

TOWARDS AN ADEQUATE DEICING SALT SCALING RESISTANCE

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Abstract

The deicing salt scaling resistance has been investigated for two types of concrete, i.e., air entrained high-volume fly ash (HVFA) concrete with a 50% cement replacement and non-air entrained concrete containing superabsorbent polymers (SAPs). A full characterization of their air void systems from the moment of casting until the freeze/thaw test was also done. Due to the presence of the highly AEA adsorptive fly ash an increased AEA dosage (7.0 ml/kg binder) was needed to achieve an adequate air void system in terms of air content and spacing factor to keep salt scaling within acceptable limits. For the novel non-air entrained concrete type with SAPs, which are able to absorb up to 500 times their weight in fluids, the salt scaling resistance is surprisingly high. The microstructural analysis revealed the formation of macro-pores due to these SAPs, creating an air void system as can be found in air-entrained concrete. Another advantage is that the strength of concrete with SAPs is much higher than for a conventional air-entrained concrete. This substantiates the further use of these SAPs as admixture in precast concrete road elements.

1. Introduction

Exposure to freeze/thaw cycles in combination with deicing salts can cause scaling or flaking of the concrete surface. Although most researchers agree upon the general definition of this damage form, there is still a lot of discussion about the underlying deterioration mechanism. Boel gives a good summarizing overview of the existing theories to explain the phenomenon [1]. In accordance with the theory of Powers salt scaling damage would be induced by occurring hydraulic and osmotic pressures in combination with gradients in salt concentration [2]. On the other hand, the theory of Hansen states that the damage is due to an oversaturated salt solution in the larger pores [3]. Furthermore, there is the theory of Snyder which is based on the variation in freezing point for the water in the concrete due to a salt concentration gradient [4]. More recently, the glue-spalling theory [5, 6, 7] has been introduced. There the

damage phenomenon is explained by the fact that the ice layer tends to contract much more than the concrete layer. The cracks in the ice layer penetrate into the underlying concrete and propagate according to a path parallel to the concrete surface [8].

Also note that there are some differences between concrete standards regarding the required minimum air content in the fresh state to achieve an acceptable salt scaling resistance. According to the European standard NBN EN 206-1, concrete exposed to freeze/thaw in combination with deicing salts should be air entrained with a minimum air content of 4% in the fresh state. However, according to the Belgian standard NBN B 15-001 artificial air entrainment is not mandatory and it should relate to the maximum aggregate size. For a maximum nominal aggregate size of 16 mm, the standard specifies an air content of at least 5%. The American standard ACI 201.2R also takes into account the higher air requirements of concrete mixtures with higher paste contents due to smaller nominal maximum aggregate sizes. However, also the severity of the exposure is of importance. A distinction is made between moderate and severe exposures. Exposure conditions are considered to be severe whenever deicing salts are present. In such an environment, an air content of 6 to 7% is recommended for a maximum nominal aggregate size of 16 mm; 5 to 5.5% of air is only sufficient in the case of moderate exposure (without deicing salts) for the same aggregate size. Nevertheless, the recommendations of ACI 201.2R are not binding. Local conditions and experience with specific mixtures and procedures could still warrant other values.

Both the ambiguity regarding the precise deterioration mechanism and the existence of different criteria in terms of air content make it not evident to properly design novel concrete compositions, such as High-Volume Fly Ash (HVFA) concrete and concrete containing superabsorbent polymers (SAPs), and make them resistant to salt scaling.

With respect to HVFA concrete, a potentially sustainable concrete type in which at least 50% of the ordinary Portland cement is replaced with pozzolanic fly ash, Malhotra and Mehta reported an adequate salt scaling resistance of HVFA concrete pavements in Wisconsin (US) and sidewalk sections in Halifax (Canada) [9]. On the other hand, they also reported higher deterioration rates for the same material under laboratory conditions. Since the applicable European standards mainly focus on performance criteria for concrete that has been exposed to accelerated laboratory testing conditions, it is quite logic that many concrete manufacturers remain for the moment sceptical about using this HVFA concrete on a larger scale. Therefore, more research on that matter is still needed.

SAPs are a new kind of material to be used in building applications. They have the ability to absorb a significant amount of liquid from the environment (up to 500 times their own weight) and to retain it without dissolving. In concrete they are used to decrease the autogenous shrinkage and self-desiccation due to internal curing [10] and to promote self-sealing and self-healing [11, 12]. Due to their swelling capacity SAPs can also be used to change the pore structure. They can extract water of the fresh concrete mixture causing the stiffening of the paste which is accompanied by a reduction of the capillary porosity. Cavities will hereby remain as empty pores afterwards. SAP particles introduce a system of fine, evenly distributed air voids after release of their absorbed water. SAPs can thus also function as air-pore entraining agents, increasing the freeze-thaw resistance and the durability [13, 14]. In this research, a HVFA concrete was developed by carefully controlling the air content in fresh and hardened state with appropriate dosages of air entraining agent. Moreover, the effects of SAPs on concrete's air content and air void system were examined. Also, the influences on the strength and freeze-thaw resistance were evaluated and compared.

2. Materials and Methods

2.1 Concrete mixtures

Regarding the research on HVFA concrete, six concrete mixtures were manufactured (Table 1). Mix T(0.45) is a non-air entrained ordinary Portland cement (OPC) concrete with the required minimum cement content (340 kg/m³) and the maximum water-to-cement ratio (W/C: 0.45) for freeze/thaw environments according to NBN B15-001. An air entrained version of the reference concrete mix (T(0.45)A) was manufactured as well. The incorporation of a fatty acid/polyglycol based air entraining agent (dry matter mass percentage: 4%) is indicated within the concrete mix name by means of the letter 'A'. Regarding the inert fraction per m³ of mixtures T(0.45) and T(0.45)A, the concrete contained 715 kg sand 0/4, 515 kg gravel 2/8 and 671 kg gravel 8/16.

Table 1. Studied HVFA and corresponding reference concrete mixtures with nomenclature.

	T(0.45)	T(0.45)A	F(1)50	F(1)50A	F(2)50	F(2)50A
W/C or W/B	0.45	0.45	0.35	0.35	0.35	0.35
AEA		x		x		x

Since a traditional concrete for freeze/thaw environments does not necessarily have to be air entrained according to NBN B15-001, both non-air entrained HVFA mixes (F(1)50 and F(2)50) and air entrained HVFA mixes (F(1)50A and F(2)50A) were evaluated in this research. The mixing time for each mix was 5 min: 1 min of dry mixing, 2 min of mixing after adding the mixing water and another 2 min of mixing after adding the polycarboxylic ether-based superplasticizer (SP) (dry matter mass percentage: 35%). In case of air entrainment, the AEA was added to the mixing water before being added to the binder, sand and aggregates. The AEA dosage was fine-tuned experimentally with the aim of achieving an air content in the fresh state of 6-7% cf. ACI 201.2R. Each HVFA composition was made twice: once with a fine fly ash F(1) (45 µm fineness: 13.2% retained) and once with a coarser fly ash F(2) (45 µm fineness: 26.6% retained). Note that all HVFA mixtures were characterized by a higher binder content B (cement + fly ash: 450 kg/m³) and a lower water-to-binder ratio (W/B: 0.35) compared to the references T(0.45) and T(0.45)A. The higher binder content and lower W/B ratio also explain the different proportioning of the inert fraction per m³ (sand 0/4: 645 kg/m³, gravel 2/8: 465 kg/m³, gravel 8/16: 606 kg/m³).

The basic composition used in the SAP concrete investigation is based on ordinary concrete in road construction with a water-to-cement ratio of 0.45; 350 kg/m³ CEM I 42.5 N, 157.5 kg/m³ water, 690 kg/m³ quartz sand 0/4, 188 kg/m³ gravel 2/8, 1003 kg/m³ gravel 8/16 and an amount of superplasticizer. The concrete was made in the framework of a round-robin test of the technical committee 225-SAP from RILEM. As a superplasticizer, a commercial product based on β-naphthalene sulphonate (BNS) was used (Woerment FM30/BV30 from BASF; 1.2 g/cm³). All concrete mixtures showed a consistency class F3 (soft) as specified in EN 1045-2. Bulk-polymerized SAPs (two types) were added on top at 0.15 m% of cement weight. Both SAPs are able to take up approximately 300 g demineralized water and 33.3 g mixing water per g SAP. Their sizes are 180 ± 45 µm (SAPa) and 70 ± 21 µm (SAPb). The amount of additional water was 17.5 kg/m³ to receive a total water-to-cement ratio of 0.50. To retain consistency class F3, the dosage of superplasticizer was increased. Additionally, mixtures with SAPs but without additional water and a mixture with the additional water on top but

without SAPs were made. A comparative reference incorporating a conventional air-entraining agent (AEA) was used as well. The air-entraining agent was a commercial product (LP75 from BASF) and was simply added on top by first homogeneously mixing it with the mixing water. The amount of air entrainer was 0.025 m% (by weight of cement). The different mixtures are shown in Table 2.

Table 2: Studied SAP and corresponding reference concrete mixtures with nomenclature.

	REF I	REF II	SAP 1	SAP 2	SAP 3	SAP 4	AEA
$(W/C)_{tot}$	0.45	0.50	0.45	0.45	0.50	0.50	0.50
$(W/C)_{add}$	-	-	0.05	0.05	0.05	0.05	-
$(W/C)_{eff}$	0.45	0.50	0.40	0.40	0.45	0.45	0.50
SAPa			x		x		
SAPb				x		x	
AEA							x

REF I and REF II have water-to-cement ratios of 0.45 and 0.50, respectively. SAP1/2 is a SAP3/4 mixture without additional water, containing SAPs. AEA is a mixture containing the air entraining agent. The total, additional and effective water-to-cement ratios are given for comparative reasons. The mixing procedure started with a 2 min homogenization of the dry powders and the dry SAPs in the respective mixtures, at low speed (Rotating pan mixer Zyklus 50 l). In the following 30 s, water (and air-entrained for the AEA mixture) was added. The whole was mixed for 1 minute and the superplasticizer was added in the next 15 s, followed with an additional 2 min of mixing at high speed.

2.2 Air content measurements in the fresh and hardened state

The air content in the fresh state was determined using the Standard EN 12350-7 with the pressure gauge method. A known volume of air at a known pressure is hereby merged in a sealed container with the unknown volume of air in the concrete sample. The dial of the pressure gauge gives the percentage of air for the resulting pressure.

The observed microstructures in hardened state were investigated by means of air void analysis based on the Standard EN 480-11. Two specimens (100 × 150 mm² area) were hereby cut perpendicular to the longitudinal axis and the surface was then ground and polished to produce a smooth flat surface finish. The specimen surface was treated to produce a better contrast between the air voids and the cementitious matrix. First, black ink was applied on the surface. Then, the surface was covered with fine barium sulphate powder which was pressed in the air voids. The excess white powder was then removed and the specimens could be microscopically studied. The air void structure was hereby examined by scanning along a series of traverse lines parallel to each other. The air voids intersected by the traverse lines were recorded and the total porosity could be determined.

2.3 Mechanical properties and salt-scaling test

The compressive strength of the materials was tested according to the Standard EN 12390-3 and the test to determine the freeze-thaw resistance was the slab test according to the Standard CEN/TS 12390-9 (Slab test) on cylindrical specimens of 100 mm in diameter and 80 mm high.

3. Results and discussion

3.1 Fresh state properties and air void system in hardened state

The use of fly ash in concrete has an impact on the required AEA dosage (Table 3). For the reference T(0.45)A, a dosage of 2.0 ml/kg binder was sufficient to have an air content (6.8%) conforming to all standards. With respect to the air entrained HVFA mixtures F(1)50A and F(2)50A, a much higher AEA dosage (5.0 ml/kg binder) resulted in an air content that was considerably lower than the 6.8% of the reference: 4.9% for mix F(1)50A and 5.2% for mix F(2)50A. As a consequence, the air content criterion imposed by ACI 201.2R was not met.

Table 3. AEA and SP dosage [ml/kg B] and fresh concrete air content [%] for HVFA and the corresponding reference concretes.

	T(0.45)	T(0.45)A	F(1)50	F(1)50A(-2)	F(2)50	F(2)50A(-2)
AEA (ml/kg B)	-	2.0	-	5.0 (7.0)	-	5.0 (7.0)
SP (ml/kg B)	2.0	4.0	7.0	7.0 (4.0)	5.0	5.0 (4.0)
Air content (%)	2.8	6.8	2.6	5.2 (6.9)	2.8	4.9 (7.3)

Two additional air entrained HVFA mixes with an increased AEA dosage (7.0 ml/kg B instead of 5.0 ml/kg B) were made: F(1)50A-2 and F(2)50A-2. Their fresh air contents amounted to 6.9% for mix F(1)50A-2 and 7.3% for mix F(2)50A-2, respectively.

Within the framework of the SAP concrete research, the fresh concrete air content was approximately 3% for all mixtures made (Table 4). The AEA mixture shows 6-7% of air content, needed for an adequate salt scaling resistance.

Table 4: Fresh concrete air content [%] for SAP and the corresponding reference concrete.

	REF I	REF II	SAP 1	SAP 2	SAP 3	SAP 4	AEA
Air content [%]	3.0	2.3	3.3	3.5	3.7	3.3	6.6

The air content and spacing factor in hardened state are important parameters to be considered when studying the salt scaling resistance. For the research related to the HVFA concrete, the obtained automated air-void analysis results are shown in Table 5. Compared to T(0.45), the air entrained reference T(0.45)A was characterized by a much higher air content (7.5-8.0%). Note that the higher values for the T(0.45)A reference mix are somewhat different from its initial air content (6.8%, Table 3) in the fresh state. Regarding the HVFA compositions, air entrainment resulted in an increase of the air content, though the values obtained were lower than 6-7%. Compared to F(1)50 and F(2)50, the average hardened air contents of F(1)50A and F(2)50A were increased by only 1.8% and 1.5%, respectively. The air contents in hardened state were more or less in accordance with the measured air contents in the fresh state (F(1)50A: 4.9%, F(2)50A: 5.2%).

Not only a sufficient total air content is of importance, but also an adequate distribution of the artificial air bubbles. In other words, their spacing should be close enough to prevent the development of pressures from freezing which would fracture the concrete. The fulfilment of this requirement is usually evaluated through the calculation of a spacing factor for the concrete. This is the maximum distance from any point within the concrete matrix to the edge of the nearest air bubble. According to NBN B15-001 and ACI 201.2R, a spacing factor of

200 μm is recommended as the maximum for concrete exposed to freeze/thaw attack. The spacing factors of all air entrained concrete compositions (T(0.45)A, F(1)50A, F(2)50A) do not exceed this maximum value, although the HVFA mixes are characterized by somewhat higher values (100-200 μm) when compared with the OPC reference ($\pm 100 \mu\text{m}$). Without air entrainment, the spacing factors of T(0.45), F(1)50 and F(2)50 range between 250 and 300 μm .

Table 5: Air content and spacing factor of HVFA and the corresponding reference concrete after automated air-void analysis.

	T(0.45)	T(0.45)A	F(1)50	F(1)50A(-2)	F(2)50	F(2)50A(-2)
Air content [%]	2.7 \pm 0.6	8.0 \pm 0.4	3.5 \pm 0.6	5.2 \pm 0.9 (7.3 \pm 0.7)	3.0 \pm 0.7	4.6 \pm 0.6 (7.9 \pm 0.2)
Spacing factor [μm]	291 \pm 36	86 \pm 3	282 \pm 71	119 \pm 12 (82 \pm 8)	252 \pm 39	130 \pm 21 (77 \pm 1)

Increasing the AEA dosage from 5.0 ml/kg B to 7.0 ml/kg B, resulted in a more adequate air void system in hardened state for HVFA concrete. The total air contents measured for F(1)50A-2 and F(2)50A-2 slightly exceeded the 6-7% criterion recommended by the ACI 201.2R guideline. Spacing factors decreased to values around 100 μm cf. T(0.45)A.

All specimens examined in view of obtaining a salt scaling resistant SAP concrete showed an air content of 5.5-11.2 % and a spacing factor of 91-277 μm (Table 6).

Table 6: Air content and spacing factor of HVFA and the corresponding reference concrete after automated air-void analysis.

	REF I	REF II	SAP 1	SAP 2	SAP 3	SAP 4	AEA
Air content [%]	9.4 \pm 0.8	8.9 \pm 1.1	5.5 \pm 0.7	6.5 \pm 1.6	10.0 \pm 0.4	10.1 \pm 0.3	11.2 \pm 1.9
Spacing factor [μm]	97 \pm 4	143 \pm 3	277 \pm 21	272 \pm 48	162 \pm 11	91 \pm 1	115 \pm 8

The cumulative air content for all studied mixtures is given in Figure 1 (average of two 100 \times 150 mm² samples). If one would start from the initial size of the superabsorbent polymers and the amount of additional water (33.3 g mixing water/g SAP for both SAPs with a SAP bulk density of 700 kg/m³), one could calculate the final swollen size of the SAP particles in the concrete. SAPa with an initial size of 180 \pm 45 μm attains a swollen size of 670 \pm 167 μm . For SAPb, this is 70 \pm 21 and 260 \pm 78 μm , respectively. These values correspond with the results of the automated air void analysis. The AEA mixture shows typical additional pores with pore sizes of approximately 250 μm . The fact that SAP macro pores are fluid-filled upon the determination of the air content in the fresh state and empty in the hardened state, explains the higher values found in Table 6 compared to Table 4.

One can calculate the total number of SAPs in the concrete, based on the m% of cement weight, the initial dry size and the bulk density of the SAPs. Assuming a uniform distribution (primitive cubic order arrangement) of the SAPs, the spacing between the centres of the SAPs can be compared. If one subtracts the found swollen sizes, an estimation of the spacing factor can be calculated, hereby neglecting all other voids. These are 925 μm and 359 μm for SAPa and SAPb, respectively. The values in Table 6 are lower due to the neglected air voids in the calculation, the random distribution of the voids and the use of aggregates. The trend for the

smaller SAPb is found when comparing SAP 3 versus SAP 4; a lower spacing factor. The standard deviation in SAP 2 is too large to make such analogous qualitative comparison for SAP 1 versus SAP 2.

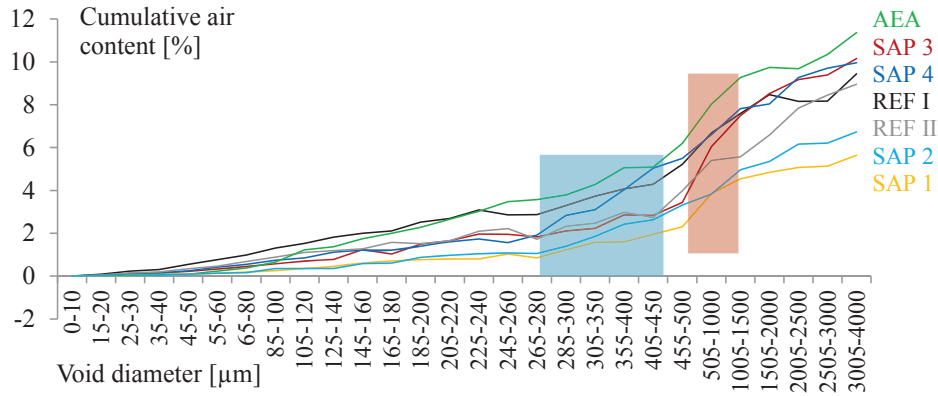


Figure 1: Cumulative air content of the studied samples.

3.2 Mechanical properties

At 28 days, the HVFA concrete mixtures have a lower compressive strength than the corresponding OPC references (Table 7). This behaviour can mainly be attributed to the slow pozzolanic reaction of the fly ash which first requires the presence of Ca(OH)_2 , one of the main hydration products of the Portland cement reaction. When looking at the effect of the applied AEA dosages, it is clear that adding 5.0 ml AEA/kg B resulted in rather limited strength reductions for the HVFA mixtures (-8 MPa). Given the rather low air contents achieved as such (see Table 5) this is not very surprising. Applying the increasing dosage of 7.0 ml AEA/kg B to achieve a more satisfactory air void system had a much more pronounced effect in terms of strength reduction (-23 MPa).

Table 7: Mechanical properties [MPa] of the HVFA and corresponding reference mixtures. The results show averages and standard deviations on the single results.

	T(0.45)	T(0.45)A	F(1)50	F(1)50A(-2)	F(2)50	F(2)50A(-2)
Compression	69.3±1.4	53.2±2.0	59.7±0.3	51.3±0.4 (36.8±3.9)	51.7±0.8	43.6±0.4 (29.1±5.6)

When using SAPs and additional water, the strength decreases within an acceptable range (Table 8). Increasing the water-to-cement ratio without adding SAPs, the strength also decreases within the range of the SAP mixtures without additional water. Using an air-entraining agent decreases the strength to a high extent, especially due to the entrained air content. When using SAPs and no additional water, the effective water-to-cement ratio decreases, thus increasing the strength. The combined effect with the strength reduction by the SAPs leads to a material with approximately the same strength as the REF I mixture.

Table 8: Mechanical properties [MPa] of the SAP and corresponding reference mixtures. The results show averages and standard deviations on the single results.

	REF I	REF II	SAP 1	SAP 2	SAP 3	SAP 4	AEA
Compression	57.8±0.7	52.0±0.9	58.0±1.0	50.0±2.1	49.3±1.0	48.6±0.8	33.8±1.4

3.3 Salt scaling resistance

The salt scaling results are given after 7, 14 and 28 cycles (Figure 2) for all studied mixtures. From Figure 2a it is clear that ordinary Portland cement concrete does not necessarily require air entrainment to ensure limited salt scaling. The addition of a limited amount of AEA (2.0 ml/kg B) to the same OPC concrete mixture was found to be very effective, since the mass loss per unit area was negligible (0.07 kg/m²). In contrast with reference mixture T(0.45), the performance of the non-air entrained HVFA mixtures subjected to the same experiment was far from acceptable. Using an AEA significantly improved the salt scaling resistance. However, the effectiveness of the admixture was less than in reference T(0.45)A due to the typical partial adsorption of the AEA by the fly ash. Although the AEA dosage was more than doubled (5.0 ml/kg B versus 2.0 ml/kg B), higher mass losses per unit area were recorded for the HVFA mixtures F(1)50A and F(2)50A. An acceptable salt scaling resistance was confirmed for mixtures F(1)50A-2 and F(2)50A-2 with a more adequate air void system. A more in-depth characterization of their air void systems in relation to their salt scaling performance can be found in Van den Heede et al [15].

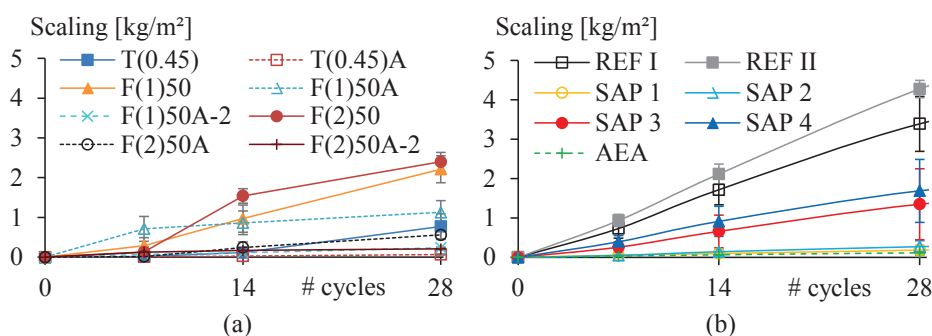


Figure 2: The amount of scaled material as a function of the number of cycles by conducting freeze-thaw resistance tests for the HVFA (a) and SAP (b) series.

The REF II mixture showed the highest scaling as the mixture was especially susceptible due to the high water-to-cement ratio. The next mixture is REF I. Both SAP 3 and SAP 4 show a lower scaling but not as good as the AEA mixture, and they are not significantly different in terms of scaling. The SAP 1, SAP 2 and AEA behave best in terms of reduced scaling. The difference between SAP 1/2 and SAP 3/4 is due to the lower apparent water-to-cement ratio. This causes a further increase in the salt scaling resistance as the strength is increased. The rate of scaling showed that the reference mixtures deteriorated more rapidly compared to the mixtures with SAPs, again proving their increased salt scaling resistance. The changes in mass loss for SAP-containing mixtures in comparison to the SAP-free concrete (REF I) after

28 freeze/thaw cycles are 60% for SAP 3, 50% for SAP 4, 95% for SAP 1, 92% for SAP 2, 97% for AEA and -26% for REF II. Thus, there was a considerable improvement in the materials performance due to the use of SAPs in terms of decreasing mass loss after a given number of freeze/thaw cycles.

4. Conclusions

- The overall results of the first series of HVFA concrete indicate that it is possible to design a HVFA concrete composition with an adequate salt scaling resistance under laboratory conditions. To achieve this, a lot of attention must be paid to the applied AEA dosage and the resulting air void system.
- Air void analysis proved to be useful to determine the evenly distributed void system created by the superabsorbent polymers. The macro-pore sizes were as expected.
- The compressive strength is mostly governed by the formation of macro pores by the SAPs when using a high water-to-cement ratio and additional water.
- Superabsorbent polymers are able to increase the freeze-thaw resistance as they caused less scaling due to the formation of an evenly-distributed pore system, when using additional water. When SAPs without additional water were used, the performance even further improved considerably. The overall improvement was hereby similar to that obtained with conventional air entrainment. However, when using an air-entraining agent, the strength decreased significantly, even more compared to using SAPs with additional water in a mixture.

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