.1	323
Silica fume	200	2.2	91
Sum			714

In a pure cement paste at w/c=0.3 self-dessication can, theoretically, be avoided by water-entrainment corresponding to an increase in w/c of 0.05 [1]. This requires 50 g (=50 cm³) of water in the above recipe. Suppose that the added hydrogel consists of N equally sized, cubic water inclusions with an edge length, D. The total volume of these inclusions should then fulfil:

$$N \cdot D^3 = 50 \text{ cm}^3$$

If these particles are distributed periodically in the paste, the total paste volume around each particle (including its own volume) is:

$$(714 + 50)/N = 764/N \text{ cm}^3$$

The edge length of this paste volume, L, is:

$$L = \sqrt[3]{764/N} \approx \sqrt[3]{764/50} \cdot D \approx 2.5 \cdot D$$

The distance from the "corner" of the water inclusion to the "corner" of the protected paste volume – the maximum distance for water transport – will then be:

$$0.5 \cdot \sqrt{3} \cdot (L - D) = 0.5 \cdot \sqrt{3} \cdot (2.5 \cdot D - D) \approx 1.3 \cdot D$$

The maximum distance for water transport in the cement paste was estimated as 150 μm. If the water inclusions are at maximum approx. 100 μm in diameter this requirement will be fulfilled.

A specific type of SAP which has been tested takes up about 14 times its own weight of pore water, and has a dry particle density of 1.25 g/cm³. During water uptake the edge length of the dry SAP particle, d, is therefore increased by a factor of:

$$D/d \approx \sqrt[3]{14 \cdot 1.25} \approx 2.6$$

The optimum size of the dry SAP particles may, therefore, be around 40 μm. Figure 5 summarizes the above points.

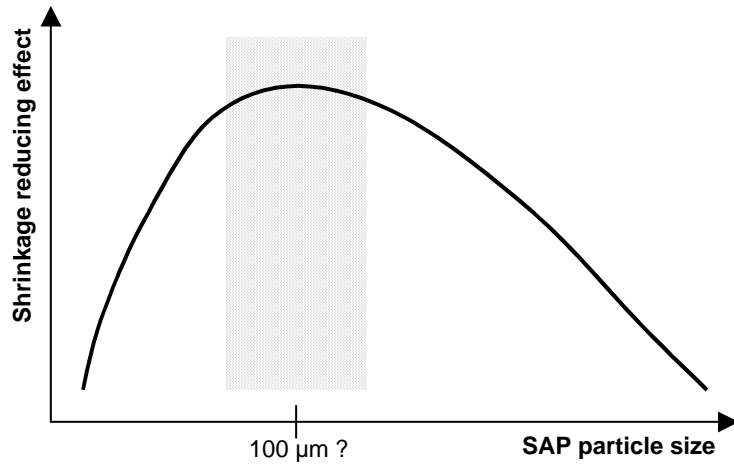


Figure 5. Suggested, schematic effect of SAP particle size on reduction of self-desiccation shrinkage. The suggested optimum size, about 100 μm , refers to the swollen state. In the dry state the optimum size is estimated to be around 40 μm .

Conclusions

Addition of super absorbent polymers, SAP, seems to be the most straightforward method to entrain water in concrete:

- The SAP particles can, conveniently, be used as a dry concrete admixture since they are able to rapidly absorb mix water.
- The water held by the SAP can be considered bulk water and is, thus, readily available for the hydration process after setting.
- The SAP particles can, potentially, be produced with a desired shape, e.g. spherical, and there are several potential options for modification of the SAP with respect to water uptake and release.
- The price of SAP is at an acceptable level, comparable to other concrete admixtures.
- The optimum size of the water inclusions is expected to be around 100 μm . This equals 40 μm for dry SAP particles with a water absorption of 14 ml/g.

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Appendix A

SYNTHETIC PORE FLUID

Expressed pore fluid from a cement paste may have the following composition [1]:

$[\text{Na}^+]: 400, [\text{K}^+]: 400, [\text{SO}_4^{2-}]: 40, [\text{Ca}^{2+}]: 20, [\text{OH}^-]: 760 \text{ mmol/l}$

These concentrations correspond to:

16.0 g/l NaOH	$(0.400 \text{ mol/l} \cdot 40.00 \text{ g/mol})$
7.0 g/l K_2SO_4	$(0.040 \text{ mol/l} \cdot 174.25 \text{ g/mol})$
18.0 g/l KOH	$((0.400 - 2 \cdot 0.040) \text{ mol/l} \cdot 56.11 \text{ g/mol})$
1.5 g/l $\text{Ca}(\text{OH})_2$	$(0.020 \text{ mol/l} \cdot 74.01 \text{ g/mol})$

The solubility product for CaSO_4 , $K_{\text{sp},\text{CaSO}_4} = 3.73 \cdot 10^{-5} (\text{mol/l})^2$ is not exceeded, contrary to the solubility product for $\text{Ca}(\text{OH})_2$, $K_{\text{sp},\text{Ca}(\text{OH})_2} = 7.88 \cdot 10^{-6} (\text{mol/l})^3$ [2]:

$$[\text{Ca}^{2+}] \cdot [\text{OH}^-]^2 = [\text{Ca}^{2+}] \cdot (0.400 + 0.400)^2 = 7.88 \cdot 10^{-6} \cdot [\text{Ca}^{2+}] = 0.012 \text{ mmol/l}$$

That means, only a minor part of the added $\text{Ca}(\text{OH})_2$ will dissolve.

If $\text{Ca}(\text{OH})_2$ were the only substance in solution the dissolved amount would be:

$$(7.88 \cdot 10^{-6})^{1/3} \cdot 74.01 = 1.47 \text{ g/l}$$

This corresponds to:

$$[\text{OH}^-] = 2 \cdot (7.88 \cdot 10^{-6})^{1/3} = 40 \text{ mmol/l}$$

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