Durability behavior of high-volume fly ash concrete.

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ABSTRACT: High-volume fly ash concrete is one of the ‘green’ concrete alternatives indirectly reducing the CO_2 emission and energy consumption related to the production of cement. This paper describes the results of durability tests on 4 concrete mixtures, each with a different fly ash replacement level, namely 0, 35, 50 and 67 %, at the age of 1 and 3 months. More specific, the resistance to freezing and thawing with and without the influence of deicing salts and chloride penetration has been examined. The vacuum saturation technique and capillary water absorption experiment are executed to get an indirect idea of porosity characteristics. An automated air-void analysis is used to determine the values of air content and spacing factor for all mixtures. The fly ash mixtures appear to exhibit a good freezing and thawing resistance while the influence of deicing salts is different on the surface and the inside of the concrete specimens. The 50 and 67% fly ash mixture exhibit a very high chloride migration coefficient at the age of 1 month but after 3 months of curing all mixtures show a good chloride migration resistance. Porosity results indicate that the capillary porosity of the fly ash mixtures still decreases after 1 month of curing. At the age of 3 months it appears that a higher fly ash replacement level causes a higher capillary and total porosity and a higher capillary water absorption rate.

1 INTRODUCTION

Since 1987 the term ‘Sustainable Development’ has become more significant. In the cement and concrete industry this caused the development of different alternative ‘green’ solutions. For example, to reduce the impact of the CO_2-emission and energy consumption, high-volume fly ash concrete was developed in Canada during the late eighties. Fly ash is a by-product of the coal fired electricity production and its worldwide production is estimated to be 500 million tonnes a year. By replacing large volumes of the cement content by fly ash, the latter gets a high quality application. But in order to obtain a sustainable construction it is essential to use durable concrete.

In almost all chemical and physical processes which have an influence on durability, the transport of fluids (water, gas and solutions) plays a crucial role. The rate, size and effect of this transport is determined to a large extent by the permeable porosity of the concrete. More specific, the permeability of the capillary network determines to what extent the fluids can enter and move through the concrete [Apers et al. 2006].

In this paper some aspects of the durability behavior of concrete mixtures with different fly ash replacement levels are studied as well as the permeable porosity and capillary properties. The effect of time is verified by executing the tests at the age of 1 and 3 months.

2 EXPERIMENTAL DETAILS

2.1 *Materials*

Four types of concrete mixtures were designed, each with a different cement - fly ash replacement level, respectively 0%, 35%, 50% and 67%. For all mixtures a normal Portland cement (CEM I 52.5), complying with EN 197-1 (2000), was used and a class F (according to ASTM standards) fly ash with a low calcium oxide content (2.8%). All used mixtures were made with a water-cementitious materials ratio (W/Cm) of 0.4 and a superplasticizer (SP), based on carboxylic acid, was added to obtain a decent workability. Former research indicated that the use of air entraining agent (AEA) in fly ash mixtures results in a significantly better resistance to freezing and thawing (in combination with deicing salts), so AEA was also added to acquire a fresh air content of about 4% [Baert 2003], determined according to the Belgian standard NBN B15-224 (1970). Table 1 summarizes some mixture properties of the 4 studied concrete mixtures.

Table 1. Mixture properties.

	FA/Cm (-)	AEA (ml/m³)	SP (ml/m³)	Air Content (%)
REF	0.00	270	1080	3.8
FA35	0.35	350	486	4.1
FA50	0.50	400	473	4.7
FA67	0.67	400	343	4.2

2.2 *Experimental procedures*

All tests are performed on samples at the age of 1 and 3 months. During this period the specimens are stored in a climate chamber with a relative humidity above 90% and temperature around 20 degrees Celsius.

Concerning the durability behaviour of high-volume fly ash (HVFA) concrete, the resistance to freezing and thawing, with and without the influence of deicing salts, and to chloride penetration is examined. Furthermore, the permeable or 'open' porosity is determined using the vacuum saturation technique, combined with specific drying procedures, and the capillary properties are verified by means of the sorptivity coefficient. Finally, the air void content and spacing factor are determined using an automated air void analysis system.

2.2.1 *Freezing and thawing*

After each curing period, 6 cubes (150 x 150 x 150 mm³) of each mixture are used to determine the resistance to freezing and thawing according to the Belgian standards NBN B15-231 and NBN B05-203. After immersion in water till constant mass (<0.1% mass change in 24h) 3 cubes are used to define the splitting tensile strength (according NBN B 15-218) before testing and the 3 others are subjected to a series of 14 freezing and thawing cycles, corresponding tot the most severe conditions of the Belgian climate. Possible changes in strength are verified by determining the splitting tensile strength of the 3 cubes after the test. Ultrasound velocity measurements before and after testing should indicate the existence of microstructural imperfections caused by freezing and thawing.

2.2.2 *Freezing and thawing with deicing salts*

For this test cores with a diameter of 100 mm are drilled out of 3 cubes (150 mm) of each mixture, perpendicular to the molded surfaces. These cores are then sawn in three so that finally each mixture has 9 cylindrical specimens with a diameter of 100 mm and thickness of ca. 50 mm, 6 containing a molded surface and 3 from the center of the cube. The former specimens are further indicated with a letter 'A', the latter with a 'B'.

These specimens are prepared for the test in a way that only one surface is covered with a 3 % NaCl solution and subjected to 28 freezing and thawing cycles according to EN 13339:2003. The experimental set-up is shown in Figure 1.

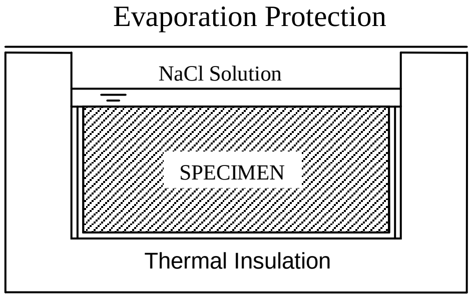

Figure 1. Test set-up deicing salts.

After every 7 cycles the scaled off material is collected, dried and weighed so that the mass loss per unit area can be determined as a function of time.

2.2.3 *Chloride diffusion*

The resistance to chloride penetration is determined according to NT Build 492, following the procedure as described in [NT Build 1999, Tang 1992]. The specimens for this test are obtained in the same manner as described in §2.2.2. For each mixture, series of 3 specimens, coming from the same cube, are tested at the same time, the specimens having a molded surface are placed in such a way that this surface is exposed to the chloride ions.

During the experiment, also called CTH, series of 3 cylinders are first vacuum saturated in a $Ca(OH)_2$ solution and then subjected to the non-steady state rapid migration test. The latter means that an external potential is applied axially across the specimen and forces the chloride ions outside (NaCl solution) 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. This enables to measure the chloride penetration and the chloride migration coefficient can be calculated by means of the following equation (1):

$$D_{nssm} = \frac{0.0239\ (273+T)L}{(U-2)t}\left(x_d - 0.022836\sqrt{\frac{(273+T)Lx_d}{U-2}}\right)$$

Where D_{nssm} = non-steady-state migration coefficient (x 12^{-12} m²/s); U = absolute value of applied voltage (V); T = average value of the initial and final temperatures in the anolyte solution (°C); L = thickness of the specimen (mm); x_d = average value of penetration depths (mm); and t = test duration (hours). The experimental set-up is given in Figure 2.

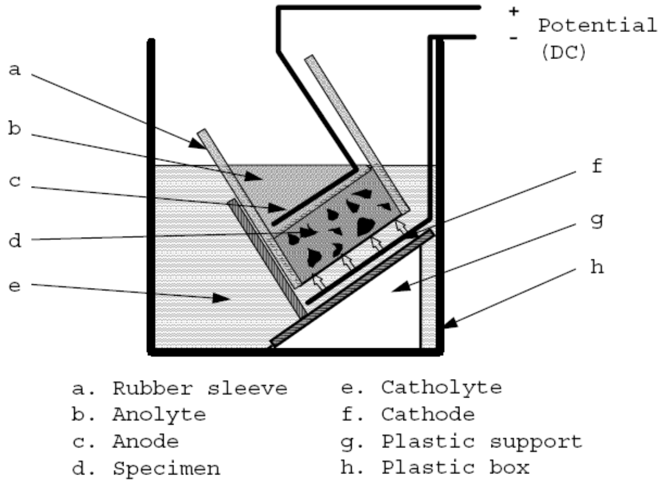

Figure 2. Arrangement of the migration set-up [NT Build 1999].

2.2.4 *Permeable porosity*

The vacuum saturation technique is the most efficient method to measure the permeable porosity of concrete [Safiuddin & Hearn 2005]. The specific method used here is described in the Belgian standard NBN B05-201 and makes it possible to determine the porosity in contact with the exterior, also called the permeable or 'open' porosity. This porosity influences to a large extent the penetration of (harmful) substances into the concrete and consequently its durability behavior.

The specimens for this test are obtained in the same manner as described in §2.2.2. This means that of each mixture 9 cylinders with a diameter of 100 mm and a thickness of ca. 50 mm are tested, 6 of them having a molded surface (A) and 3 others taken from the center of the cubes (B). The specimens are first dried in a ventilated oven at 45°C to constant mass, then vacuum saturated and weighed and at last the specimens are dried at 105°C (again to constant mass). Vacuum saturation is performed by placing the specimens in a vacuum with a residual pressure of 2.7 kPa during 2.5 hours, then water is added at a rate of 5 cm/h. After immersion the atmospheric pressure is re-established and the specimens are kept immersed for another 24 hours. This test procedure makes it possible to get a rough estimation of the capillary and gel porosity of the concrete [Audenaert 2006].

Generally, the porosity can be described as follows:

$$\varphi = \frac{\text{Volume that can be filled with fluids}}{\text{Volume of the dry concrete}} \qquad (2)$$

Since water is used for the saturation and as a fluid to determine the volume of the specimen, the equation can be simplified:

$$\varphi = \frac{m_s - m_d}{m_s - m_l} \qquad (3)$$

Where φ = permeable porosity (-); m_s = saturated mass of specimen measured above water (kg); m_l = saturated mass of specimen while submerged in water (kg); and m_d = mass of the dry specimens (kg). Calculating this porosity after drying at 45°C and 105°C gives an idea of respectively the capillary and total porosity, the latter being the sum of capillary and gel porosity.

Former work [Audenaert 2006] showed that this method provides values of porosity well corresponding to reality, except that the capillary porosity is a little underestimated because the specimens are dried at 45°C for only 2 weeks. After these two weeks the mass is still decreasing but less fast than 0.1 mass% in 24 hours. This underestimation is shown to be about 1.78 % for mixtures with portland cement [Audenaert 2006].

2.2.5 *Capillary properties*

Capillary transport of water, especially near an unsaturated concrete surface, is probably the most dominant invasion mechanism for deleterious substances like, for instance, chlorides. And the overall durability of concrete is to a large extent determined by the ease with which fluids can penetrate into the concrete. Clearly, the rate of capillary sorption will depend on the degree of saturation of the porous medium [Martys & Ferraris 1997]. For the investigated mixtures, the capillary suction is determined after drying at 45°C and at 105°C to constant mass .

A measure for the capillary behavior is the water absorption coefficient, also called the sorptivity coefficient S ($kg/m^2s^{0.5}$). The sorptivity is determined from the slope of the straight line obtained by plotting the cumulative volume of water absorbed per unit area against $t^{0.5}$. The general equation defining one-dimensional water absorption is as follows [Claisse et al. 1997, Hall 1989, Wilson et al. 1999]:

$$i = St^{0.5} + B \tag{4}$$

For this test 3 cubes of 150 mm x 150 mm x 150 mm of each mixture are tested. First, the lower parts of the sides adjoining the inflow face are sealed with a self-adhesive tape to avoid the exchange of water vapor and absorption into the surface pores and achieve unidirectional flow. The upper part of the specimens is not covered to prevent pore pressure from building up. After each drying procedure the specimens are placed on rods and immersed in water to a maximum depth of 5 mm in a climate chamber with a constant temperature of 20 ± 3 °C and relative humidity of 60 ± 3 %. The uptake of water by capillary absorption is measured by weighing the specimens at time intervals of 0, 1.5, 3, 6, 24, 72, 168 and 240 hours.

2.2.6 *Air void properties*

An air void analysis was executed on 3 cylindrical specimens with a diameter of 80 mm of each mixture according to the ASTM C457 and EN 480-11 standards. Therefore, the Rapid Air 457 is used, which is a fully automatic system for analyzing the air void content of hardened concrete. This analysis requires polishing of the examined surface, as described in ASTM C 457 as well as a contrast enhancement of the surface by use of black ink and white $BaSO_4$ powder. The 3 specimens were analyzed in two perpendicular directions using 10 probe lines per frame with a total traverse length of 17733 mm and the scanned surface is about 50 mm x 50 mm. These settings are chosen to obtain an optimal situation of maximum scanned surface and minimum scanning time [Annerel 2005].

However, it should be mentioned that the automatic system measures what it sees and that may be more or less accurate depending upon sample preparation quality, especially the ordinary procedure with cutting, grinding and lapping. The sample surface has to be without scratches and the air voids must have sharp edges [Jakobsen et al. 2006].

3 RESULTS AND DISCUSSION

3.1 *Freezing and thawing*

To verify if the specimens subjected to the 14 freezing and thawing cycles have suffered any internal damage, the splitting tensile strength (STS) and ultrasonic pulse transmission time have been examined before after the experiment. The values of STS, before and after testing, for each mixture at the age of 1 and 3 months are shown in Figure 3.

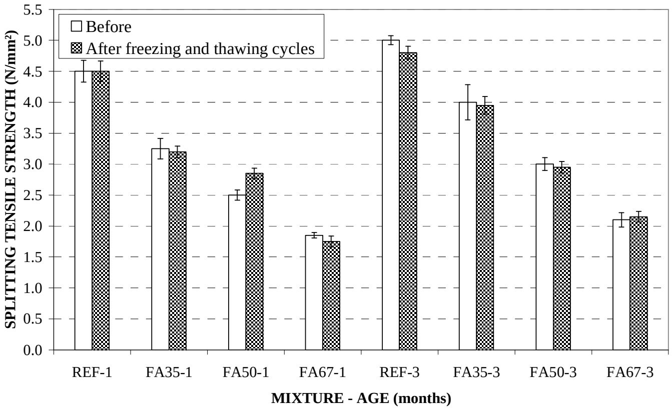

Figure 3. Splitting tensile strength before and after freezing and thawing.

It can be seen in Figure 3 that the specimens of all mixtures are apparently not affected by the influence of freezing and thawing. The values of STS are quite similar before and after testing. Figure 3 shows clearly that the influence of time is very important, the STS still increases significant after 1 month. It also appears that the STS decreases with increasing fly ash replacement level.

As previously mentioned the ultrasonic pulse transmission time (UPTT) through the concrete was also measured before and after the test to detect possible internal damage. For each measuring point (5 in total) on the different cubes, the relative ultrasonic pulse transmission time ($UPPT_\gamma$) was calculated given by [Tang & Petersson 2001]:

$$\gamma = \frac{t_i}{t_0} \, x \, 100 \quad (\%) \qquad (5)$$

Where t_i = transmission time measured after i cycles of freezing and thawing (in this case 14) (µs) and t_0 = initial transmission time (µs). When the UPPT_γ value is about 100% it can be assumed that the specimens are not deteriorated. Values higher than 110% indicate a possible damage. The maximum and minimum UPPT_γ value of the 5 measurements on each cube is shown in Figure 4.

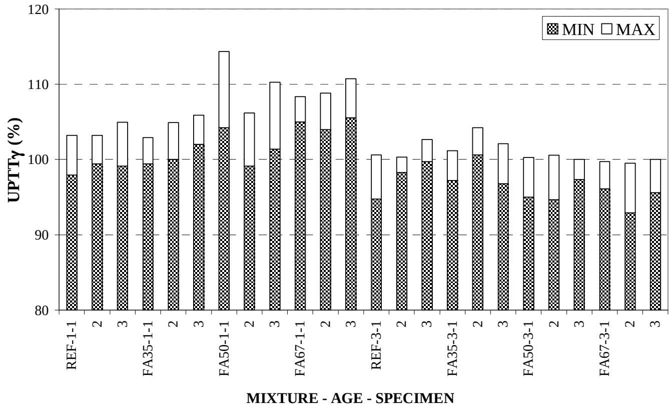

Figure 4. Minimum and maximum UPPT_γ values for each cube of each mixture.

Figure 4 shows that specimens subjected to the freezing and thawing cycles at the age of 1 month will possibly be slightly deteriorated, especially the 50% and 67% mixtures. Nevertheless, when the specimens are 3 months old, no more deterioration can be detected. The maximum UPPT_γ value measured on each cube is for all mixtures about 100%, which means that no negative microstructural changes have occurred.

3.2 *Freezing and thawing with deicing salts*

For each mixture, the mass loss due to scaling of the 9 cylinders is measured after 7, 14, 21 and 28 freezing and thawing cycles. The results of this test are provided in Figure 5. As previously mentioned, a distinction has been made between ‘A’ and ‘B’ specimens, A and B respectively referring to specimens of which a molded and inner surface is exposed.

The results of this test show that there’s a major difference in mass loss between the two types of surfaces. It appears that the molded surface (usually exposed to the outdoors conditions), scales off more significantly compared to the interior surface, especially with the high-volume fly ash mixtures.

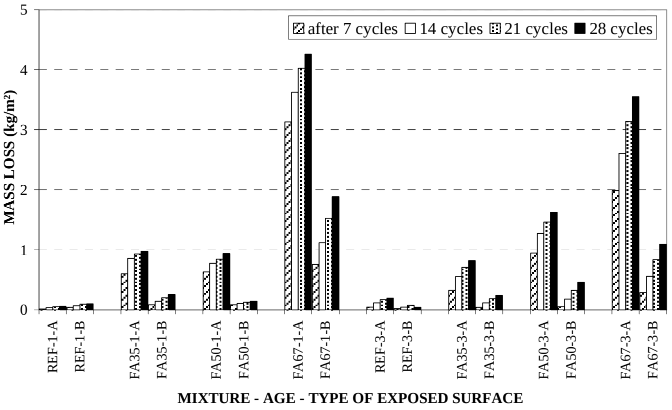

Figure 5. Mass loss due to scaling.

This indicates that the outer layer of a fly ash concrete structure shows significantly different properties compared to the interior of the concrete. What exactly the role is of the molded oil in this matter should be further investigated as well as the microstructural properties of these surfaces and the influence of the AEA. After all, the resistance of concrete to freezing and thawing is strongly affected by an air void system of which the air voids have the proper dimensions and are well distributed [Sutter 2002]. The air void system of all mixtures is examined using the Rapid Air 457, as mentioned in §2.2.6, and some air void properties are given in §3.6.

From figure 5 it also appears that age mostly but not always has a positive influence on the freezing and thawing resistance with deicing salts; it can be noticed from figure 5 that the 50% fly ash mixture exhibits a higher mass loss after 3 months curing compared with 1 month.

Different Belgian standards require that the average mass loss after the test is equal to or smaller than 1 kg/m² [Boel 2006]. This means that at the age of 1 month all mixtures meet this requirement, except for the 67% fly ash mixture, and after 3 months all test results of the B-surfaces are satisfying with the 67% mixture just exceeding the restriction.

3.3 *Chloride diffusion*

The non-steady-state migration coefficient D_{nssm} (x 12^{-12} m²/s) is plotted as a function of the fly ash replacement level in Figure 6.

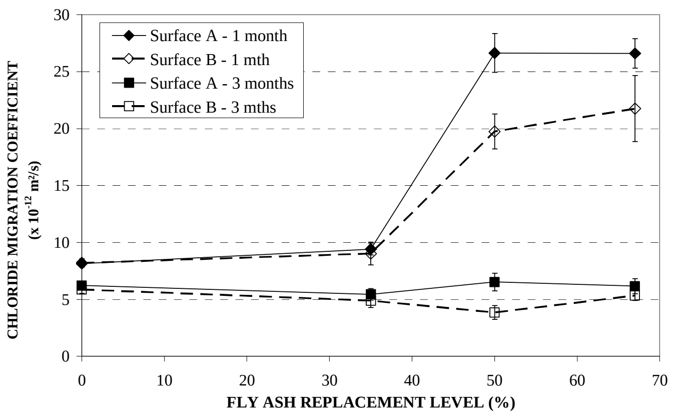

Figure 6. The chloride migration coefficient as a function of the fly ash replacement level and curing period.

It seems that after 1 month the high-volume fly ash mixtures exhibit a much higher D_{nssm} compared to the reference and 35% fly ash mixture. However, the difference disappears at the age of 3 months. This indicates that between 1 and 3 months an important change in the pore system of the concrete occurs, calculations of the porosity should make it possible to confirm this (see §3.4).

When comparing surface A and B, it can be noticed that for the 50 and 67% fly ash mixtures at 1 month the B specimens exhibit a lower migration coefficient. This demonstrates again that with these mixtures the molded surface has probably a much more porous structure, compared to the inner part of the concrete. This difference is not noticeable with the reference and less remarkable with the 35% fly ash mixture. At an age of 3 months the difference between A and B is less explicit except for the 50% mixture. When looking at the B specimens at 3 months, the 50 % mixture shows the lowest migration coefficient, followed by the other two fly ash mixtures and finally the reference mixture. The A specimens at the same age all exhibit a value around 6 x 10^{-12} m/s². At the age of 3 months all values are below 8 x 10^{-12} m/s² which indicates a good resistance to chloride penetration according to [Tang 1992] and these values are expected to decrease even more in time.

3.4 *Permeable porosity*

The durability behavior of concrete will always be most influenced by the largest connected pores. Because the air voids, being the largest pores, are less connected the capillary porosity, more specifically the permeable porosity, is most important concerning durability aspects. Figure 7 shows the values of total permeable porosity (TPP) and capillary permeable porosity (CPP) as a function of the fly ash replacement level, at the age of 1 and 3 months.

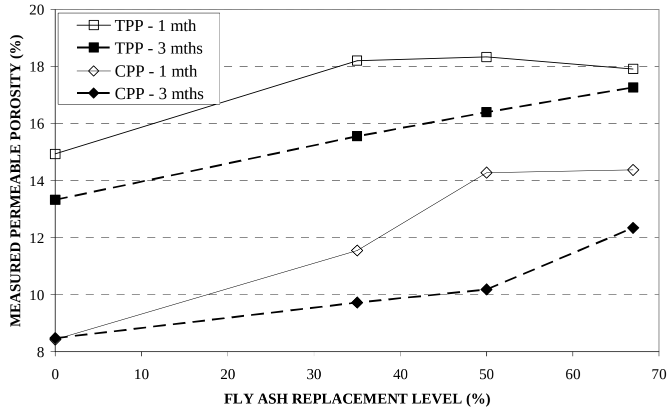

Figure 7. Values of total and capillary permeable porosity at the age of 1 and 3 months.

It seems that at the age of 1 month the value of total porosity of all fly ash mixtures is quite similar, being significantly higher than the reference. However, when looking at the capillary porosity the 35 % fly ash mixture shows a lower percentage capillary pores. It can be noticed that the higher the percentage fly ash replacement, the higher the capillary porosity, with the 50% and 67% mixtures exhibiting a similar value.

After 3 months a significant linear correlation (correlation coefficient $R^2 = 0.997$) can be found between the total porosity and the fly ash replacement level. The capillary porosity also increases with higher fly ash percentages but a perfect linear relation cannot be found, especially the 67% fly ash is an outlier with a much higher capillary porosity. For all other mixtures the proportion TTP/CPP is quite similar.

It can also be mentioned that after 3 months the total porosity decreases the most with the 35% and 50% fly ash mixture, followed by the reference and 67% mixture. Considering the capillary porosity, all fly ash mixtures show a significant lower capillary porosity after 3 months, especially the 50% fly ash mixture. Moreover, the FA50 and FA67 exhibit both an increasing gel porosity, which clearly illustrates the transformation of capillary pores into gel pores in time, or also called the refinement of the pores.

It should be noted that the permeability of concrete is not a simple function of its porosity, but is considerably affected by the size, distribution and tortuosity of pores, the type and distribution of aggregates, and the nature and thickness of interfacial transition zone [Safiuddin & Hearn 2005].

3.5 *Capillary properties*

As mentioned before, a measure for the capillary behavior of concrete is the sorptivity coefficient. This coefficient describes the ease with which a dry material absorbs water. To examine the influence of the saturation degree the capillary suction test was executed after drying at 45°C and after drying at 105°C, each time to constant mass. Again, these tests were performed with specimens at the age of 1 and 3 months. The results are plotted in Figure 8.

The cumulative water absorption curves of all mixtures dried at 45°C and 105°C show that after 6 respectively 24 hours the water uptake slows down. The data points of these periods, except the origin point, are used to calculate the sorptivity coefficient.

Figure 8 clearly shows that the initial water content indeed plays an important role as the sorptivity rises with the about the same proportion for all mixtures, except for the 67% fly ash mixture.

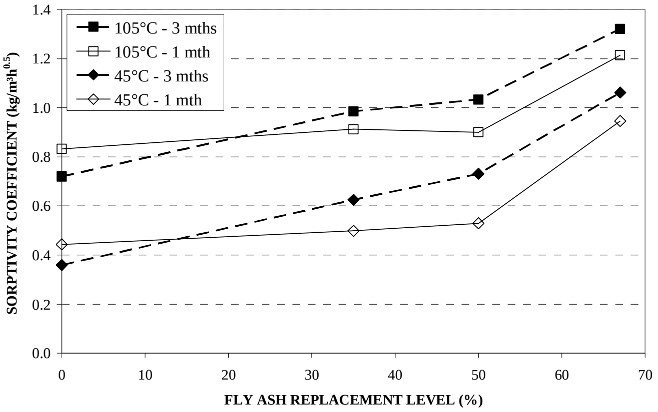

Figure 8. Sorptivity coefficient as a function of the fly ash replacement level.

Considering the time effect, a significant difference can be noted between the reference and fly ash mixtures. While the sorptivity decreases in time with the reference, the sorptivity coefficient of the fly ash mixtures increases after 3 months. This could possibly be explained by the lower capillary porosity (refinement of the pores) of the fly ash mixtures at the age of 3 months (see Figure 7). The presence of more smaller capillaries may cause a stronger capillary suction force and, as a consequence, a larger water absorption rate at the start of the sorption experiment. The total uptake of water at the end of the experiment will probably be more determined by the larger capillaries.

The influence of the capillary porosity at the age of 3 months becomes clear when the sorptivity coefficient is plotted against the measured capillary porosity as determined in §3.4, see Figure 9. The empty and filled symbols refer to sorptivity coefficients respectively determined after drying at 105°C and 45°C. Linear equations and correlation coefficients of the regression lines are given within Figure 9.

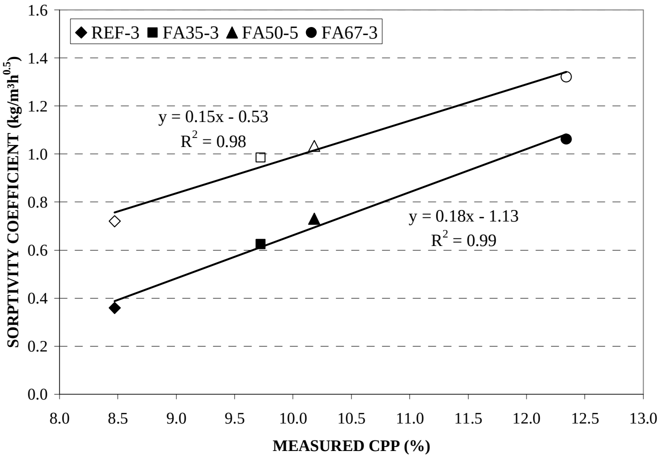

Figure 9. Sorptivity coefficient as a function of the measured capillary permeable porosity (CPP).

An almost perfect linear correlation ($R^2 = 0.98$ and 0.99) is found between the sorptivity and measured CPP. As the fly ash replacement level increases, the capillary porosity increases and a higher sorptivity coefficient can be found. A similar relation is noticed when looking at the total porosity at 3 months. At the age of 1 month such correlations are absent, with especially the 67% fly ash mixture having a deviant behavior.

3.6 *Air void properties*

To obtain a satisfying freezing and thawing resistance most standards prescribe an air content of minimum 3.5 to 4 % (in case of moderate exposure) [ACI 1992] and a spacing factor of maximum 200 µm [ASTM 2006]. These criteria are valid for concrete containing air entraining agent. The air content (AC) and spacing factor (SF) of each mixture is listed in Table 2.

Table 2. Air content and spacing factor of the mixtures at the age of 1 and 3 months.

Mixture	1 month		3 months	
	AC (%)	SF (mm)	AC (%)	SF (mm)
REF	3.78	0.12	2.57	0.19
FA35	4.68	0.10	3.30	0.16
FA50	6.12	0.09	3.09	0.12
FA67	5.03	0.10	5.93	0.08

It should be indicated that the air content values of different samples of the same mixture strongly deviate, sometimes up to 2 % and the air void properties are measured on specimens taken from the inner part of the concrete.

According to the average values, the mixtures should be resistant to freezing and thawing. Indeed, results of the freezing and thawing test show that the mixtures are not severely damaged by the freezing and thawing cycles. When looking at the test results of freezing and thawing in combination with deicing salts, an important difference was noticed between the specimens with molded surface (A) and the ones taken from the inner part of the concrete (B). The mass loss of the A specimens was significantly higher compared to the B specimens. The values given in Table 2 refer to the B specimens and confirm that when the air void requirements are met, the freezing and thawing resistance is satisfactory. However, it should be mentioned that the 67% fly ash mixture generally exhibits a significant larger mass loss. This indicates that not only air content and spacing factor determine the freezing and thawing resistance with deicing salts. The air void properties of the molded surface should be studied to know what causes the extreme difference in mass loss between A and B.

4 CONCLUSIONS

This paper describes the results of different tests executed to study durability aspects of concrete mixtures with different levels of fly ash replacement (up to 67%). Because durability is, to a large extent, determined by the ease with which fluids can enter and migrate through the concrete, the porosity and capillary properties of the mixtures were also examined. Following conclusions could be drawn:

- At the age of 1 month there can possibly be internal damage due to repeated freezing and thawing in the 50 and 67 % fly ash mixture, but no significant changes in the splitting tensile strength are noticed. After 3 months of curing all mixtures exhibit a very good freezing and thawing resistance;
- When considering the freezing and thawing resistance with the influence of deicing salts, the fly ash mixtures seem to exhibit a rather noticeable behavior. The mass loss due to scaling is a lot higher at the molded surfaces compared to the inner part of the concrete. This indicates important differences of the air void properties and pore characteristics at the molded surface and inside the concrete;
- Concerning chloride migration the 50 and 67% fly ash mixture appear to have a significant higher value of the chloride migration coefficient. However, after 3 months all mixtures exhibit a good chloride migration resistance;
- Tests results of the permeable porosity show that the capillary porosity of the fly ash mixtures still decreases significantly after one month. After 3 months a perfectly linear correlation exists between measured total porosity and fly ash replacement. The higher the fly ash replacement level, the higher the total and capillary porosity;
- The capillary water absorption experiments show that fly ash mixtures appear to have a higher water absorption coefficient after 3 months of curing, unlike the reference which has the highest sorptivity coefficient at 1 month. This could possibly be explained by the refinement of the pores which causes a stronger capillary suction force. The influence of the capillary porosity on the sorptivity appears clearly at the age of 3 months where a linear correlation exists between the measured capillary porosity and the sorptivity coefficient.

The results of these tests indicate that further research is necessary to examine the microstructural characteristics of the upper layer and the inside of fly ash concrete specimens. More information about the size, distribution and tortuosity of the pores, nature and thickness of the interfacial transition zone is needed to better understand the durability behavior of the high-volume fly ash concrete.

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