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CHLORIDE TRANSPORT TESTING OF BLAST FURNACE SLAG CEMENT FOR DURABLE CONCRETE STRUCTURES IN NORWAY: FROM 2 DAYS TO ONE YEAR AGE

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ABSTRACT

Blast furnace slag cement (BFSC) has been used in reinforced concrete structures in marine and road environment in The Netherlands for nearly a century. Experience is good and long service lives can be obtained. In Norway experience with BFSC is scarce. In The Netherlands, a high resistance against chloride penetration and a high electrical resistivity, was demonstrated both in the field and in the laboratory for CEM III/B with 65-75% of slag by mass of binder. Such a cement is not readily available on the Norwegian market. The introduction of CEM III/A with c. 50% slag is anticipated. An experimental program was carried out to study the properties of five mortar mixes made with: CEM III/B (two brands); a CEM III/A (with c. 50% slag) with silica fume; and a CEM I and a CEM II/A-V (fly ash cement), both with silica fume. Rapid Chloride Migration (RCM) and electrical resistivity were tested at ages between two days and one year. RCM values showed consistent decrease and resistivity showed consistent increase between two days and one year. The program is intended to collect data up to three years age. The ultimate goal is to provide a basis for evaluating the use of slag cement in Norway for durable concrete structures. The paper reports the results and discusses the preliminary conclusions.

Keywords: service life; reinforcement corrosion; blast furnace slag cement; chloride penetration; electrical resistivity; new structures.

1. Introduction

In many countries including Norway, using blast furnace slag in cement is a novelty and long term experience is lacking. In The Netherlands, however, blast furnace slag cement (BFSC), nowadays called CEM III/B with at least 65% slag, has been used to build reinforced concrete structures in marine and road environments for nearly a century. The experience is good and structures with long service lives can be obtained, as has been shown by several field studies [Wiebenga 1980, Bamforth & Chapman-Andrews 1994, Bijen 1996, Polder & Rooij 2005, Polder et al. 2014]. Particularly coastal bridges could benefit from using a cement type with an inherent high chloride penetration resistance. Although slag cement use in Norway is limited, blast furnace slag cement (CEM III/B) has been used in a few projects. One example is the Tjuvholmen project in Oslo, which comprises a number of business and apartment buildings built on concrete substructures positioned in seawater [Gjørv 2012]. Portland slag cement (CEM II/B-S) with 30 % slag has been in use in Norway for a decade in all kind of structures including coastal bridges. In the framework of the Norwegian Public Road Administration (NPRA) program Durable Structures, a desk study was carried out in order to investigate the potential for use of BFSC in Norway [Polder et al. 2014]. Based mainly on work in The Netherlands, the performance of slag cement with regard to chloride induced corrosion stands out positively. This is caused by a high resistance against chloride penetration, prolonging the initiation period; and a high electrical resistivity reducing the corrosion rate, thus prolonging the propagation period. Both effects have been demonstrated in the field as well as and in the laboratory [Polder & Peelen 2002, Visser & Polder 2006, Osterminski et al. 2012, Polder 2012]. Under exposure conditions relevant to The Netherlands, neither carbonation nor frost-thaw damage are problems in practice. The use of CEM III/B has, also in practice, proven to be an effective preventive measure against deleterious alkali-silica reaction [Polder et al. 2014 and references therein]. Due to the high slag content, related CO_2 emission and embedded energy are lower than for Portland cement.

Dutch experience is mainly based on CEM III/B with 65-75% of slag by mass of binder. Such a cement is not in general use in Norway, but only available for special purposes. There are restrictions on the use of these cement types according to national regulations (NS-EN 206 and NPRA guidelines), in particular related to salt/frost resistance. However, in the future introduction of CEM III/A with c. 48% slag is anticipated in Norway. In order to compare performance of this cement with that of CEM III/B and binders currently used in Norway, an experimental program was drawn up to study the properties of concrete made with two CEM III/B's (one Dutch, one German); CEM III/A (with 48% slag), CEM I (Portland cement) and CEM II/A-V (fly ash cement), the latter three with addition of 5% silica fume to the mixture. This program is aimed at determining basic chloride transport properties of mortars prepared with the binders, from early age up to several years. The ultimate goal is to provide a basis for evaluation of use of slag cement in Norway for durable marine and road structures. This paper reports results from the first year of testing. The complete program will run for three years.

2. Materials, methods and testing schedule

2.1 General

The overall goal was to obtain data 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. Regarding this paper, mortar specimens were made with five binders and were tested for chloride penetration resistance at ages 2 days, 7 days, 28 days, c. 130 days, 180 and 360 days, in triplicate. In addition, electrical resistivity was tested at 9 ages between 2 days and one year; either in triplicate or on all available specimens (10 – 20).

2.2 Materials

Specimens were prepared according to the standard for testing of cement [NEN-EN 196-1-2005], however, with a water-to-binder ratio of 0.40 instead of 0.50 in order to stay close to common Norwegian concrete technology. The mortar had a composition of cement:sand:water 1:3:0.4 (by mass), made using rounded siliceous sand of 0-2 mm. Mixing and casting were carried out according to the standard. A superplasticiser was added in order to obtain the same workability of all mixes. Its dosage was determined using trial mixes. A relatively small mixer was used to prepare the mortar and about 20 batches were made to cast all specimens for each individual mix. Binders used were:

- CEM I 52.5 N (LA) Rapid from Aalborg with 5% silica fume, denoted with mix code A-CEM I+5%SF.
- CEM II/A-V 42.5 N Anlegg FA from Norcem (17% fly ash), with 5% silica fume, mix code B-CEM II/A-V+5%SF.
- CEM III/B 42.5 N from the Netherlands (71% slag), mix code C-CEM III/B(NL).
- CEM III/B 42.5 N-SR/LH/NA from Germany (75% slag), mix code D-CEM III/B(D).
- CEM III/A 42.5 N-NA from Germany (49% slag) with 5% silica fume, mix code E-CEM III/A+5%SF.

2.2 Methods

Specimens were tested for chloride penetration resistance using the rapid chloride migration (RCM) test according to [Tang & Nilsson 1992, NTBuild 492 1999]. Specimens were cast in PVC cylinder moulds with 100 mm diameter and 50 mm height, in order to avoid drilling and cutting. 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 at 20°C until the time of testing. Specimens were not vacuum saturated, except those tested at 360 days age. Voltage and time were chosen as per NTBuild 492, except for specimens at young age. For young specimens, voltage and time were chosen based on previous work on young mortar [Caballero et al. 2012].

Resistivity was tested using an AC resistance meter at 120 Hz with the following procedures:

- 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 (no vacuum saturation); the resistivity was calculated using the geometrical cell constant (surface area/length) [Polder et al. 2000, Polder 2001]; the result is denoted Rtem.

2.3 Parallel testing

Specimens were sent to NPRA for parallel testing in their laboratory at two ages. These results are not reported here for simplicity.

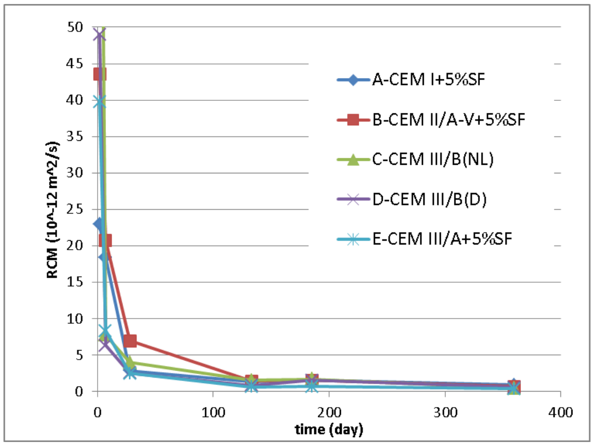
3. Results

An overview of RCM results is given in Table 1. Penetration depths are added for 360 days age specimens and the duration of the test is mentioned to allow discussion of applying the test to mature materials. The RCM results are graphically shown in Figure 1 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 time. Chloride penetration depths ranged from 5 to 31 mm, with a few less than 10 mm and the majority between 20 and 30 mm. Generally, correspondence between three tested samples was good, with one exception (mix C at 28 day with 3, 3 and 6*10^{-12} m^2/s). Variation coefficients (VC, 100%*standard deviation/average) were generally about 10%. Companion specimens tested at NPRA at 90 and 360 days, all with vacuum saturation, produced generally similar results, although slight differences were observed.

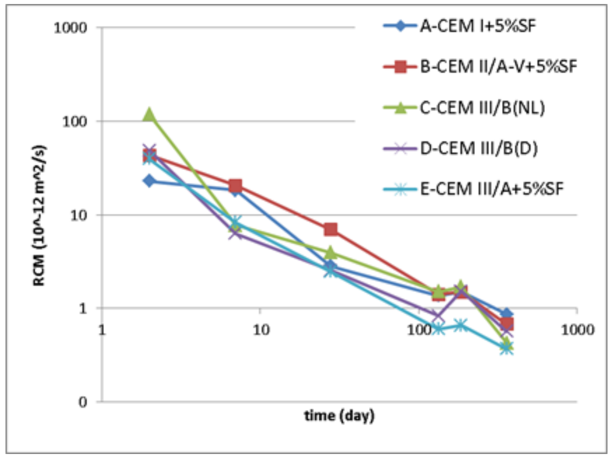
Table 1. Overview of RCM results obtained at TNO; mean and standard deviation in (); penetration depth in specimens at 360 days

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)
	penetration depth (mm)				
360 #	11 *	9 *	6 *	15 $	10 $

#specimens were vacuum saturated before testing * tested for three days $ tested five days



(a) linear plot (mix C at 2 days age is out of scale at 119)



(b) log-log plot

Fig. 1. RCM results up to 360 days

Resistivities were measured using the two-electrode method (TEM) on specimens taken out of the saturated lime water, either on three specimens or on the complete set of available specimens. Resistivities (Rtem) measured at ages between 2 and 360 days are reported in Table 2. Figure 2 provides a log-log plot of all TEM results up to 360 days age.

Table 2. Resistivity measured by TEM (Rtem, in Ωm) between 2 and 360 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

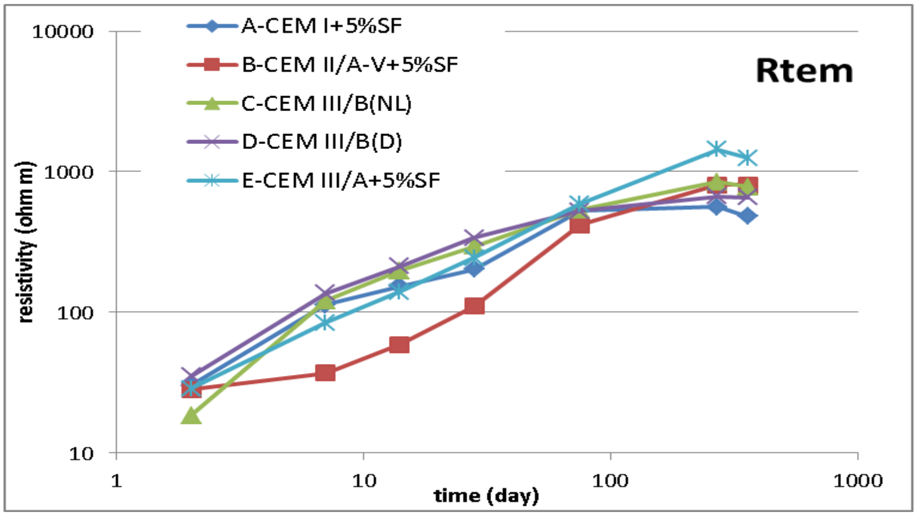


Fig. 2. Resistivity measured on specimens between steel plates (Rtem) up to 360 days age in Ωm, log-log plot.

4. Discussion

The most important general observations and trends in the results can be discussed as follows.

The migration coefficient (RCM) decreases with increasing specimen age, which is according to the expectation, due to continued hydration of cementitious materials.

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. Without the 5 % silica fume addition, the value would have been even higher. Silica fume contributes quite a lot in this early phase [Thomas et al. 1999].

Slag reacts more strongly within the first 28 days than fly ash (mixes C, D, E). This is according to expectation [Polder 2012, Polder et al. 2014]. 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 age, all RCM values are below $1*10^{-12}$ m^2/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. Differences between A, B and (C, D, E) are statistically significant (about one standard deviation); differences between C, D and E are probably not significant. From these results, it can be inferred that hydration of slag continues rather long. For mix A without slag or fly ash, it remains to be seen if the value will further reduce with time. For mix B, there is further potential for reduction due to continued hydration of fly ash. According to literature fly ash, maybe in particular in combination with silica fume, causes a very large reduction in diffusivity in the long term [Thomas et al. 1999]. In [Larsen 2006] diffusion coefficients (D) from profile fitting after 2,5 years to 10 years were documented for different concretes exposed to marine environment. A three-powder mix containing CEM I, silica fume and fly ash had the lowest diffusion coefficient at 10 years age and the strongest reduction between 2.5 and 10 years of all mixes. All other mixes, composed of CEM I from various origins and silica fume in various dosages, showed limited reduction of D over this period.

Regarding the RCM test, the following observations were made. Vacuum saturation before testing of specimens at later ages was found to be useful. Related to low RCM values at 360 days, the observed chloride penetration depths after 3 to 5 days of polarization were quite low. It appears that for these materials with high chloride transport resistance, even at 60 V testing for at least five days is required, and longer testing times may be needed at later ages.

Resistivity increases with age, with mix B-CEM II/A-V+5%SF having the lowest value at 7, 14 and 28 days; this mix is catching up around 75 days age and beyond; mix E-CEM III/A 5%SF has the highest resistivity at 360 days. 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.

Reproducibility for RCM is about 10-15%, for resistivity about 10%, which are good values for these kind of tests.

The correspondence of results from TNO and NPRA was generally good. Part of the differences found is thought to be related to differences in pre-treatment (vacuum saturation or not). Initially TNO did not perform vacuum saturation. When TNO did apply vacuum saturation the differences between the RCM results of TNO and NPRA became smaller.

5. Conclusions and outlook

Five mortar mixes have been tested during one year for rapid chloride migration (RCM) and electrical resistivity. The mixes comprised different binders: Portland cement plus silica fume (5%), Portland fly ash (19%) cement plus silica fume (5%); blast furnace slag cement with a high slag content (71 or 75%), from two different producers and medium slag (c. 50%) cement 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 one year. At early ages substantial differences occurred between mixes. At 28 days all values were rather low, with the fly ash mix/silica fume 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. These trends over time reflect continued hydration of cementitious materials, in particular slag and fly ash, and densification of the pore structure of the mortar. It should be noted that according to a study by [Larsen 2006], Portland plus silica fume material (without fly ash) would not show such a strong reduction over longer periods.

The development of electrical resistivity over time confirms the trends observed in the RCM testing: resistivities become higher with age, further supporting that densification of the material is ongoing. The lowest value at one year age is for the Portland/fly ash/silica fume mix. This mix probably has a high potential for a strong further densification.

As a preliminary overall conclusion based on results of up to one year age, 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. In Dutch practice, fly ash cement would be used with at least 25% of fly ash (CEM II/B-V) without silica fume. Experience with silica fume is low in The Netherlands. On the other hand, a mix with 15% fly ash and 5% silica fume was tested in the laboratory in the 1990s and proved to have good chloride penetration resistance [Polder 1996, Polder et al. 2014], which corresponds to Norwegian findings in marine exposure [Larsen 2006].

Testing for RCM and resistivity at later ages up to 3 years of age is foreseen. It is possible that significant differences between the five mixes will develop. For RCM testing of these dense materials, vacuum saturation is needed. Furthermore for RCM testing, applying a high voltage (60 V) and a long testing time (5, 7 or even more days) is recommended. Exposure to natural carbonation is ongoing (outdoor sheltered) and testing at 2 and 3 years age is foreseen. Finally, characterization of the microstructure using polarization and fluorescence microscopy of both carbonated and non-carbonated materials is planned.

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