



3 years of durability research on slag based mortars

Final report

STATENS VEGVESENS RAPPORTER

Nr. 599



Tittel

3-årig bestandighetsundersøkelse av mørtler med slaggsement

Undertittel

Sluttrapport

Forfatter

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Sammendrag

Et eksperimentelt program ble gjennomført for å studere langsiktige bestandighetsegenskaper for ulike tilsetningsmaterialer. I alt fem blandinger ble studert bestående av en Portlandsement med silikastøv, en blanding av Portlandsement og flygeaske og tre varianter av slaggsement. Prøvene ble studert ved hjelp av «Rapid Chloride Migration» (RCM), elektrisk resistivitetstest, naturlig karbonatisering og polarisasjons- og fluorescensmikroskopering (PFM). Denne rapporten inneholder data fra to dager opp til tre år. Konklusjonen er at systemene med slagg og flygeaske som brukes i denne undersøkelsen viser bedre bestandighetsegenskaper enn Portlandsement kombinert med silikastøv. Sementer med høye slaggmengder (> 65%) viser imidlertid noe redusert motstand mot karbonatisering i forhold til de øvrige bindemidlene.

Title

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Key words

durability, reinforcement corrosion, blast furnace slag cement, fly ash, chloride penetration, resistivity, carbonation, microscopy

Summary

An experimental program was carried out to study long-term durability behavior of supplementary cementitious materials (SCM) systems. A total of five mixes were studied consisting of one Portland cement with silica fume, one Portland-fly ash system and three blast furnace slag systems. The samples were studied using Rapid Chloride Migration (RCM), electrical resistivity test, natural carbonation and polarization- and fluorescence microscopy (PFM). This report contains data from two days up to three years. The conclusion is that the SCM systems used in this research, perform overall better in durability properties than just Portland cement. For high slag amounts (> 65%) a tradeoff between chloride and carbonation resistance is appearing

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1 Introduction

The Norwegian Public Roads Administration (Staten's Vegvesen; NPRA) is currently reviewing the durability performance of concrete mixes in existing bridges and tunnels and is considering possible future concrete mixes (including slag or fly ash) for the next generation of bridges and tunnels. This research is part of NPRA's research program *Durable structures*.

The Netherlands have almost a century experience in the use of ground granulated blast furnace slag (GGBS) cements with high slag content, comparable to current CEM III/B (66-80 % slag) as defined in EN 197-1:2011 [1] for major infrastructure, including marine concrete. Over decades in practice and in abundant laboratory investigations, CEM III/B concrete has demonstrated considerably better performance on durability issues such as chloride induced reinforcement corrosion and alkali-silica reaction than pure Portland cement.

The NPRA and TNO have established a collaboration in order to make this Dutch experience and knowledge available for evaluation within the aforementioned context, and identify possible knowledge gaps with regard to durability performance and needs for future research relevant to the aforementioned Norwegian research programme. In 2013 a state-of-the-art review of experience with CEM III/B cement in concrete in the Netherlands was written and published as NPRA report 270 [2]. Following discussion of the state-of-the art review, it was considered useful to collect experimental data on materials incorporating slag, based on binder mixtures that could potentially be available on the Norwegian market. Results up to one year age have been reported in [3] and [4]. This report documents the results of the experiments up to the age of three years, which concludes the testing program.

This study is part of the collaboration was between NPRA and TNO under agreement 2012082035 "RnD Collaboration Durable Structures – FB1 Durability of concrete with slag cement", as agreed in document 2012/082035-003 of August 28, 2013, which was extended in document "Agreement and extension of agreement 2012/082035-004 RnD Collaboration Durable Structures– FB1 Durability of concrete with slag cement signed October 10, 2013. This report is the final document provided under this extended agreement.

2 Materials, methods and testing schedule

2.1 General

The experimental program agreed on was the following.

The overall goal was to obtain data over time, from relatively young age up to several years, in order to document chloride penetration resistance, electrical resistance and carbonation of the different binders over time.

To this end, mortar specimens were made with five binders and were tested for chloride penetration resistance as well as electrical resistivity at ages between 2 days and three years. In addition, carbonation was tested at ages of one, two and three years. Moreover, specimens were sent to NPRA for RCM and resistivity testing at 120 days, and at one, two and three years age.

Materials used are described in section 2.2, methods applied in section 2.3 and the overall testing schedule in section 2.4.

2.2 Materials

Specimens were prepared using binders (see below) and sand according to the standard for testing of cement, NEN-EN 196-1:2005 [5], with the following deviation: a water-to-binder ratio was used of 0.40 instead of 0.50 in order to stay close to common Norwegian concrete technology. This means that mortar with composition cement:sand:water equals 1:3:0.4 (by mass) was made using rounded siliceous sand of 0-2 mm grain size. Mixing and casting were carried out according to the standard. A superplasticiser Cugla HR (35% solids) was added in order to obtain the same workability of all mixes. Its dosage was determined using trial mixes. Mix compositions are reported in detail in Annex A.

Binders used were:

- CEM I 52.5 N (LA) Rapid from Aalborg with 5% silica fume, denoted as A-CEM I+5%SF
- CEM II/A-V 42.5 N Anlegg FA from Norcem, with 5% silica fume, mix code B-CEM II/A-V+5%SF
- CEM III/B 42.5 N from NL (ENCI), mix code C-CEM III/B(NL)
- CEM III/B 42.5 N-SR/LH/NA from Cemex (Germany), mix code D-CEM III/B(D)
- CEM III/A 42.5 N-NA from Cemex with 5% silica fume, mix code E-CEM III/A+5%SF.

All binder materials were obtained via NPRA except the Dutch slag cement.

2.3 Methods

2.3.1 Microscopy

Polarization-and-fluorescence microscopy (PFM) on thin sections has been performed on all five mortars at an age of 2 years, according to procedures outlined in Nijland & Larbi [6].

Table 1 Overview of samples studied.

| Mix code | Binder | Casting date (2014) | Thin section code |
|---------------------|--------------------|---------------------|-------------------|
| A - CEM I+5%SF | CEM I + 5% SF | January 14 | TNO 01792 |
| B - CEM II/A-V+5%SF | CEM II/A-V + 5% SF | January 21 | TNO 01793 |
| C --CEM III/B (NL) | CEM III/B (NL) | January 28 | TNO 01794 |
| D - CEM III/B (D) | CEM III/B (D) | February 4 | TNO 01795 |
| E - CEM III/A+5%SF | CEM III/A + 5% SF | February 11 | TNO 01796 |

2.3.2 RCM testing

Specimens were tested for chloride penetration resistance using the rapid chloride migration (RCM) test according to NTBuild 492 [7]. For RCM testing, specimen moulds were PVC cylinders with approximate inner dimensions of 100 mm diameter and 50 mm height. After casting, the moulds were covered with plastic foil and stored in the laboratory at 20 °C and 95% RH for 24 hours. After 24 hours the specimens were demoulded and then immersed in saturated lime solution until the time of testing.

The test description involves vacuum saturation of cylindrical specimens (100 x 50 mm), mounting between chambers with electrodes filled with NaOH or NaCl solutions, and applying a DC voltage for a certain time. Voltage and time must be chosen from a table based on the initial current flowing through a specimen when 30 V is applied. After application of the voltage for the designated time, the specimens are split and sprayed with silver nitrate, upon which the chloride penetration front becomes visible. From the average penetration depth, the voltage and the time, the chloride migration coefficient is calculated.

The test was applied with the following deviations:

- Vacuum saturation was omitted for specimens up to 180 days age, considering that the specimens would not dry out significantly because they were stored at high humidity; and that vacuum treatment might cause damage to the microstructure, in particular at young ages; specimens tested at one, two and three years of age were vacuum saturated.
- Voltage and time were chosen based on previous experience on young mortars [8] and with slag cements; in practice this means an extension of Table 2 given in NTBuild 492 to the high applied voltage side for very dense mortars (Table 3) and to the lower applied voltage side for young mortars (Table 4). Note 2 below Table 2 was neglected. The overall objective of choosing voltage and time is to obtain about 25 mm of penetration. At three years age, test duration was varied between 5 and 9 days, in order to obtain penetrations close to 25 mm.

In addition, the following details of casting and testing specimens are given. Specimens were cast as discs with mix codes written on the finished (top) surface. The (non-marked) bottom-of-the-mould surface was exposed to the chloride solution (cathode side) in the RCM test. For the test, possible defects in the bottom surface are sealed by silicone sealant to prevent leakage.

Specimens sent to Norway for testing by NPRA were removed from the lime solution and packed in plastic to prevent drying out as much as possible.

Table 2 Settings for time and voltage according to NTBuild 492.

| Initial current I_{30V} [mA] (at 30 V) | Applied Voltage U [V] (after adjustment) | Possible new initial current I_o [mA] (at adjusted voltage U) | test duration t [h] |
|--|--|---|--------------------------|
| < 5 | 60 | < 10 | 96 |
| 5-10 | 60 | 10-20 | 48 |
| 10-15 | 60 | 20-30 | 24 |
| 15-20 | 50 | 25-35 | 24 |
| 20-30 | 40 | 25-40 | 24 |
| 30-40 | 35 | 35-50 | 24 |
| 40-60 | 30 | 40-60 | 24 |
| 60-90 | 25 | 50-75 | 24 |
| 90-120 | 20 | 60-80 | 24 |
| 120-180 | 15 | 60-90 | 24 |
| 180-360 | 10 | 60-120 | 24 |
| > 360 | 10 | > 120 | 6 |

Note1: the original Table in NTBuild 492 is titled: Test voltage and duration for concrete specimens with normal binder content.

Note2: The Table has a note stating: For specimens with a special binder content, such as repair mortars or grouts, correct the measured current by multiplying by a factor (approximately equal to the ratio of normal binder content and actual binder content) in order to be able to use the above table.

Table 3 Suggested voltage and time based on experience with dense (slag) concrete specimens.

| initial current I_{30V} [mA] (at 30V) | applied voltage U [V] (adjusted) | expected new initial current I_o [mA] (at adjusted voltage U) | test duration t [h] |
|---|--|---|--------------------------|
| < 2 | 60 | < 5 | 168 |
| 2-5 | 60 | 5-10 | 96 |

Table 4 Suggested voltage and time based on experience with young mortars [8].

| age [day] | CEM I | | CEM III/B | | Other binders with 5% SF | |
|-----------|--|---------|-----------|---------|--------------------------|---------|
| | Volt | Time | Volt | Time | Volt | Time |
| 1 | 10 | 150 min | 10 | 150 | | |
| 2 | 15 | 240 min | 15 | 240 min | 15 | 240 min |
| 7 | 15 | 24 hour | 15 | 24 hour | 15 | 24 hour |
| ≥ 14 | Measure current at 30 V and test (voltage and time) according to Table 2 | | | | | |

Note 1: testing at 1 day is not foreseen

Note 2: testing of pure CEM I is not foreseen

2.3.3 Resistivity

Resistivity was tested using an AC resistance meter (ESCORT LCR) at 120 Hz following either one or both of two procedures (see section 2.4 and Table 5):

- The cell resistance was measured after a specimen had been inserted in an RCM cell, before the actual application of the (initial) voltage; the cell resistance was also measured after the RCM test; the resistivity was calculated from the initial cell resistance by multiplying with the geometrical cell constant (surface area/length); the result is denoted **R_{rcm}**
- The resistance of a specimen was measured (after surface drying) by placing it between two steel plates with wetted cloth, after removal from the saturated lime solution; the resistivity was calculated using the geometrical cell constant (surface area/length); the result is denoted **R_{tem}**.

The geometrical cell constant is given by

$$\frac{\text{surface area}}{\text{thickness}} = \frac{\pi r^2}{d} = \frac{\pi (0.05)^2}{0.05} = 0.157 \text{ m} \quad (2.1)$$

Minor deviations from nominal dimensions are neglected.

2.3.4 Carbonation testing

For carbonation testing, specimens were 160 x 40 x 40 mm³ mortar bars cast in steel moulds. They were demoulded at 24 hours and stored in saturated lime solution. At seven days age, they were placed on the roof of the (old) TNO laboratory building without shelter. At one year age they were moved and placed in a roofed shed near the new TNO laboratory. At two years of age they were placed on the roof of the (new) TNO laboratory, under a transparent plastic roof. One mortar bar per mix remains still exposed in this location.

Carbonation depths were determined by splitting off a part of the prisms and spraying the freshly broken surface with phenolphthalein.

2.4 Testing schedule

The time schedule for testing is given in Table 5. Tests at nominally two years of age were carried out around 740 days after casting. Tests at nominally three years of age were carried out around 1100 days after casting.

Table 5 Planned testing schedule; for RCM: 3 specimens tested.

| age (day) | RCM and Rrcm \$ | resistivity Rtem & | Carbonation depth |
|-----------|-----------------|---------------------|-------------------|
| 2 | yes | yes | - |
| 7 | yes | yes | - |
| 14 | - | yes | - |
| 28 | yes | yes | - |
| 56 | - | yes | - |
| 60 - 90 | - | yes (all specimens) | - |
| 90# @ | yes | yes | - |
| 180 | yes | - | - |
| 270 | - | yes (all specimens) | - |
| 360@ | yes | yes (all specimens) | yes |
| 740@ | yes | yes | yes |
| 1100@ | yes | yes | yes |

carried out at 133 days age at TNO and at 120 days at NPRA.

@ specimens were sent to NPRA for testing at their laboratory

\$ Rrcm denotes resistivity tested on specimens in the RCM cell before the voltage was applied

& Rtem denotes resistivity tested on discs between steel plates

Rrcm was measured on all specimens subjected to the RCM test. Rtem was measured on planned occasions, in principle on three specimens. Measurements on 56 days age were not measured at that exact age because of relocation of the TNO laboratory. Instead all specimens were measured in April 2014 when the age of the specimens was between 60 and 90 days of age. Rtem was measured again on January 29, 2015 on all remaining discs. Specimen age was between 360 and 385 days. At later ages (2 and 3 year), Rtem was tested as follows. Three specimens per mix were tested on February 24, 2016 (about two years of age). All remaining specimens were tested on February 15, 2017 (about three years of age); that is, on four specimens for mix C, six specimens for mix B and on five specimens for the other mixes.

3 Results

3.1 Casting dates

Mortars were coded, prepared and cast as indicated in Table 5. For the first mix a trial mix was made, which was very stiff. Subsequently, a superplasticiser was added in order to get a plastic mix. For subsequent mixes, superplasticiser dosage was determined by trial and error for similar workability. Dosages used are reported in Table 6. Full mix proportions are reported in Annex A. Mixing batches were relatively small; a complete group of specimens for each mix (36 cylinders and 3 prisms) was composed of about 20 batches.

Table 6 Mix codes and casting information.

| mix code | binder | casting date (2014) | superplasticiser dosage |
|-------------------|--------------------|---------------------|-------------------------|
| A-CEM I+5%SF | CEM I + 5% SF | January 14 | 0.65% |
| B-CEM II/A-V+5%SF | CEM II/A-V + 5% SF | January 21 | 0.33% |
| C-CEM III/B(NL) | CEM III/B (NL) | January 28 | 0.13% |
| D-CEM III/B(D) | CEM III/B (D) | February 4 | 0.13% |
| E-CEM III/A+5%SF | CEM III/A + 5% SF | February 11 | 0.22% |

3.2 Microscopy observations

A detailed description of the microstructure findings with optical microscopy is given in Annex D. Here the main observations are summarized.

Mortar A

Mortar A has a homogeneous, dense microstructure with a relatively high entrapped air content (4-5 vol%, Figure 1). The binder is made up by well hydrated, fine grained Portland cement and some silica fume. At least part of the silica fume has agglomerated and did not react. Possibly, also some silica gel developed (Figure 2). Only a few narrow microcracks occur. Carbonation is visible by the higher order beige birefringence colours in cross polarized light. Carbonation depth is about 0.1 mm.

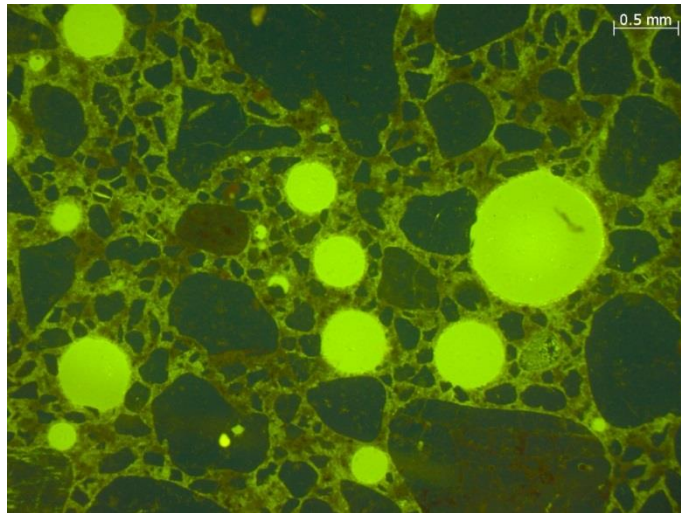


Figure 1 Microphotograph illustrating the relatively high entrapped air content of mortar A (TNO 01792, UV fluorescence).

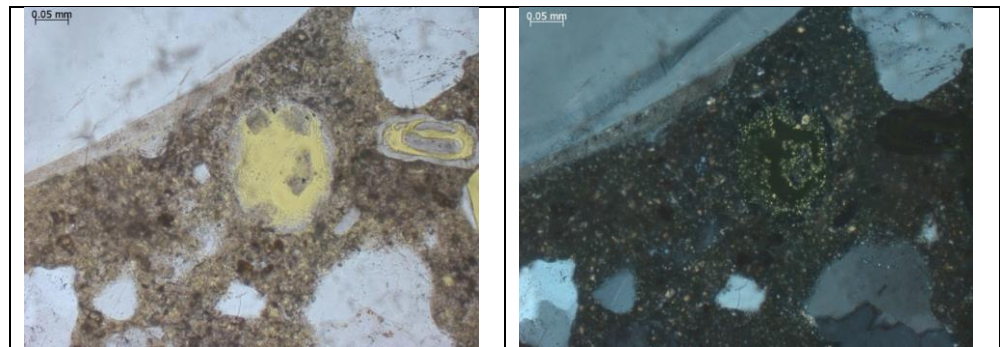


Figure 2 Microphotograph showing deposition of silica gel (?) at the interface between aggregate and cement paste (TNO 01792, left plane, right cross polarized light).

Mortar B

Mortar B has a homogeneous microstructure with a relatively high entrapped air content (3-4 vol.%). The binder of Portland clinker, powder coal fly ash and some silica fume is well hydrated; well-developed portlandite crystals are clearly visible. Silica fume has, at least to a certain amount, agglomerated and, in these agglomerates, did not react. Microcracks are almost absent. Carbonation is about 0.3 -0.5 mm.

Mortar C

Mortar C has a homogeneous microstructure with a relatively high entrapped air content (ca. 4 vol.%). A few very narrow microcracks occur. The cement paste shows a good coherence, the binder of Portland clinker with a high ground granulated blast slag content (i.e. CEM III/B) is well hydrated. Carbonation depth is about 1-2 mm.

Mortar D

Mortar D has a microstructure and binder comparable with that of mortar C, with several narrow microcracks. Entrapped air content is 4 -5 vol%, carbonation depth 1-2 mm.

Mortar E

Mortar E also has a homogeneous microstructure, with a binder made up by Portland clinker, a moderate amount of ground granulated blast furnace slag (i.e. CEM III/A) and some silica fume. At least part of the latter agglomerated and did not react. Otherwise, the binder is well hydrated. Some narrow microcracks occur. Carbonation depths are typical about 0.5 mm; locally, depths of about 2-3 mm are reached.

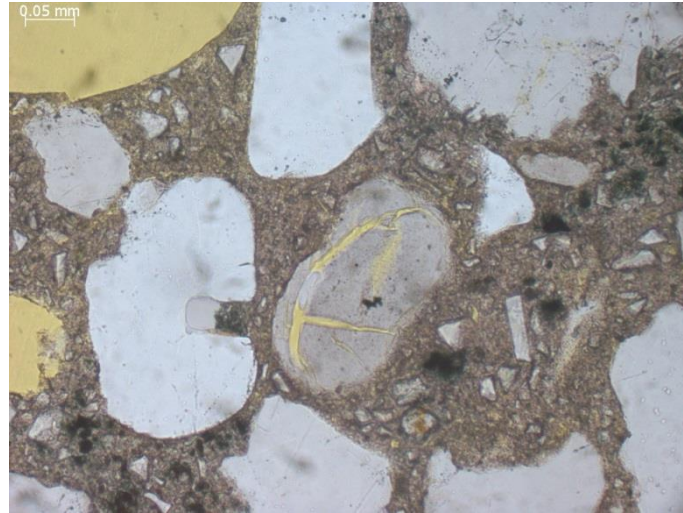


Figure 3 Microphotograph with agglomerated silica fume (centre) in mortar E (TNO 01796, plane polarized light).

3.3 RCM results

3.3.1 TNO results

An overview of RCM results is given in Table 7. Full experimental data of RCM testing up to 1100 days are given in Annex B.

The RCM results are graphically shown in Figure 4, Figure 5 and Figure 6 on linear and log-log scales. The linear plot shows a strong decrease during the early stages, the log-log plot better shows the development over the complete testing period.

The testing that was originally planned at 90 days' age was carried out around 130 days age due to the moving of the laboratory. As mentioned in section 2.3.2, specimens tested for RCM from an age of 360 days on were vacuum saturated as per NT Build 492.

Table 7 Overview of RCM results obtained at TNO * 10⁻¹² m²/s ; mean and standard deviation in ()

| mix code | A-CEM I 5%SF | B-CEM II/A-V 5%SF | C-CEM III/B NL | D-CEM III/B D | E-CEM III/A 5%SF |
|--------------------|---|----------------------|----------------|---------------|---------------------|
| age (day) | Drcm (10 ⁻¹² m ² /s) mean and standard deviation () | | | | |
| 2 | 23 (2.8) | 44 (1.9) | 119 (10) | 49 (4.9) | 40 (2.4) |
| 7 | 18 (0.9) | 21 (1.2) | 7.8 (0.3) | 6.3 (0.5) | 8.3 (0.6) |
| 28 | 2.8 (0.3) | 6.9 (0.7) | 4.0 (1.5) | 2.5 (0.15) | 2.5 (0.4) |
| 133 | 1.4 (0.2) | 1.4 (0.0) | 1.5 (0.1) | 0.83 (0.05) | 0.60 (0.15) |
| 185 | 1.5 (0.1) | 1.5 (0.2) | 1.7 (0.4) | 1.5 (0.2) | 0.66 (0.1) |
| 360 # | 0.87 (0.15) | 0.68 (0.1) | 0.43 (0.1) | 0.57 (0.03) | 0.37 (0.01) |
| 740 # | 1.2 (0.1) | 0.52 (0.06) | 0.51 (0.05) | 0.43 (0.1) | 0.26 (0.04) |
| 1100 # | 1.2 (0.08) | 0.52 (0.05) | 0.56 (0.04) | 0.56 (0.09) | 0.41 (0.05) |
| mean 1 – 3 year | 1.1 | 0.57 | 0.50 | 0.52 | 0.35 |

#specimens were vacuum saturated before testing

Note: for practical purposes, RCM results are commonly rounded to 0.5 unit or to 1 unit above a value of 10; here we have rounded to 0.1 unit (for values below 10) or 0.01 (for values below 1) in order not to lose information.

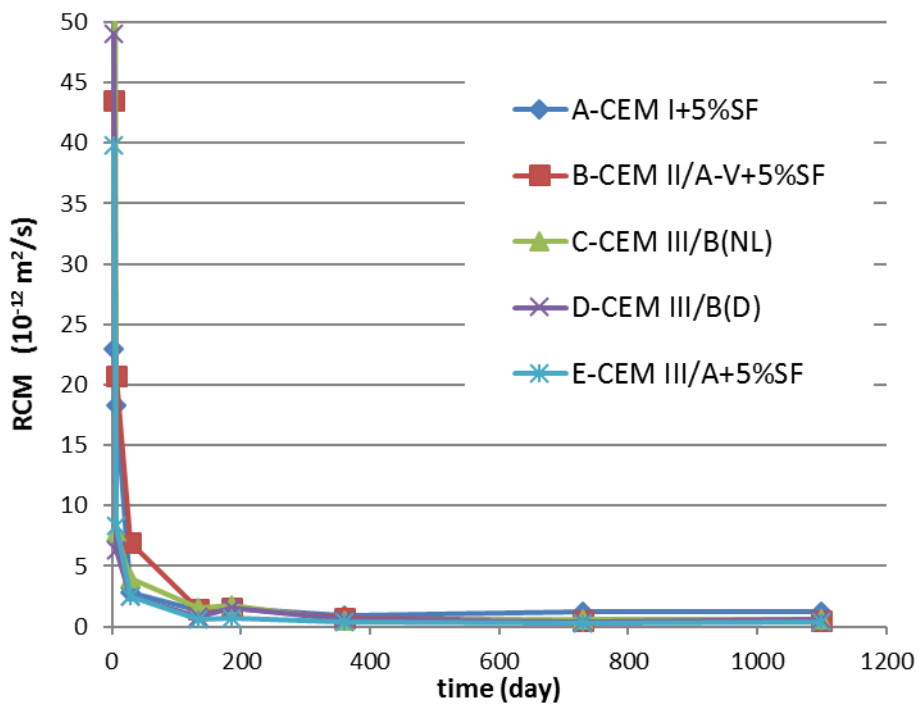


Figure 4 RCM results up to 1100 days; linear plot.

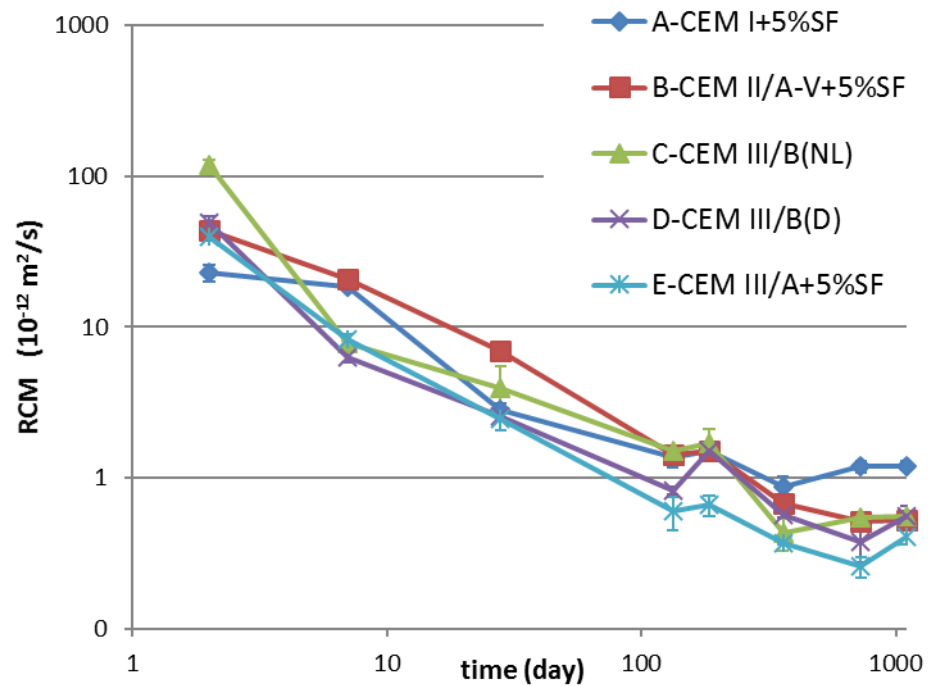


Figure 5 RCM results up to 1100 days; log-log plot.

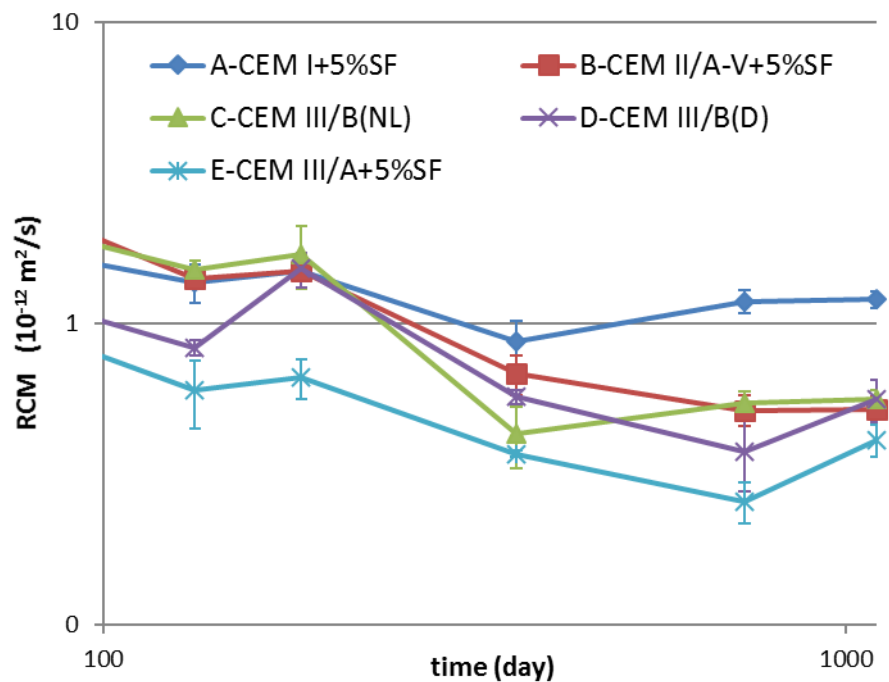


Figure 6 RCM results up to 1100 days, magnification of Figure 5; log-log plot.

Aim of the chloride penetration front is around 25 mm. For samples during the first year, penetration depths ranged from 5 to 31 mm, with a few less than 10 mm and the majority between 15 and 30 mm. For the series at 1100 days an attempt was made to influence the penetration depth by varying the test duration of individual specimens between 5 and 9 days. This was only partially successful. For example,

for series A the test duration was reduced from 7 to 5 days, which reduced the average penetration depth (for single specimens) from 38 to 25 mm. However, for the other mixes the penetration depth did not increase significantly between 7 and 9 days. Nevertheless, in these specimens the penetration depth varied between 11 and 24 mm, which was considered better than having several values below 10 mm.

Generally, the comparison between three individually tested samples was good, with few exceptions. Variation coefficients (VC, $100\% \cdot \text{standard deviation/average}$) varied between 10% and 25%. One exception was mix C-CEM III/B(NL) at 28 days, which had individual values of 3, 3 and $6 \cdot 10^{-12} \text{ m}^2/\text{s}$, resulting in a VC of 38%. Another exception was mix D at 1100 days, with a VC of 36%, due to one specimen having a higher D_{rcm} by almost a factor 2 compared to the other two specimens.

The 2 day specimens of mix A were tested at 15 V for 4 hours, as suggested in Table 3. The penetration depths were small (5 mm), so for the following batches of age 2 days the testing time was increased or the voltage was set at 30 V.

The overall trend of RCM values over time was a strong decrease up to one year age. From one year up to three years, trends are not so clear anymore. Figure 6 suggests that only the values for mix B are really decreasing; values for mix A are quite stable; and for the other mixes it is hard to see, due to variation of the mean values over time.

To provide an overview of the RCM results, the mean values for one to three years testing have been calculated and shown in Table 7. The overall picture is that mix A has the highest value, followed by B, C and D, which are fairly equal; and mix E has the lowest value.

3.3.2 NPRA results

RCM tests have also been performed by NPRA at 120, 360, 730 and 1140 days age. Table 8 provides an overview of their results. Experimental details include vacuum saturation before testing. Figure 7 provides an overview of the RCM results as measured by NPRA.

Table 8 Overview of RCM results obtained at 120, 360, 740 and 1140 days' age by NPRA in triplicate.

| mix | A-CEM I 5%SF | B-CEM II/A-V 5%SF | C-CEM III/B NL | D-CEM III/B D | E-CEM III/A 5%SF |
|--|-----------------|----------------------|-------------------|------------------|---------------------|
| 120 days | | | | | |
| Average RCM (10^{-12} m ² /s) | 0.92 | 1.51 | 1.14 | 1.37 | 0.39 |
| St.dev. RCM (10^{-12} m ² /s) | 0.04 | 0.22 | 0.09 | 0.10 | 0.04 |
| VC (%) | 5 | 15 | 8 | 7 | 10 |
| 360 days | | | | | |
| Average RCM (10^{-12} m ² /s) | 0.80 | 0.62 | 0.73 | 0.88 | 0.31 |
| penetration depth (mm) | 7.2 | 11.0 | 6.6 | 7.8 | 5.8 |
| 730 days | | | | | |
| Average RCM (10^{-12} m ² /s) | 1.0 | 0.50 | 0.73 | 0.69 | 0.38 |
| penetration depth (mm) | 16.5 | 9.0 | 12.7 | 11.8 | 6.9 |
| 1140 days | | | | | |
| Average RCM (10^{-12} m ² /s) | 1.5 | 0.50 | 0.63 | 0.77 | 0.47 |
| penetration depth (mm) | 6.8 | 9.0 | 11.1 | 6.9 | 8.6 |

Note: rounded to 0.01 unit in order not to lose information

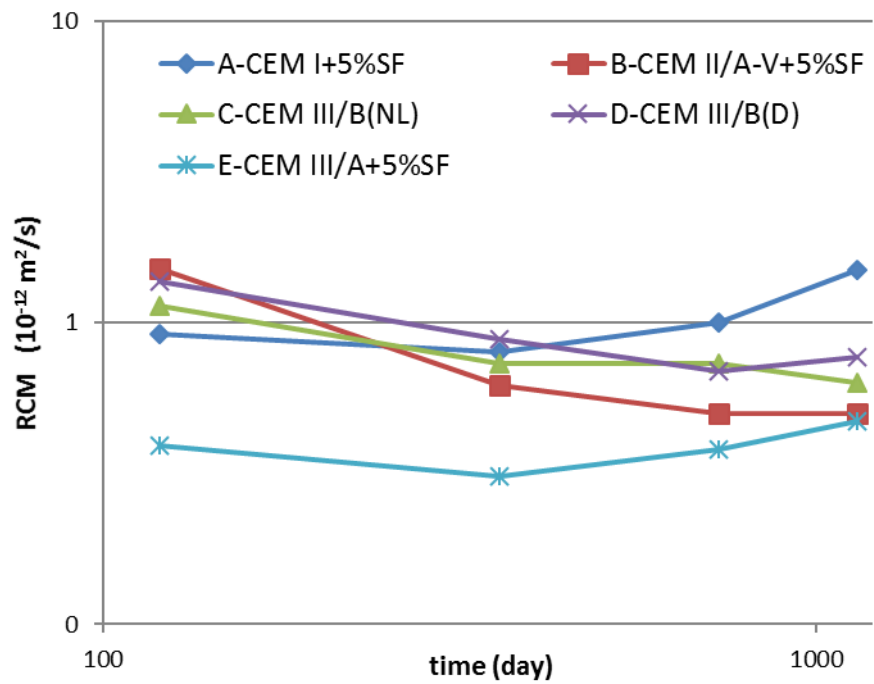


Figure 7 Overview of the NPRA results (RCM).

3.3.3 Observations and comparison

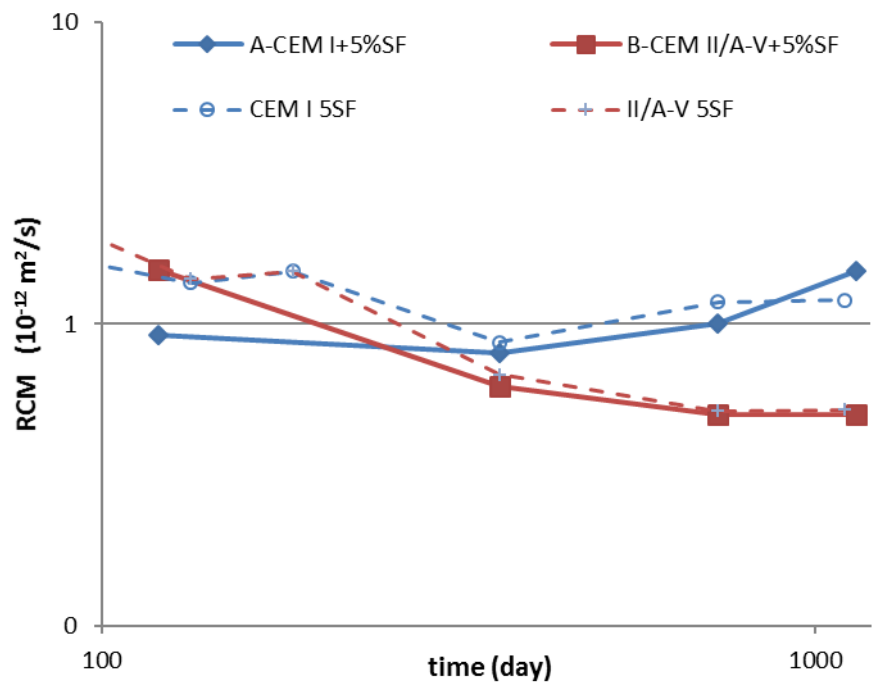


Figure 8 Comparison for mix A and B between NPRA results (solid lines) and TNO results (dashed lines).

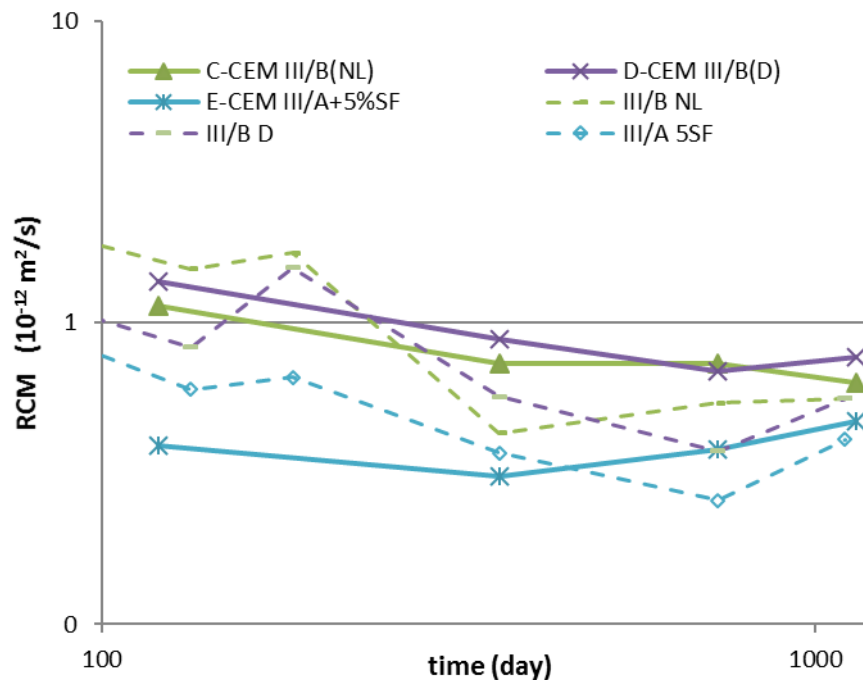


Figure 9 Comparison for mix C, D and E between NPRA results (solid lines) and TNO results (dashed lines).

In Figure 8 and Figure 9 comparisons are provided graphically between the results from TNO and NPRA.

Here some remarks about the testing details are shared.

Two specimens from mix B tested at two years' age were tested in the inverse direction, i.e. the finished surface was exposed to chloride penetration instead of the mould face. This did not seem to have influenced the penetration depth.

Erratic profiles and gas evolution

Some specimens tested at two years of age showed highly erratic penetration depths, showing areas with zero penetration and areas with 20 or more mm. Split and silver nitrate sprayed specimens were re-examined at three years, after storage in a climate room at 20°C and 65% RH. Subsequently, one half of three specimens was split and again sprayed. Observed penetrations showed less variation possibly due to redistribution of the chlorides.

A possible explanation for erratic penetration is accumulation of (hydrogen) gas under the specimens. In order to avoid gas accumulation, at three years age testing the specimen holders were gently moved daily to remove gas. Indeed, considerable gas was seen to escape upon moving the specimen holders. Results from these tests did not show erratic profiles as found at two years. Apparently gently moving the specimen holders is able to prevent disturbances by gas accumulation.

penetration depth and test duration

The testing at 360 days produced relatively low penetration depths, c. 6 to 11 mm after testing for 3 to 5 days. This was taken to suggest applying longer testing

durations. Consequently for three years age testing, the test duration was varied, starting with seven days for one specimen of each mix. After the penetration depths were determined, the test duration for individual specimens in the second testing batch was adjusted, and similarly for the third testing batch. The main result was that for mix A, the testing time was reduced to five days and for the other mixes it was increased to nine days. Overall, penetration depths between 11 and 38 mm were obtained for specimens of three years old.

Test durations and penetration depths for one and three years age testing are reported in Annex C.

3.4 Resistivity results

3.4.1 TNO results

An overview of resistivity results measured on specimens in migration cells before the start of the RCM-test (R_{rcm}) is given in Table 9. Unfortunately, some values noted in the test files showed strong deviations from expected values by at least an order of magnitude. These are probably due to measuring errors. For those cases, the applied cell voltage U was divided by the cell current I_0 to obtain an approximate (estimated) resistivity value (marked red in Table 9). Table 9 provides average values of three specimens and also standard deviations and VC's, except for estimated values. Figure 10 provides a log-log plot of resistivity values. In TNO's experience, for properly made and tested specimens the coefficient of variation for resistivity measurements is normally of the order of 10%. This is valid for all sets of values reported here.

Resistivity tested in the RCM cells clearly increased over time, at least up to about half a year of age. Focusing on one to three year data (Figure 11), the general trend indicates stabilisation, possibly except for mixes B and C. Mix A has the lowest resistivity, Mix E the highest from half a year of age.

Table 9 Overview of resistivity measured on specimens in RCM cells (R_{rcm}), average and standard deviation in Ωm ; VC coefficient of variation in %; values in red/bold/italic were obtained from cell voltage/current (no standard deviation and VC calculated)

| age (day) | | A-CEM I 5%SF | B-CEM II/A-V 5%SF | C-CEM III/B NL | D-CEM III/B D | E-CEM III/A 5%SF |
|-----------|---------|-----------------|----------------------|-------------------|------------------|---------------------|
| 2 | average | 34 | 31 | 26 | 33 | 31 |
| | stdev | | 5 | 13 | 0 | 3 |
| | VC % | | 16 | 50 | 0 | 10 |
| 7 | average | 70 | 38 | 120 | 118 | 80 |
| | stdev | | 8 | 14 | 7 | 4 |
| | VC % | | 21 | 12 | 6 | 5 |
| 28 | average | 179 | 108 | 289 | 317 | 242 |
| | stdev | 4 | 3 | 4 | 3 | 7 |
| | VC % | 2 | 3 | 1 | 1 | 3 |
| 133 | average | 314 | 428 | 525 | 482 | 900 |
| | stdev | | 33 | 81 | 27 | |
| | VC % | | 8 | 15 | 6 | |
| 185 | average | 441 | 450 | 369 | 501 | 1077 |
| | stdev | 22 | | | 5 | 108 |
| | VC % | 5 | | | 1 | 10 |
| 360 | average | 465 | 354 | 324 | 543 | 945 |
| | stdev | 18 | 26 | 14 | 2 | 79 |
| | VC % | 4 | 7 | 4 | 0 | 8 |
| 740 | average | 352 | 886 | 1011 | 551 | 1116 |
| | stdev | 99 | 81 | 32 | 218 | 75 |
| | VC % | 28 | 9 | 3 | 39 | 7 |
| 1100 | average | 393 | 681 | 837 | 807 | 750 |
| | stdev | 40 | 215 | 230 | 107 | 505 |
| | VC % | 10 | 32 | 27 | 13 | 67 |

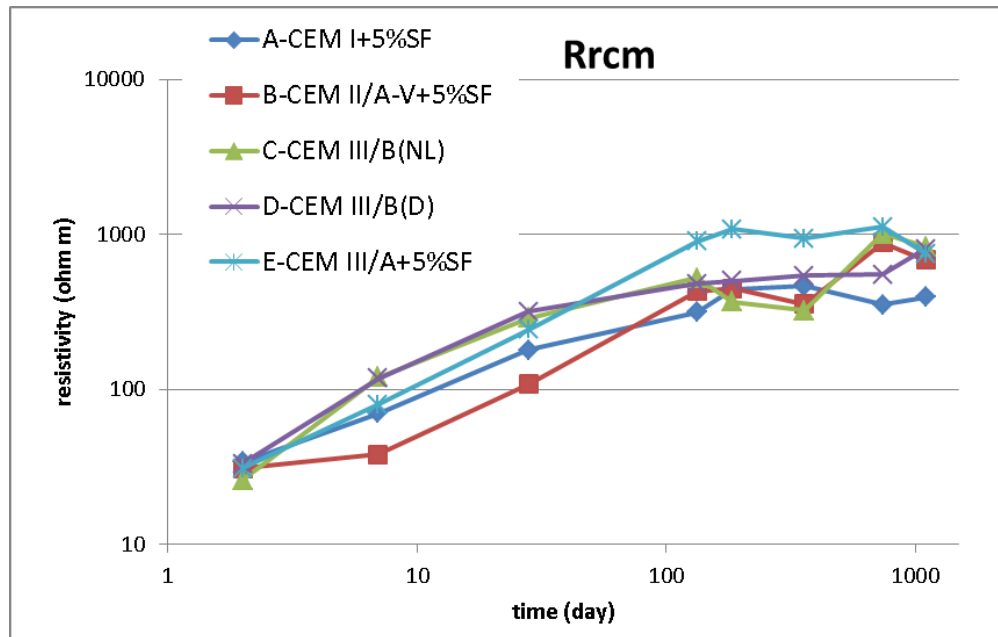


Figure 10 Log-log plot of resistivity values measured on specimens mounted in migration cells (Rrcm) as a function of age.

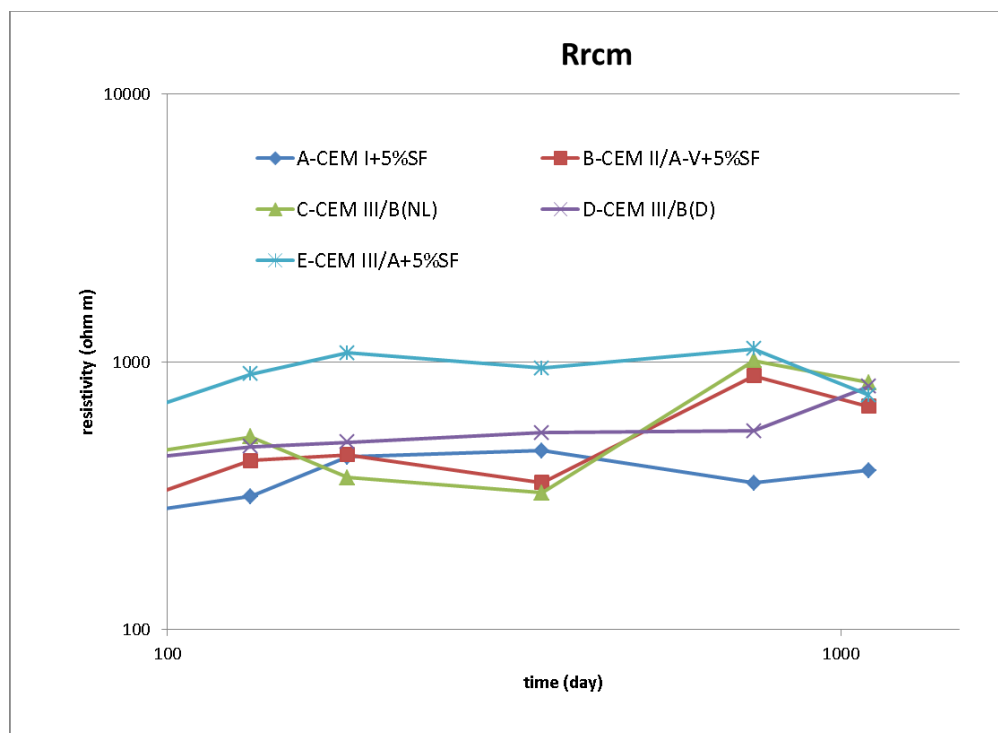


Figure 11 Magnification of Figure 10 at later age range.

Resistivities were also measured using the two-electrode method (TEM) on specimens taken out of the saturated lime water, generally on three specimens. Average R_{tem} values between ages 2 days and 1100 days are reported in Table 10. Coefficients of variation for 740 day R_{tem} measurements varied between 1 and 22%; and for 1100 day measurements between 2 and 12%.

Table 10 Resistivity measured by TEM (R_{tem} , in Ωm) between 2 and 1100 days age.

| age (day) | A-CEM I +5%SF | B-CEM II/A-V +5%SF | C-CEM III/B (NL) | D-CEM III/B (D) | E-CEM III/A +5%SF |
|-----------|---------------|--------------------|------------------|-----------------|-------------------|
| 2 | 30 | 28 | 19 | 35 | 29 |
| 7 | 114 | 37 | 121 | 137 | 84 |
| 14 | 154 | 59 | 198 | 212 | 140 |
| 28 | 203 | 110 | 296 | 339 | 244 |
| 75 | 530 | 419 | 534 | 526 | 591 |
| 270 | 560 | 802 | 843 | 666 | 1439 |
| 360 | 479 | 802 | 781 | 658 | 1251 |
| 740 | 585 | 1300 | 1050 | 974 | 1303 |
| 1100 | 520 | 1146 | 1054 | 951 | 1243 |

Measurements around 56 days were carried out in a single campaign in April, 2014, when specimens had ages between 60 and 90 days (depending on individual mixes' casting date). For simplicity, their age is averaged at 75 days. These results are reported in Table 11.

Table 11 Resistivity by TEM (R_{tem} , in Ωm) of 18 or 19 specimens measured in April 2014 at ages between 60 and 90 days.

| mix | mean | stdev | VC (%) | test date | cast date | age |
|-------------------|------|-------|--------|-----------|-----------|-----|
| A-CEM I 5%SF | 530 | 40 | 8 | 9-apr | 9-1-2014 | 90 |
| B-CEM II/A-V 5%SF | 419 | 52 | 12 | 17-apr | 16-jan | 91 |
| C-CEM III/B NL | 534 | 45 | 9 | 9-apr | 23-jan | 76 |
| D-CEM III/B D | 526 | 70 | 13 | 9-apr | 30-jan | 69 |
| E-CEM III/A 5%SF | 591 | 49 | 8 | 8-apr | 6-feb | 61 |

Figure 12 provides a log-log plot of all TEM results up to 1100 days age.

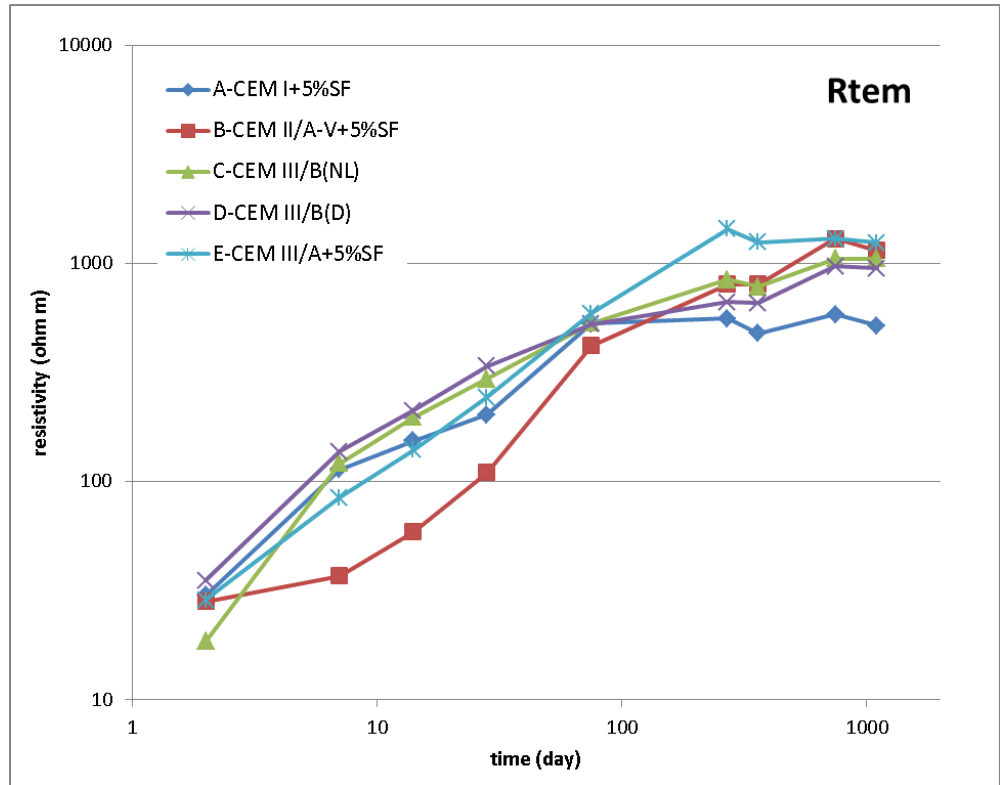


Figure 12 Log-log plot of resistivity values measured on specimens between steel plates (R_{tem}) as a function of age.

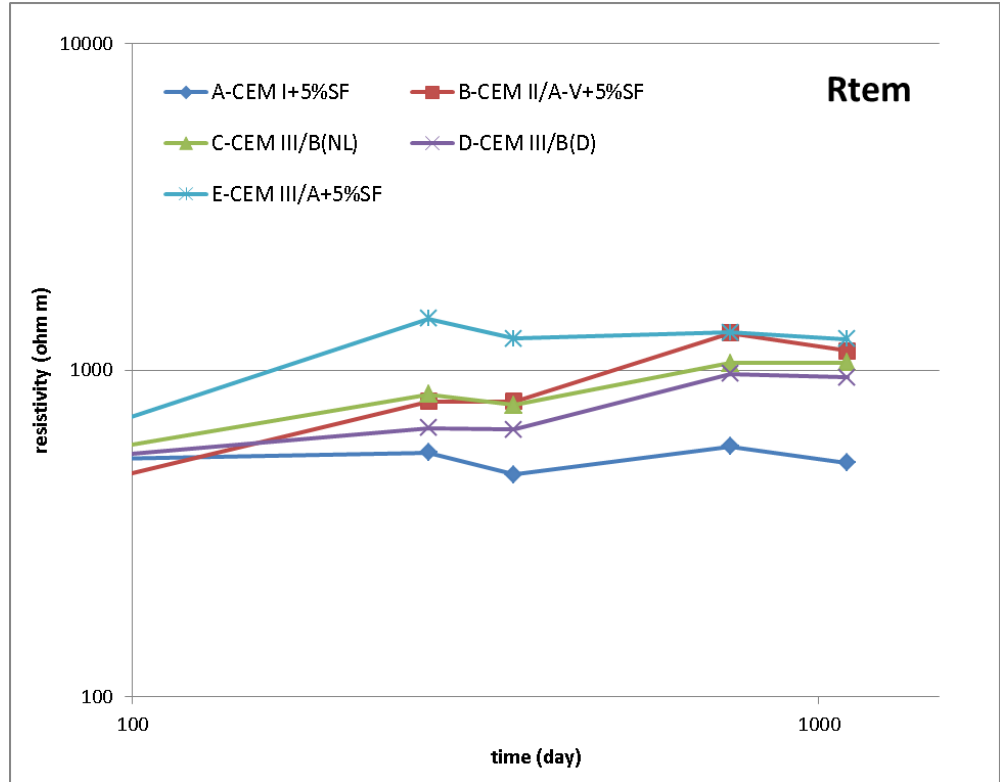


Figure 13 Magnification of Figure 12 for later ages.

3.4.2 NPRA results

Table 12 provides an overview of resistivity results obtained by NPRA between 120 and 1150 days age. All specimens were tested before vacuum saturation.

Table 12 Resistivity in Ω m measured by NPRA at age 120, 360, 730 and 1140 days; supposedly all measured after vacuum saturation; and increase in % between 120 and 1140 days

| mix age (day) | A CEM I +5%SF | B CEM II/A-V +5%SF | C CEM III/B (NL) | D CEM III/B (D) | E CEM III/A +5%SF |
|------------------|---------------------|--------------------------|------------------------|-----------------------|-------------------------|
| 120 | 544 | 536 | 638 | 517 | 1176 |
| 360 | 557 | 1153 | 1073 | 798 | 1393 |
| 730 | 556 | 1169 | 1154 | 972 | 1455 |
| 1150 | 548 | 1364 | 1296 | 1204 | 1550 |

Measurements performed by NPRA at about one year age are presented in Table 13 and compared with the measurement results from TNO.

Table 13 Resistivity measured by TEM (R_{tem} , in Ω m) at one year age at TNO and NPRA.

| mix | NPRA | TNO | | | |
|-------------------|------|------|-------|--------|-----------|
| | | mean | stdev | VC (%) | age (day) |
| A-CEM I 5%SF | 601 | 479 | 30 | 6 | 385 |
| B-CEM II/A-V 5%SF | 1246 | 802 | 58 | 7 | 378 |
| C-CEM III/B NL | 1159 | 781 | 80 | 10 | 371 |
| D-CEM III/B D | 862 | 658 | 77 | 12 | 364 |
| E- CEM III/A 5%SF | 1478 | 1251 | 50 | 4 | 357 |

3.4.3 Observations

Resistivity tested by TEM clearly increased over time, at least up to about half a year of age. Focusing on one to three year data (Figure 13), the general trend indicates stabilisation. Mix A has the lowest resistivity, Mix E the highest from half a year of age. This corresponds very well with resistivity measured in RCM cells.

The resistivity-time plots measured by Rrcm in the RCM cell versus R_{tem} between steel plates are generally similar, which is confirmed by a comparison of results between 2 and 1100 days' age provided in Figure 14 (identified by mix) and Figure 15 (identified by age). For the latter, it should be noted that RCM specimens were vacuum saturated for tests at 360, 740 and 1100 days; specimens tested between 2 and 180 days were not vacuum saturated.

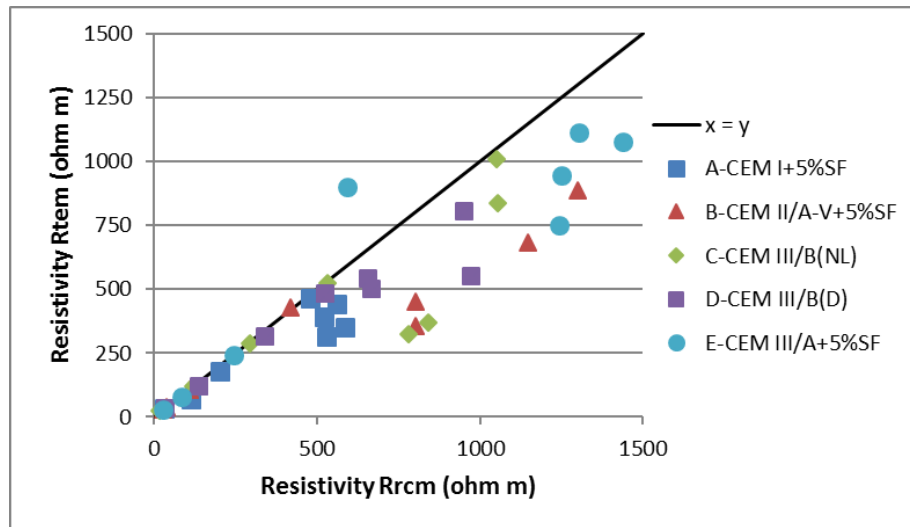


Figure 14 Resistivity measured in migration cells (Rrcm) versus those measured on specimens between steel plates (Rtem) up to 1100 days age in Ωm and line of equality; for each of the five mixes.

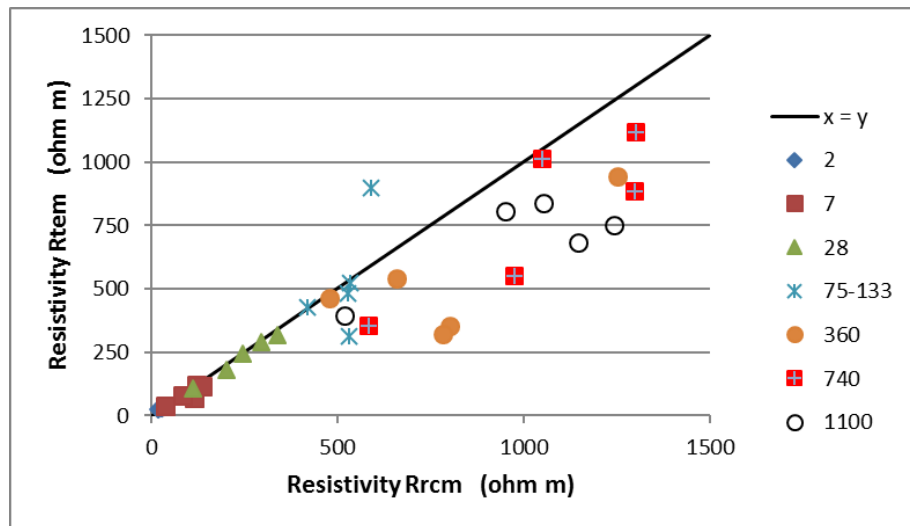


Figure 15 Resistivity measured in migration cells (Rrcm) versus those measured on specimens between steel plates (Rtem) up to 1100 days age in Ωm and line of equality; for different ages.

Comparison with TNO results is not straightforward. On the one hand, NPRA's specimens were not vacuum saturated, while TNO's Rrcm specimens were. On the other hand, NPRA's testing was done between aluminum plates covered with a fabric soaked in acetic acid to provide good electrical contact, while TNO's Rrcm was measured in the RCM cell. Considering this, NPRA's results are thought to be best comparable to TNO's Rtem results (Table 10).

The comparison of NPRA's results with TNO's Rtem results measured between 360 and 1150 days is given in Figure 16 with individual mixes recognisable.

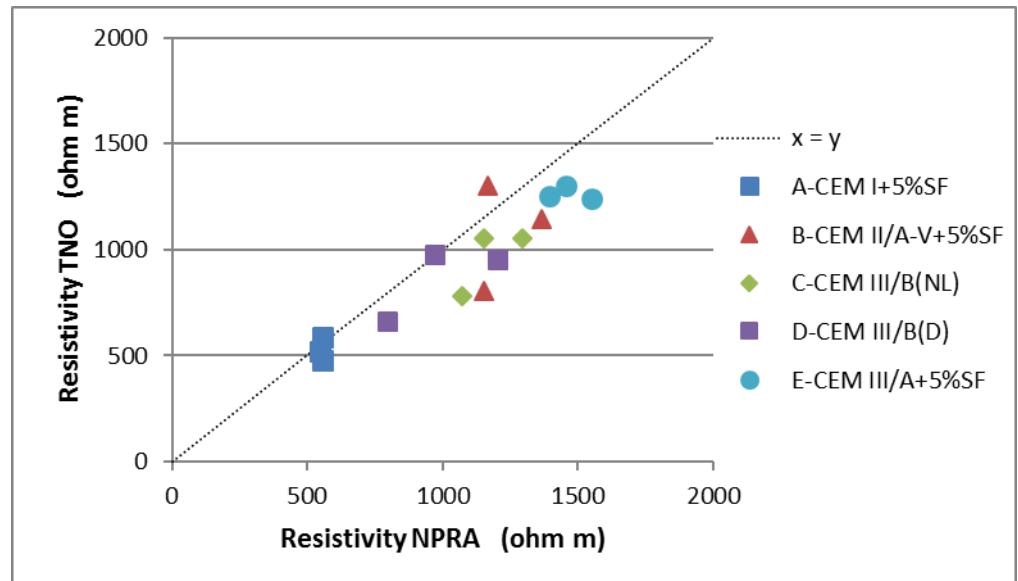


Figure 16 Comparison of resistivity results in Ωm between TNO and NPRA at 360, 730 and 1140 days for measurements between steel plates R_{tem} , which were not vacuum saturated at TNO, and vacuum saturated at NPRA.

3.5 Carbonation results

Carbonation depths of mortar prisms were measured at one, two and three year age. It should be noted that, from 7 days age on, the prisms had been fully exposed (not sheltered) on the roof of the (old) laboratory until the end of 2014. In February 2015 (about one year age) they were placed outside the (new) laboratory in a shelter, and since February 2016 on the roof of the laboratory under a shelter. Table 14 reports carbonation depths as measured on the prisms in the three mould faces (not finished face).

Table 14 Carbonation depth in mm at 360, 740 and 1100 days.

| age (day) | A-CEM I 5%SF | B-CEM II/A-V 5%SF | C-CEM III/B NL | D-CEM III/B D | E-CEM III/A 5%SF |
|-----------|-----------------|----------------------|-------------------|------------------|---------------------|
| 360 | 0 | 0 | 2 | 4 | 1 |
| 740 | 0 | 0 | 1-4 | 1-2 (max 4) | <1 |
| 1100 | 0 | 0 | 3.1 | 2.9 | 1.4 |

4 Discussion

4.1 Regarding RCM

The most important general observations and trends in the results are:

- The migration coefficient (RCM) decreases with increasing specimen age, at least up to one year of age, which is according to the expectation, due to continued hydration of cementitious materials
- However, this ongoing trend slows down or even disappears for ages between one and three years.
- At 28 days all mixes have roughly the same RCM, except mix B-CEM II/A-V+5%SF, whose value is about twice of that of the other mixes (the difference is far beyond the scatter); this is attributed to the relatively slow hydration of fly ash in this mix, which has not really taken off yet at 28 days. Slag reacts more strongly within the first 28 days than fly ash (mixes C, D, E). This is according to expectation. The Dutch slag mix (C) has a relatively high RCM value at 28 days, which is probably caused by one specimen deviating from the other two due to unknown causes.
- At 360 days and up to three years age, all RCM values are around or below $1 \cdot 10^{-12}$ m²/s. Mix A-CEM I 5%SF has the highest value, followed by mix B-CEM II/A-V 5%SF; mixes C-CEM III/B NL, D-CEM III/B D and E-CEM III/A 5%SF have quite similar (low) values, with mix E-CEM III/A 5%SF having the lowest value. From these results, it can be inferred that hydration of slag continues rather long. For mix A without slag or fly ash, no reduction with time beyond one half to one year is observed. For mix B, there may be further potential for reduction due to continued hydration of fly ash.
- Vacuum saturation before RCM testing of specimens at later ages was found to be useful.
- Increasing the test duration beyond 5 days, up to 9 days, for dense mixes shows slightly increased penetration depths. Reduction of the test duration for the more permeable mix (A) from 7 to 5 days produced a reduced penetration depth.
- Gently moving RCM specimen holders daily showed to be useful in avoiding accumulation of gas bubbles under the specimens, which was thought to locally obstruct chloride penetration.

4.2 Regarding resistivity

The most important general observations and trends in the results are:

- Resistivity increases with age, with mix B-CEM II/A-V+5%SF having the lowest value at 7 and 28 days; this mix is catching up around 270 days age and beyond; mix E-CEM III/A 5%SF has the highest resistivity from 360 days up to three years. The lower early values of mix B reflect slower hydration of fly ash, as observed for the RCM values. The same remarks can be made for further potential of resistivity increase for individual mixes as for further RCM reduction.
- Resistivity measured in RCM cells provide similar indications to resistivity measured on specimens between steel plates.

- Reproducibility for RCM is about 10-20%, for resistivity about 10-15%, which are good values for these types of tests.
- The correspondence of results from TNO and NPRA is generally good. Part of the differences found is related to differences in pre-treatment (vacuum saturation or not).

4.3 Regarding carbonation

The most important general observations and trends in the results are:

- Carbonation depth after one year of outdoor exposure was negligible for mixes A, B and E; small for mix C and slightly above the expected (low) value for mix D. It should be noted that non-sheltered outdoor exposure results in rather wet specimens. Under those conditions, carbonation should be expected to be low.
- From one to three year exposure conditions were sheltered (under a roof), which resulted in increased carbonation depths. Carbonation after two and three years was virtually absent in mixes A and B; up to 3 mm after three years in mixes C and D (with c. 70% slag); and about 1.5 mm in mix E (with c. 50% slag and 5% SF). Apparently a higher slag content resulted in increased carbonation.

5 Conclusions

Five mortar mixes have been tested up to three years age for rapid chloride migration (RCM), electrical resistivity and carbonation in outdoor exposure conditions. The mixes comprised different binders: Portland cement plus silica fume (5%), Portland cement plus fly ash (15-25%) and silica fume (5%); high blast furnace slag cement (more than 65%, with cements from two different producers) and medium slag cement (c. 50%) plus silica fume (5%). The water/binder ratio was 0.40 in all cases. The test data allow the following conclusions to be drawn.

RCM was tested from two days age up to three years. At early ages (up to 28 days) differences occurred between mixes. At 28 days all values were rather low, with the fly ash mix having the highest values. From 28 days until one year age, a strong reduction occurred in all mixes. At one year age (after vacuum saturation), all results were quite low, with Portland plus silica fume and Portland, fly ash and silica fume having comparatively higher results. At one year, the mix with a medium slag content and silica fume (mix E-CEM III/A+5%SF) has a very low value. Between one and three years of age, the Portland/fly ash/silica fume mix showed some further RCM reduction.

These trends over time reflect continued hydration of cementitious materials and densification of the pore structure of the mortar. The fly ash is clearly the most slowly reacting component, whose hydration (in submerged conditions) on the other hand appears to continue for a long time. Slag hydrates faster than fly ash and slower than pure Portland cement.

The microscopy results on specimens after two years showed dense microstructure, but also some indications that the mixtures with silica fume did not hydrate completely (agglomerated silica fume particles were observed). If the silica fume would have been better dispersed initially, this could lead to an increased densification at younger ages. If this would also lead to a further increase in resistivity than has been measured in this investigation is unknown at this time.

The development of electrical resistivity confirms the trends observed in the RCM testing: resistivity's become higher with time, further supporting that densification of the material is ongoing. The resistivity of the Portland/SF mix has stabilised by about one year age; that of the fly ash mix continues to increase.

Carbonation was low in all specimens after one year of outdoor exposure (open to wind and rain). From one to three year's age specimens were exposed in sheltered conditions, which resulted in increased carbonation depths for some mixes. Carbonation was virtually absent in the Portland/SF and Portland/fly ash/SF mixes. It was highest in the two high slag mixes (with c. 70% slag); and intermediate in the medium slag mix (with c. 50% slag and 5% SF). Apparently a higher slag content resulted in increased carbonation.

As an overall conclusion, it appears that in terms of performance under chloride load the three slag mixes are equivalent with or even slightly better than the "reference" mix B-CEM II/A-V 5%SF with fly ash and silica fume. The mix with c. 50% slag and 5% silica fume performed equivalent to the two high slag mixes (70%

slag) in terms of chloride penetration resistance and electrical resistivity. In addition, its resistance against carbonation in outdoor sheltered exposure conditions appears to be better than the high slag mixes.

6 References

- [1] NEN-EN 197-1, *Cement - Deel 1: Samenstelling, specificaties en conformiteitscriteria voor gewone cementsoorten*, CEN, 2011.
- [2] T. G. N. M. d. R. Rob B. Polder, "Blast furnace slag cement concrete with high slag content (CEM III/B) - Experiences with the durability in the Netherlands since the 1920s," NPRA report No. 270, 2014.
- [3] R. Polder, "Chloride resistance of slag cement mortars," NPRA report SVV 500, 2016.
- [4] R. R. M. d. L. C. P. B. Polder, "Chloride transport testing of blast furnace slag cement for durable concrete structures in Norway: from 2 days to one year age," in *Ed. H., Beushausen, fib Symposium, Performance-based approaches for concrete structures*, Cape Town, 2016.
- [5] NEN-EN 196-1, "Beproevingmethoden voor cement - Deel 1: Bepaling van de sterkte, Methods of testing cement - Part 1: Determination of strength," CEN, 2005.
- [6] T. & L. J. Nijland, "Microscopic examination of deteriorated concrete," in *Maierhofer, C., Reinhardt, H.W. & Dobmann, G., red., Non-destructive evaluation of reinforced concrete structures. Vol. 1: Deterioration processes and standard test methods*, Oxford, Woodhead, 2010, pp. 137-179.
- [7] NT Build 492, "Concrete, mortar and cement-based repair materials: Chloride migration coefficient from non-steady-state migration experiments," Nordtest, 1999.
- [8] J. P. R. L. G. F. A. Caballero, "Chloride penetration into cementitious mortar at early age," *HERON*, vol. 57, no. 3, pp. 185-196, 2012.

7 Signature

Delft, December 2017



Prof. dr. R.B. Polder
Author



Dr.ir. M.R. de Rooij
Review



Ir. E. Hagen
Research Manager
Structural Reliability

A Mortar mix compositions

Table A.1 Mix codes and binder type.

| mixture | binder | origin | SF | w/b |
|---------|------------|-------------|----|-----|
| A | CEM I | Norway | 5% | 0.4 |
| B | CEM II/A-V | Norway | 5% | 0.4 |
| C | CEM III/B | Netherlands | 0% | 0.4 |
| D | CEM III/B | Germany | 0% | 0.4 |
| E | CEM III/A | Norway | 5% | 0.4 |

Table A.2 Mix compositions

| <u>Mix A</u> | | | density (kg/m ³) | mass (g) | volume (ml) |
|-------------------|-------------|------------------|---------------------------------|----------|-------------|
| binder | Norway | CEM I | 2950 | 450 | 152.54 |
| SF | 5% | | 2200 | 22.5 | 10.23 |
| Standard sand | | | 2650 | 1350 | 509.43 |
| Super plasticizer | 0.65% | 35% solid weight | | 2.93 | |
| w/b | 0.40 | | 1000 | 178.98 | 178.98 |
| Total volume | | | | | 851.19 |
| <u>Mix B</u> | | | | | |
| binder | Norway | CEM II/A-V | 2950 | 450 | 152.54 |
| SF | 5% | | 2200 | 22.5 | 10.23 |
| Standard sand | | | 2650 | 1350 | 509.43 |
| Super plasticizer | 0.33% | 35% solid weight | | 1.5 | |
| w/b | 0.40 | | 1000 | 179.98 | 179.98 |
| Total volume | | | | | 852.18 |
| <u>Mix C</u> | | | | | |
| binder | Netherlands | CEM III/B | 2950 | 450 | 152.54 |
| SF | 0% | | 2200 | 0 | 0.00 |
| Standard sand | | | 2650 | 1350 | 509.43 |
| Super plasticizer | 0.13% | 35% solid weight | | 0.6 | |
| w/b | 0.40 | | 1000 | 180.39 | 180.39 |
| Total volume | | | | | 842.37 |
| <u>Mix D</u> | | | | | |
| binder | Germany | CEM III/B | 3150 | 450 | 142.86 |
| SF | 0% | | 2200 | 0 | 0.00 |
| Standard sand | | | 2650 | 1350 | 509.43 |
| Super plasticizer | 0.13% | 35% solid weight | | 0.6 | |
| w/b | 0.40 | | 1000 | 180.39 | 180.39 |
| Total volume | | | | | 832.68 |
| <u>Mix E</u> | | | | | |
| binder | Norway | CEM III/A | 2950 | 450 | 152.54 |
| SF | 5% | | 2200 | 22.5 | 10.23 |
| Standard sand | | | 2650 | 1350 | 509.43 |
| Super plasticizer | 0.22% | 35% solid weight | | 1.0 | |
| w/b | 0.40 | | 1000 | 180.65 | 180.65 |
| Total volume | | | | | 852.85 |

Note: sand was CEN Standard sand (0-2)

Note 2: typical variations between batches are as follows: sand ± 5 g; cement and water ± 0.5 -1 g

B Experimental details of RCM testing up to 360 days per mix

Mixes

A-CEM I+5%SF
 B-CEM II/A-V+5%SF
 C-CEM III/B(NL)
 D-CEM III/B(D)
 E-CEM III/A+5%SF

Table B.1 Mix A-CEM I+5%SF

| mix & age | A2 | A7 | A28 | A133 | A185 | A360 |
|---|-----|------|------|------|------|------|
| Initial voltage (V) | 15 | 30 | 30 | 30 | 30 | 30 |
| Initial current (mA) | 70 | 67 | 23 | 10 | 10 | 10 |
| Adjusted voltage (V) | 15 | 30 | 40 | 60 | 60 | 60 |
| Adjusted current (mA) | 71 | 67 | 32 | 26 | 20 | 20 |
| Test duration (h) | 4 | 24 | 24 | 24 | 24 | 72 |
| Average penetration (mm) | 5 | 21 | 9 | 6 | 7 | 11 |
| Average RCM [10^{-12} m ² /s] | 23 | 18 | 2.8 | 1.37 | 1.50 | 0.87 |
| St.dev. RCM [10^{-12} m ² /s] | 2.8 | 0.85 | 0.32 | 0.19 | 0.01 | 0.15 |
| VC (%) | 12 | 5 | 11 | 14 | 1 | 17 |

Table B.2 Mix B-CEM II/A-V+5%SF

| mix & age | B2 | B7 | B28 | B133 | B185 | B360 |
|---|-----|-----|------|------|------|------|
| Initial voltage (V) | 30 | 30 | 30 | 30 | 30 | 30 |
| Initial current (mA) | 196 | 130 | 120 | 10 | 9 | 5 |
| Adjusted voltage (V) | 30 | 15 | 20 | 60 | 60 | 60 |
| Adjusted current (mA) | 195 | 63 | 80 | 20 | 18 | 10 |
| Test duration (h) | 6 | 24 | 24 | 66 | 48 | 72 |
| Average penetration (mm) | 23 | 22 | 11 | 17 | 13 | 9 |
| Average RCM [10^{-12} m ² /s] | 44 | 21 | 6.9 | 1.4 | 1.5 | 0.68 |
| St.dev. RCM [10^{-12} m ² /s] | 1.9 | 1.2 | 0.68 | 0.01 | 0.16 | 0.1 |
| VC (%) | 4 | 6 | 10 | 1 | 11 | 15 |

Table B.3 Mix C-CEM III/B(NL)

| mix & age | C2 | C7 | C28 | C133 | C185 | C360 |
|---|-----|------|-----|------|------|------|
| Initial voltage (V) | 30 | 30 | 30 | 30 | 30 | 30 |
| Initial current (mA) | 268 | 35 | 14 | 8 | 8 | 5 |
| Adjusted voltage (V) | 15 | 35 | 60 | 60 | 60 | 60 |
| Adjusted current (mA) | 127 | 41 | 29 | 17 | 17 | 10 |
| Test duration (h) | 6 | 24 | 24 | 65 | 48 | 72 |
| Average penetration (mm) | 31 | 20 | 13 | 18 | 14 | 6 |
| Average RCM [10^{-12} m ² /s] | 120 | 7.8 | 4.0 | 1.5 | 1.7 | 0.43 |
| St.dev. RCM [10^{-12} m ² /s] | 9.9 | 0.28 | 1.5 | 0.01 | 0.43 | 0.1 |
| VC (%) | 8 | 4 | 38 | 1 | 25 | 2 |

Table B.4 Mix D-CEM III/B(D)

| mix & age | D2 | D7 | D28 | D133 | D185 | D360 |
|---|-----|------|------|------|------|------|
| Initial voltage (V) | 30 | 30 | 30 | 30 | 30 | 30 |
| Initial current (mA) | 153 | 35 | 13 | 10 | 9 | 1.3 |
| Adjusted voltage (V) | 15 | 35 | 60 | 60 | 60 | 60 |
| Adjusted current (mA) | 72 | 41 | 27 | 22 | 17 | 2.8 |
| Test duration (h) | 6 | 24 | 65 | 65 | 48 | 145 |
| Average penetration (mm) | 14 | 16 | 28 | 10 | 13 | 15 |
| Average RCM [10^{-12} m ² /s] | 49 | 6.3 | 2.5 | 0.83 | 1.5 | 0.57 |
| St.dev. RCM [10^{-12} m ² /s] | 4.9 | 0.45 | 0.15 | 0.05 | 0.24 | 0.05 |
| VC (%) | 10 | 7 | 6 | 6 | 16 | 10 |

Table B.5 Mix E-CEM III/A+5%SF

| mix & age | E2 | E7 | E28 | E133 | E185 | E360 |
|---|-----|------|------|------|------|------|
| Initial voltage (V) | 30 | 30 | 30 | 30 | 30 | 30 |
| Initial current (mA) | 135 | 160 | 18 | 5 | 5 | 0.5 |
| Adjusted voltage (V) | 30 | 15 | 60 | 60 | 60 | 60 |
| Adjusted current (mA) | 143 | 80 | 37 | 9 | 9 | 1.1 |
| Test duration (h) | 6 | 24 | 24 | 63 | 48 | 144 |
| Average penetration (mm) | 22 | 10 | 21 | 10 | 6 | 10 |
| Average RCM [10^{-12} m ² /s] | 40 | 8.3 | 2.5 | 0.60 | 0.66 | 0.37 |
| St.dev. RCM [10^{-12} m ² /s] | 2.4 | 0.59 | 0.40 | 0.15 | 0.1 | 0.01 |
| VC (%) | 6 | 7 | 16 | 25 | 16 | 2 |

C Experimental details of RCM testing at 1100 days

Table C.1 Test duration, penetration depth and RCM coefficient (in 10^{-12} m²/s) at 1100 days age for individual specimens, average (av), standard deviation (sd) and coefficient of variation (VC) of RCM

| | | | | |
|---|------------------|---------|---------|---------|
| A | time (h) | 169 | 168 | 121 |
| | penetration (mm) | 38 | 32 | 25 |
| | RCM | 1.3E-12 | 1.1E-12 | 1.2E-12 |
| | av RCM | | 1.2E-12 | |
| | sd RCM | | 1.3E-13 | |
| | VC RCM (%) | | 11 | |
| B | time (h) | 169 | 168 | 216 |
| | penetration (mm) | 19 | 17 | 13 |
| | RCM | 6.5E-13 | 5.7E-13 | 3.4E-13 |
| | av RCM | | 5.2E-13 | |
| | sd RCM | | 1.4E-13 | |
| | VC RCM (%) | | 26 | |
| C | time (h) | 169 | 168 | 216 |
| | penetration (mm) | 21 | 17 | 16 |
| | RCM | 7.1E-13 | 5.7E-13 | 4.1E-13 |
| | av RCM | | 5.6E-13 | |
| | sd RCM | | 1.4E-13 | |
| | VC RCM (%) | | 26 | |
| D | time (h) | 169 | 168 | 216 |
| | penetration (mm) | 24 | 12 | 18 |
| | RCM | 8.2E-13 | 4.0E-13 | 4.6E-13 |
| | av RCM | | 5.5E-13 | |
| | sd RCM | | 2.0E-13 | |
| | VC RCM (%) | | 36 | |
| E | time (h) | 169 | 216 | 216 |
| | penetration (mm) | 15 | 18 | 11 |
| | RCM | 4.9E-13 | 4.6E-13 | 2.8E-13 |
| | av RCM | | 4.1E-13 | |
| | sd RCM | | 1.1E-13 | |
| | VC RCM (%) | | 26 | |

Table C.2 Test duration and penetration depth for average of specimens tested at 360 days age

| mix | A | B | C | D | E |
|------------------------|----|----|----|-----|-----|
| test duration (h) | 72 | 72 | 72 | 120 | 120 |
| penetration depth (mm) | 11 | 9 | 6 | 15 | 10 |

D Polarization- and fluorescence microscopy (PFM)

D.1 Method and samples

Polarization-and-fluorescence microscopy (PFM) on thin sections has been performed on all five mortars at an age of 2 years, according to procedures outlined in Nijland & Larbi (2010).

Table D.1 Overview of samples studied.

| Mix code | Binder | Casting date (2014) | Thin section code |
|---------------------|--------------------|---------------------|-------------------|
| A - CEM I+5%SF | CEM I + 5% SF | January 14 | TNO 01792 |
| B - CEM II/A-V+5%SF | CEM II/A-V + 5% SF | January 21 | TNO 01793 |
| C --CEM III/B (NL) | CEM III/B (NL) | January 28 | TNO 01794 |
| D - CEM III/B (D) | CEM III/B (D) | February 4 | TNO 01795 |
| E - CEM III/A+5%SF | CEM III/A + 5% SF | February 11 | TNO 01796 |

D.2 Results

Mortar A has a homogeneous, dense microstructure (Figure D.1) with a relatively high entrapped air content (4-5 vol%, Figure D.1, D.2). The binder is made up by well hydrated, fine grained Portland cement and some silica fume. At least part of the silica fume has agglomerated and did not react (Figure D.3). Possibly, also some silica gel developed (Figure D.4). Only a few narrow microcracks occur. Carbonation is visible by the higher order beige birefringence colours in cross polarized light. Carbonation depth is about 0.1 mm (Figure D.5).

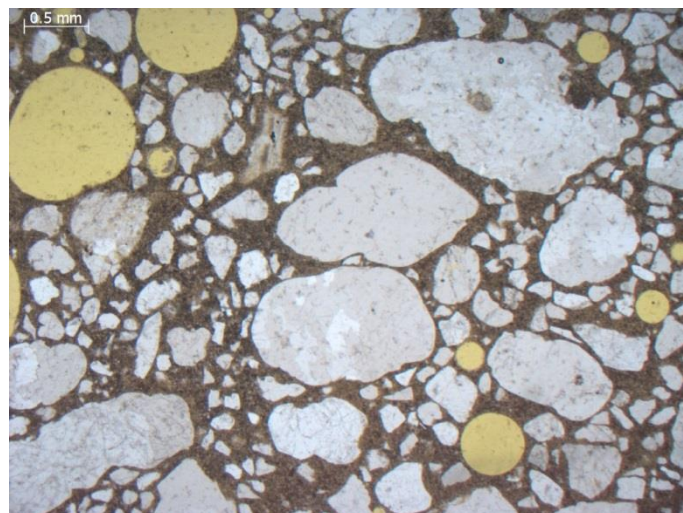


Figure D.1. Microphotograph providing an overview of the microstructure of mortar A (TNO 01792, plane polarized light).

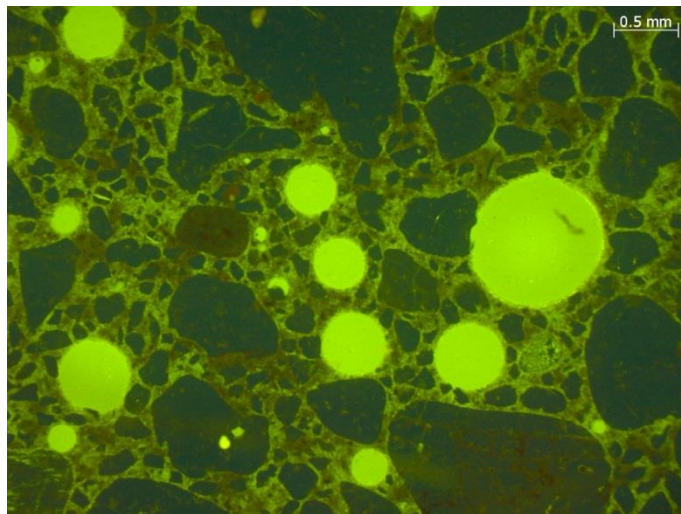


Figure D.2. Microphotograph illustrating the relatively high entrapped air content of mortar A (TNO 01792, UV fluorescence).

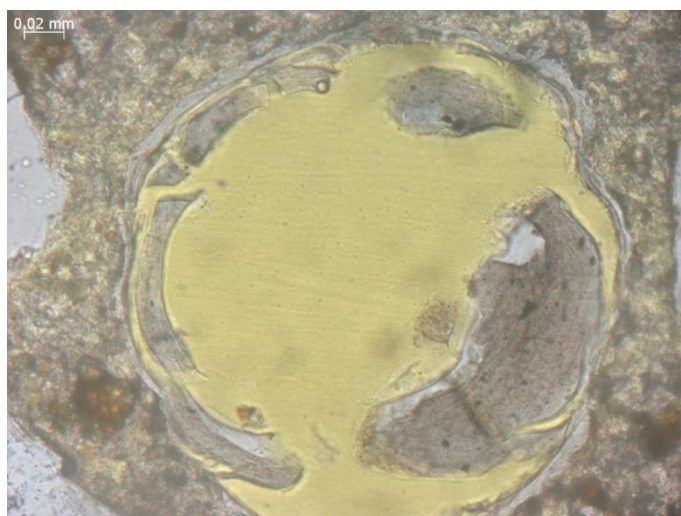
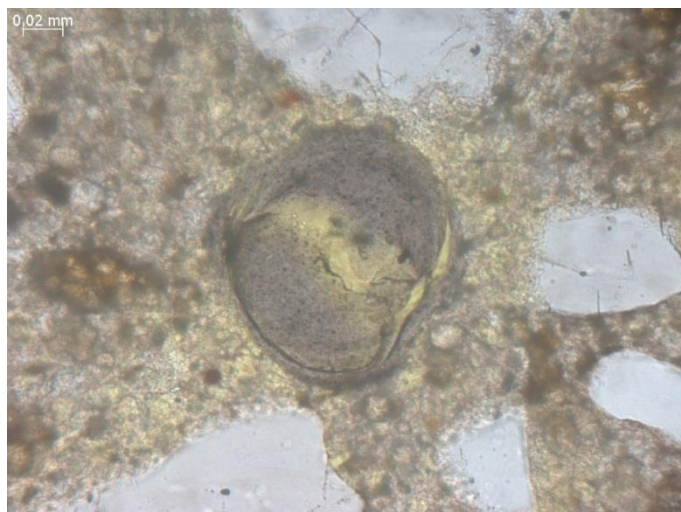


Figure D.3. Microphotographs of agglomerated silica fume in mortar A (TNO 01792, plane polarized light).

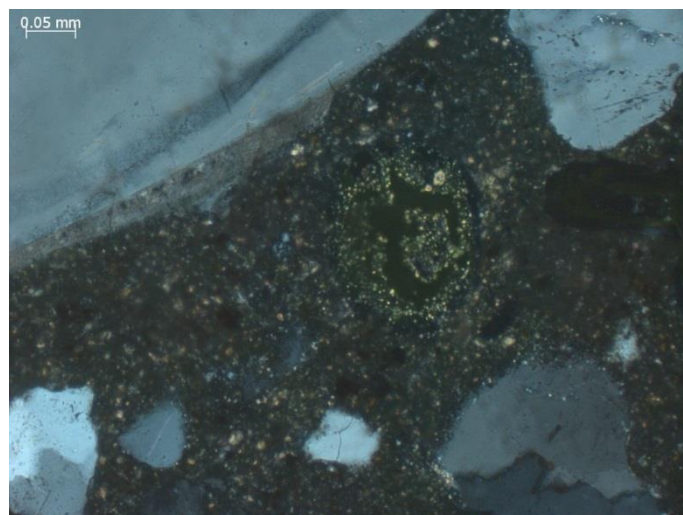
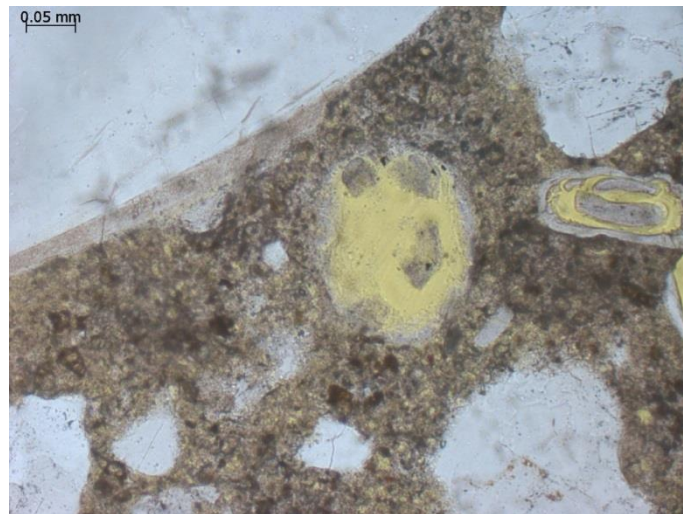


Figure D.4. Microphotograph showing deposition of silica gel (?) at the interface between aggregate and cement paste (TNO 01792, above plane, below cross polarized light).

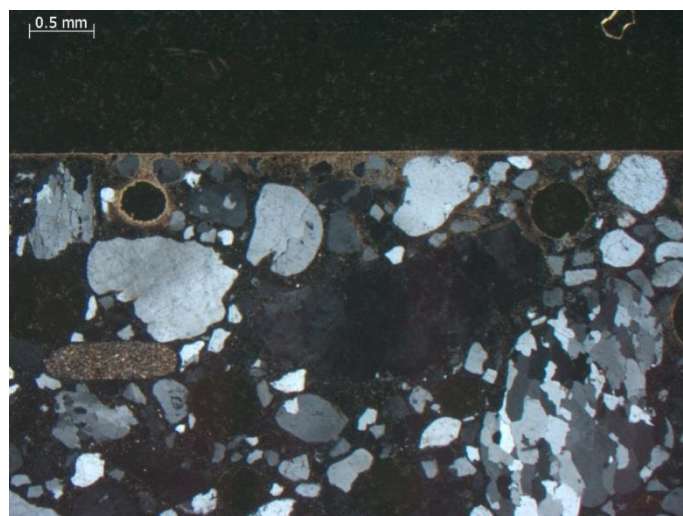


Figure D.5. Microphotograph of very shallow carbonation below the surface of mortar A (TNO 01792, cross polarized light).

Mortar B has a homogeneous microstructure with a relatively high entrapped air content (3-4 vol.%, Figure D.6). The binder of Portland clinker, powder coal fly ash and some silica fume is well hydrated; well-developed portlandite crystals are clearly visible. Silica fume has, at least to a certain amount, agglomerated and, in these agglomerates, did not react. Microcracks are almost absent. Carbonation is about 0.3 -0.5 mm (Figure D.7).

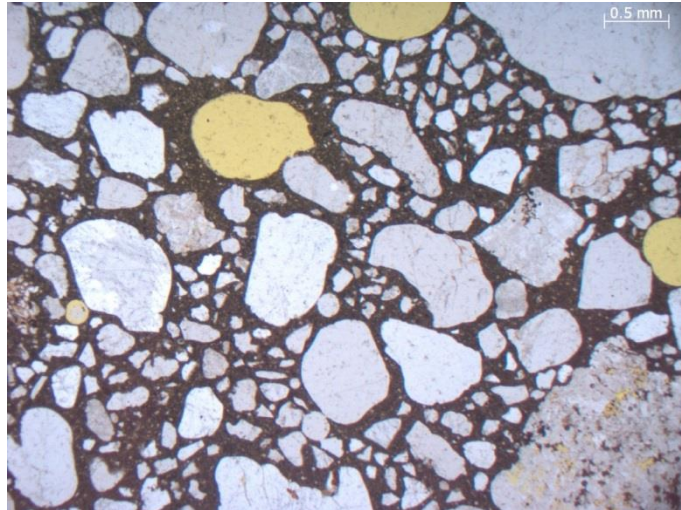


Figure D.6. Microphotograph giving an overview of the microstructure of mortar B with some large air voids (TNO 01793, plane polarized light).

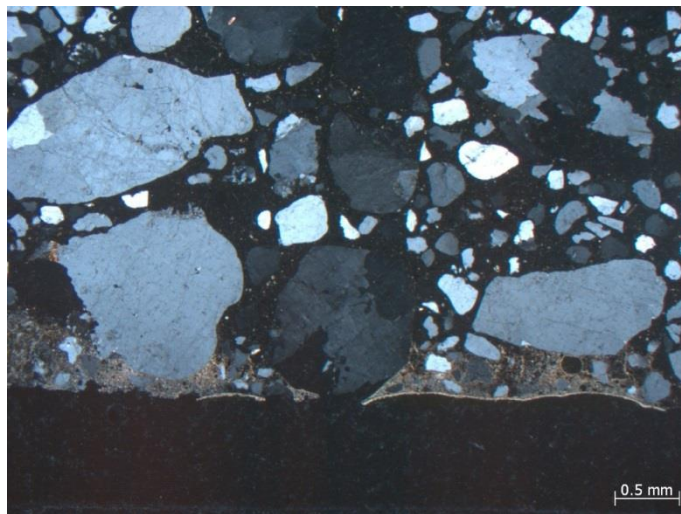


Figure D.7. Microphotograph showing carbonation in the outer layers in mortar B (TNO 01793, cross polarized light).

Mortar C has a homogeneous microstructure with a relatively high entrapped air content (ca. 4 vol.%; Figure D.8). A few very narrow microcracks occur. The cement paste shows a good coherence, the binder of Portland clinker with a high ground granulated blast slag content (i.e. CEM III/B; Figure D.9) is well hydrated. Carbonation depth is about 1-2 mm (Figure D.10).

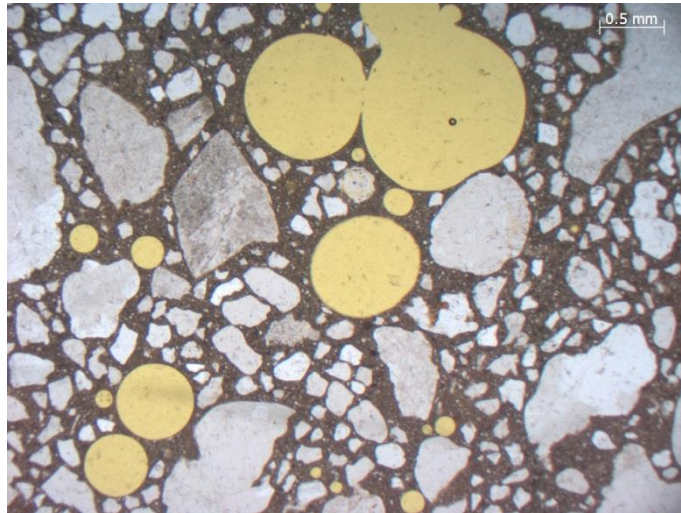


Figure D.8. Microphotograph providing an overview of the microstructure of mortar C, with high entrapped air void content (TNO 01794, plane polarized light).

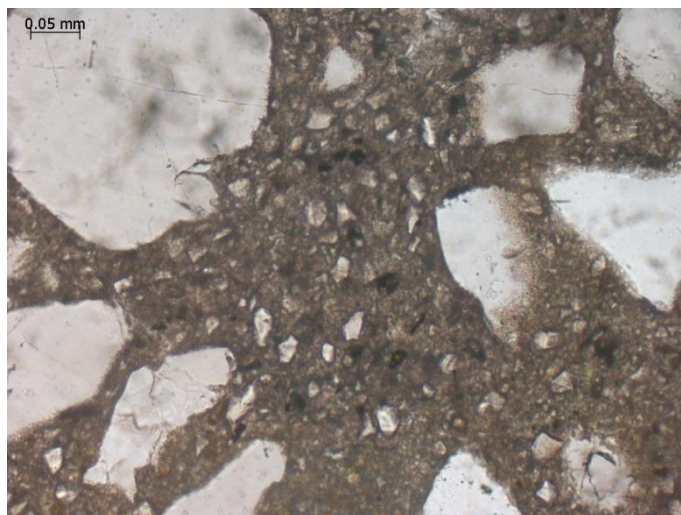


Figure D.9. Microphotograph providing a detail of the cement paste of mortar C (TNO 01794, plane polarized light).

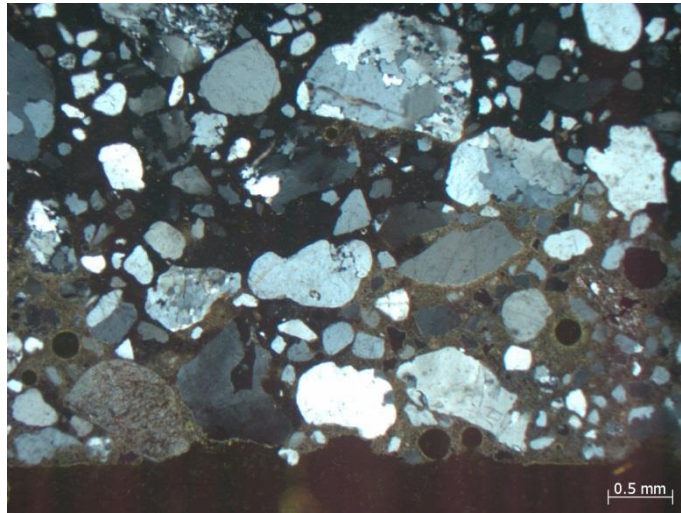


Figure D.10. Microphotograph showing carbonation in the lower (outer) zones in mortar C (TNO 01794, cross polarized light).

Mortar D has a microstructure and binder comparable with that of mortar C (Figures D.11, D.12), with several narrow microcracks. Entrapped air content is 4 -5 vol% (Figure D.11), carbonation depth 1-2 mm (Figure D.13).

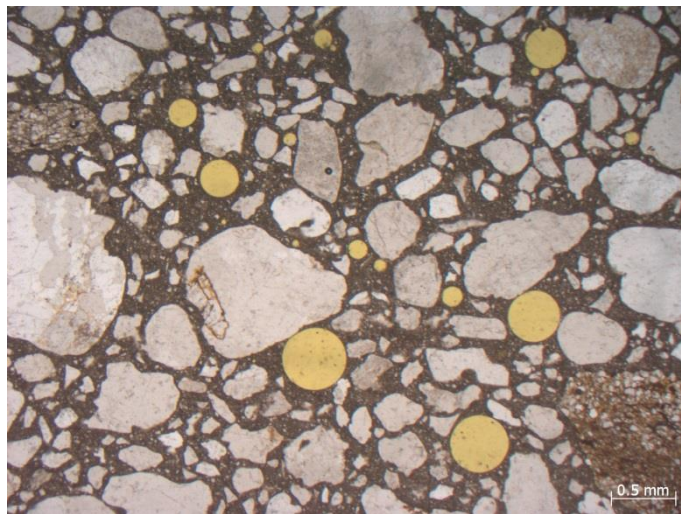


Figure D.12. Microphotograph providing an overview of the microstructure of mortar D, with relatively high entrapped air void content (TNO 01795, plane polarized light).

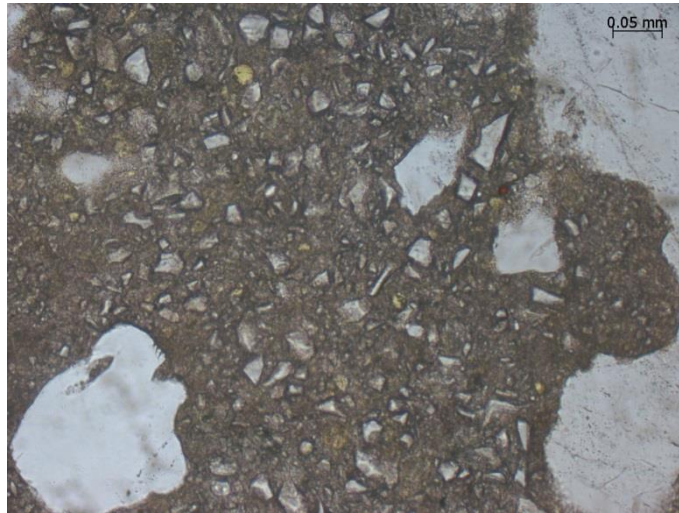


Figure D.12. Microphotograph providing a detail of the cement paste of mortar D (TNO 01795, plane polarized light).

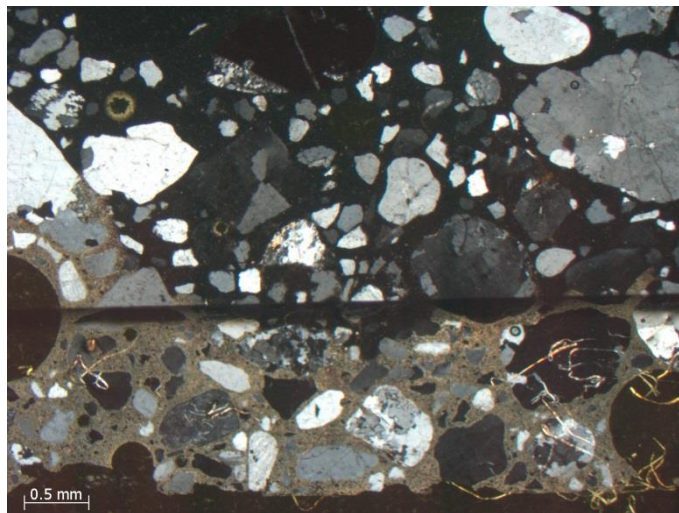


Figure D.13. Microphotograph showing carbonation depth in mortar D (TNO 01795, cross polarized light). Note: horizontal band is artefact (edge of covering glass), hairy shapes in lower part are preparation artefacts

Mortar E also has a homogeneous microstructure, with a binder made up by Portland clinker, a moderate amount of ground granulated blast furnace slag (i.e. CEM III/A) and some silica fume. At least part of the latter agglomerated and did not react. Otherwise, the binder is well hydrated. Some narrow microcracks occur. Carbonation depths are typical about 0.5 mm; locally, depths of about 2-3 mm are reached.

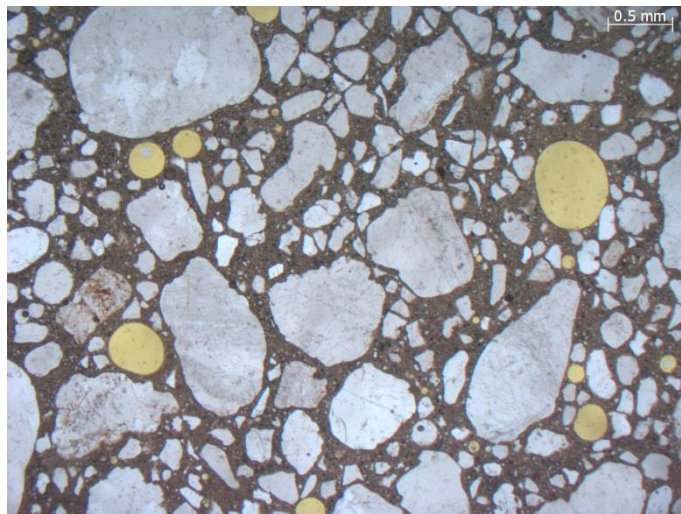


Figure D.14. Microphotograph with overview of the microstructure of mortar E (TNO 01796, plane polarized light).

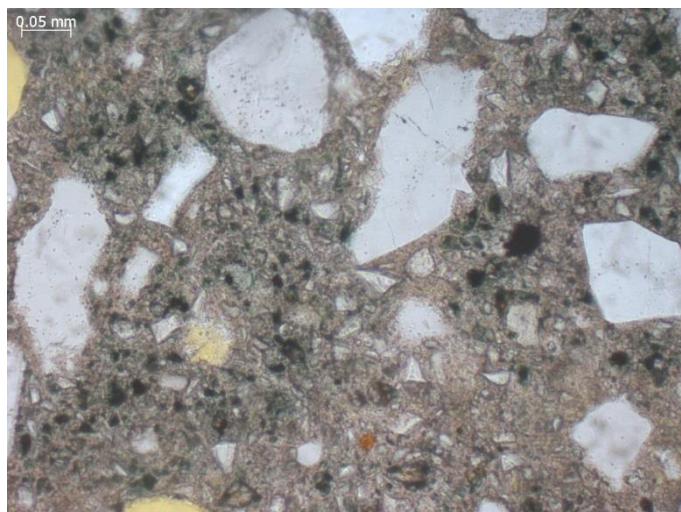


Figure D.15. Microphotograph with detail of the cement paste in mortar E (TNO 01796, plane polarized light).

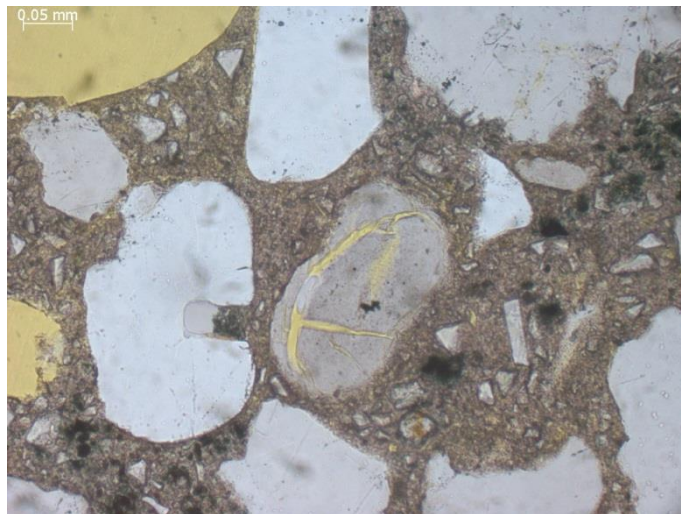


Figure D.16. Microphotograph with agglomerated silica fume (centre) in mortar E (TNO 01796, plane polarized light).

D.3 References

Nijland, T.G. & Larbi, J.A., 2010. Microscopic examination of deteriorated concrete. In: Maierhofer, C., Reinhardt, H.W. & Dobmann, G., red., Non-destructive evaluation of reinforced concrete structures. Vol. 1: Deterioration processes and standard test methods. Woodhead, Oxford, 137-179.



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