International RILEM Conference on Materials, Systems and Structures in Civil Engineering
Conference segment on Concrete with SCM's
22-24 August 2016, Technical University of Denmark, Lyngby, Denmark

DURABILITY OF CONCRETE MADE WITH TERNARY CEMENTS CONTAINING SLAG OR FLY ASH AND LIMESTONE FILLER

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Abstract

The durability of concrete made with ternary cements containing Portland cement (OPC), blast-furnace slag (S) or fly ash (FA) and limestone filler (L) has been investigated according to a comparative approach to assess the suitability for use of these composite cements for concrete. Eight blended cements of different proportions of OPC (from 45% to 65%), S and FA (from 10% to 30%) and L (from 5% to 35%) were selected and compared with five reference cements (CEM I 52.5 R HES, CEM II/B-M (L-S) 32.5 R, CEM II/B-M (LL-S-V) 32.5 N, CEM II/B-V 32.5 R and CEM III/A 42.5 N LA). Two types of mixes were produced using constant water to cement (*w/c*) ratios of 0.45 and 0.55 and cement contents of 340 kg/m³ and 300 kg/m³ respectively. Carbonation, freeze-thaw with de-icing salts, chloride diffusion and sulfate attack tests were carried out on concretes and mortars. The results indicate that a high content of slag and fly ash reduces chloride diffusion and sulfate attack. Resistance to carbonation and freeze-thaw decreases with limestone filler content. Ternary cements containing slag seem overall to give a better concrete durability than those containing fly ash.

1. Introduction

The development of alternative binders such as binary and especially ternary cements has rapidly increased these past decades. Blast-furnace slag (S) and fly ash (FA) are by-products commonly used as supplementary cementitious materials (SCMs). In Belgium, the production of these by-products tends to decrease while limestone filler (L) is abundantly available.

Several studies investigated the synergetic action of these SCMs [1–5]. It has been found that a small dosage of limestone filler could have a beneficial effect on the early age properties of concrete, while fly ash and blast-furnace slag contribute to the long-term mechanical properties as pozzolanic reaction rate is slower than hydration of Portland cement (OPC). These antagonist properties make these ternary mixes particularly interesting. These SCMs are also favourable to sulfate resistance [6] but have a negative effect on carbonation [7]. Some effects of ternary cements on concrete durability are not thoroughly investigated yet, namely frost with de-icing salts resistance [8]. The European standard EN 197-1 [9] includes requirements for cements for use in concrete. Questions arise as to whether these requirements are sufficient to use these cements in all applications. The aim of this study is to evaluate the performance of concrete made with newly developed ternary cements OPC-S-L and OPC-FA-L, according to a comparative approach to assess their suitability for use for concrete. This approach is based on the determination of the performance equivalence of concretes made with these ternary cements compared to reference concretes made with industrial cements, which satisfy the requirements of standard EN 197-1. This paper focuses on the durability properties of these concretes. Carbonation, freeze-thaw with de-icing salts, chloride diffusion and sulfate attack tests were carried out on concretes and mortars. Two methods (EN 12390-11 and NT Build 443) were used and compared for the chloride diffusion test. Fresh and mechanical properties of these concretes are the subject of a companion paper by the same authors [10].

2. Experimental program

2.1 Materials

Composite cements were prepared by mixing CEM I 52.5 R HES (OPC), ground granulated blast-furnace slag (S), fly ash (FA) and limestone filler (L). As a set regulator, gypsum had been added with a concentration to obtain a total sulfate content of 3%. Chemical and mineralogical characterization showed that the studied SCMs meet all the requirements from standard EN 197-1, regarding the properties of the cement constituents. Slag contains more than 60% by mass of CaO, MgO and SiO_2 and the ratio (CaO+ MgO)/SiO_2 is above 1. Fly ash is characterized by a low loss on ignition (category A), a CaO content below 10% and a SiO_2 content above 25%. Limestone filler contains 100% of calcite. The clay content is inferior to 1.2 g/100 g filler and the total organic content (TOC) is inferior to 0.5%.

Then the physical, chemical and mechanical properties of the ternary cements, with different proportions of Portland cement (OPC between 45 and 65%), blast-furnace slag or fly ash (S or FA between 10 and 30%) and limestone filler (L between 5 and 35%), were determined. Two series had been conducted. In series 1, ternary blended cements with slag (OPC-S-L) were studied, with reference cements CEM I 52.5 R HES, CEM II/B-M (L-S) 32.5 R and CEM III/A 42.5 N LA. In series 2, ternary cements with fly ash (OPC-FA-L) were investigated with reference cements CEM I 52.5 R HES, CEM II/B-M (LL-S-V) 32.5 N and CEM II/B-V 32.5 R. For the sulfate attack test, a high sulfate resistant cement has also been tested: CEM I 52.5 R LA – SR 3. Nearly all the ternary cements meet the requirements from the standard EN 197-1, regarding the compressive strength, initial setting time and soundness. Eight cements compositions have been chosen and are presented in Tab. 1. The results show that ternary OPC-S-L cements reach higher compressive strength classes than OPC-FA-L cements.

2.2 Concrete mixes

Two types of concrete have been chosen, named T(0.45) and T(0.55). The cement content, *w/c* ratio and the desired slump are 340 kg/m³, 0.45, 120 ± 30 mm and 300 kg/m³, 0.55, 180 ± 30 mm respectively for the two types. The fine aggregates consist of sea sand and rolled sand. The coarse aggregates consist of crushed limestone with a maximum nominal size of 20 mm.

Table 1: Composition (% by mass), Blaine surface and compressive strength class for cements of series 1 (OPC-S-L) and series 2 (OPC-FA-L).

	OPC (%)	S (%)	FA (%)	L (%)	Blaine surface (cm²/kg)	Strength class (mortar)
Series 1 (OPC-S-L)						
CEM I 52.5 R HES (batch 1)	100	0	0	0	4780	52.5 R
CEM II/B-M (L-S) 32.5 R	74	12	0	14	3737	32.5 R
CEM III/A 42.5 N LA	59	41	0	0	4591	42.5 N
CEM 1 [65OPC 30S 5L]	65	30	-	5	4770	52.5 N
CEM 3 [45OPC 30S 25L]	45	30	-	25	4630	42.5 N
CEM 6 [50OPC 20S 30L]	50	20	-	30	4430	42.5 N
CEM 10 [55OPC 10S 35L]	55	10	-	35	4260	42.5 R
Series 2 (OPC-FA-L)						
CEM I 52.5 R HES (batch 2)	100	0	0	0	4930	52.5 R
CEM II/B-M (LL-S-V) 32.5 N	71	7	9	13	3360	32.5 N
CEM II/B-V 32.5 R	78	0	22	0	2960	32.5 R
CEM 1 [65OPC 30FA 5L]	65	-	30	5	4110	52.5 N
CEM 3 [45OPC 30FA 25L]	45	-	30	25	4120	-
CEM 6 [50OPC 20FA 30L]	50	-	20	30	4230	32.5 R
CEM 10 [55OPC 10FA 35L]	55	-	10	35	4440	32.5 R

The concrete mixes have been formulated so that the grading curve follows the limits specified in the standard EN 480-1 [11], as shown in Fig. 1. There exists a slight difference between the two series: series 2 contains more fines below 0.5 µm. A polycarboxylic-ether superplasticiser has been used. The dosage has been adjusted to reach the desired slump. The water absorbed by the aggregates and supplied by the superplasticiser have been taken into account in the mixing water. The mix proportions are detailed in Tab. 2.

The concrete samples are conserved during 24h at a temperature of 20 ± 2°C. After removal, the specimens are placed in water at the same temperature for curing. For the durability tests, a water curing period of 91 days has been chosen to provide ideal hydration conditions for these cements. Indeed, a good curing is important for these composite cements as highlighted by several authors [8, 12–14]. For sulfate attack test, standard mortars (EN 196-1 [15]) were cured in water during 28 days.

2.3 Testing methods

Accelerated carbonation tests were carried out following standard EN 13295 [16] on three prismatic samples of 10 cm x 10 cm 40 cm in size, for the two types of concretes. After curing,

the samples were conserved in a climatic chamber during 14 days until constant mass. Then they were transferred in the carbonation chamber with a CO_2 content of 1% at a temperature of 20 ± 2°C and relative humidity of 60 ± 10%. The carbonation depth was measured with a phenolphthalein solution at 7, 14, 28, 56 and 91 days of carbonation.
The resistance of freeze-thaw cycles with de-icing salts was measured according to EN 12390-9 (slab test) [17] on T(0.45) concretes. Four cylindrical samples of 113 mm x 50 mm were cored after curing. They were conserved in a climatic chamber during 14 days and then prepared with resin. They were subjected to 56 cycles of freeze-thaw with a solution of NaCl (1 cycle lasts 24h). The loss of material at the surface was collected and weighted after oven-drying at 105°C, at 7, 14, 28, 42 and 56 days.
Chloride diffusion test was carried out according to EN 12390-11 [18] and NT BUILD 443 [19] on T(0.45) concretes. Three cylindrical samples of 100 mm x 70 mm were cored after curing, then saturated in water under vacuum and then immersed in a solution of $Ca(OH)_2$ (EN method) or directly saturated and immersed in a $Ca(OH)_2$ solution (NT method). The samples were then immerged in a NaCl solution with a concentration of 30 g/l during 91 days (EN method) or with a concentration of 165 g/l during 35 days (NT method). Samples are taken at different depths (x) to measure the chloride concentration (C(x,t) and initial content C_i). The diffusion coefficient (D_{nss}) is then calculated from the chloride concentration profile, according to the following Eq. (1), by least-squares method:

$$C(x,t) = C_i + (C_s - C_i)\left(1 - erf\left(\frac{x}{\sqrt{4\, D_{nss}\, t}}\right)\right) \quad (1)$$

Sulfate attacks were carried out on mortar samples of 2 cm x 2 cm x 16 cm in size at a temperature of 20°C, according to CUR 48 [20]. Three prisms were immerged in a sulfate solution (16 g/l SO_4) and three others were placed in water, as control specimens. The length variation is measured and compared with the control specimens, during 365 days.

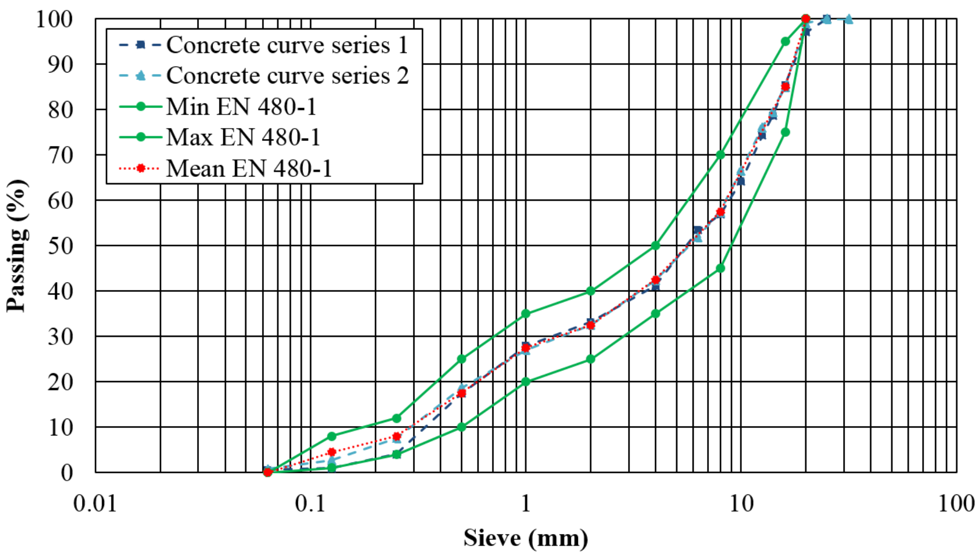

Figure 1: Grading curves for concretes (series 1 and 2) and the limits of standard EN 480-1.

Table 2: Concrete mix proportions (kg/m³) for series 1 (OPC-S-L) and series 2 (OPC-FA-L).

	Series 1 (OPC-S-L)		**Series 2 (OPC-FA-L)**	
	T(0.45)	**T(0.55)**	**T(0.45)**	**T(0.55)**
Cement	340	300	340	300
Fine aggregates (sea sand and rolled sand) 0/5	703	705	-	-
Fine aggregates (rolled sand) 0/4	-	-	739	742
Coarse aggregates (crushed limestone) 4/20	1198	1201	1146	1149
Superplasticiser* (%)	0.15 – 0.8	0.2 – 0.25	0.7 – 2	0.6 – 1.3
Mixing water	161	173	160	172

*The superplasticiser dosage (% by mass cement) was adjusted to reach the desired slump.

2.4 Comparative approach

The evaluation of performance of concrete made with these new ternary cements OPC-S-L and OPC-FA-L consists in a comparative approach inspired by the methodology from Belgian standard NBN B 15-100 [21]. All the results are compared with the ones obtained on reference concretes, with a weighting factor of 20% or 40%. For series 1, the reference could be either concrete made with CEM I 52.5 R HES or with CEM III/A 42.5 N LA. For series 2, the reference is CEM I 52.5 R HES. The criteria are indicated by a red line in the graphs.

3. Results and discussion

The fresh and mechanical properties are presented in the companion paper [10]. The compressive strengths of OPC-S-L cements are higher than OPC-FA-L cements, which has already been observed on mortars (strength class in Tab. 1).

3.1 Carbonation

The results of the carbonation depths of T(0.55) concretes are presented in Fig. 2 for series 1 and Fig. 3 for series 2. The results of T(0.45) concretes are lower, as w/c ratio is lower and cement content is higher, but the trends are similar.

The comparative approach requires that at 56 days, the carbonation depth (d) should be inferior or equals to 1.2 times the depth of the reference concrete: $d_{56} \leq 1.2\ d_{56,\mathrm{ref}}$. For series 1, at 56 days, the reference concrete made with CEM III/A 42.5 N LA has a carbonation depth of 6.5 mm. Only the CEM 1 [65OPC 30S 5L] concrete, with the highest clinker content and the lowest limestone filler content, meets the criterion of 6.5 x 1.2 = 7.8 mm. For series 2, no OPC-FA-L cement satisfies the criterion as CEM I 52.5 R HES concrete shows a high resistance to CO_2 penetration. The difference for concretes with the same reference cement CEM I 52.5 R HES could be due to the difference in the grading curve (see Fig. 1).

Carbonation resistance decreases with decreasing clinker content and increasing limestone filler content. Indeed, carbon dioxide present in the atmosphere reacts with cement hydration products such as portlandite $Ca(OH)_2$, which buffers the pH. The quantity of portlandite in blast-furnace slag or fly ash concrete is significantly reduced due to pozzolanic reaction and lower clinker content [5, 12, 14]. Limestone filler worsens the carbonation resistance, which has also been observed by Courard and Michel. They found that the "open" porosity increases with limestone filler content [5]. When comparing the two series, ternary OPC-S-L cements present a higher resistance to carbonation than OPC-FA-L cements. The presence of slag seems to give a denser material than fly ash, as it has pozzolanic as well as hydraulic properties [3].

3.2 Freeze-thaw with de-icing salts

The results of cumulated loss of materials of T(0.45) concretes are presented in Fig. 4 for series 1 and in Fig. 5 for series 2. For series 1, results of concretes with cements CEM 1, CEM 3 and CEM 6 have been withdrawn due to incoherent results.

The comparative approach requires that at 28 cycles, the cumulated loss of material (S) should be inferior or equals to 1.2 times the one of the reference concrete: $S_{28} \leq 1.2\, S_{28,\mathrm{ref}}$. At 28 days, concrete reference CEM I 52.5 R HES has a cumulated loss materials of 1.1 kg/m² for both series. In series 1, CEM 10 [55OPC 10S 35L] concrete is just above the criteria (1.1 x 1.2 = 1.32 kg/m²) but it performs better than the reference concrete CEM II/B-M (L-S) 32.5 R. In series 2, no OPC-FA-L concrete satisfies the criteria but CEM 1 [65OPC 30FA 5L] concrete performs slightly better than reference concrete CEM II/B-M (LL-S-V) 32.5 N.

The criteria at 28 days appears to discriminate against concrete containing slag, as pointed out by Chidiac and Panesar [8]. Indeed, after 56 freeze-thaw cycles, CEM 10 [55OPC 10S 35L] concrete resists better than CEM I 52.5 R HES concrete. In series 1, it is difficult to report trends as only four different cements have been tested. In series 2, the scaling resistance seems to be reduced with increasing limestone filler content. CEM 1 [65OPC 30FA 5L] concrete presents a better resistance than CEM II/B-M (L-S) 32.5 R, which contains 13% of limestone filler. CEM 10 [55OPC 10FA 35L] concrete shows the worst performance, with a cumulated material loss of over 15 kg/m² after 56 cycles. When comparing series 1 and 2, concretes made with OPC-FA-L cements seem to have more scaling damage than OPC-S-L cements. For binary slag cements, several authors indicated that an adequate entrained air void system could prevent such freezing and thawing attack [12, 14, 22].

3.3 Chloride diffusion

The diffusion coefficients obtained for T(0.45) concretes are presented in Fig. 6 and Fig. 7 for series 1 and series 2 respectively. Two different test methods have been compared for OPC-S-L cements: EN 12390-11 and NT BUILD 443.

The comparative approach requires that the diffusion coefficient (D_{nss}) should be inferior or equals to 1.4 times the one of the reference concrete: $D_{\mathrm{nss}} \leq 1.4\, D_{\mathrm{nss}}$. In series 1, concrete reference CEM I 52.5 R HES has a diffusion coefficient of 9.85 10^{-12} m²/s with the European method, which gives a criteria of 13.8 10^{-12} m²/s. All concretes made with OPC-S-L cements, except the CEM 10 [55OPC 10S 35L], satisfy the requirement. This is also observed for the NT BUILD method, with a criteria of 9.5 10^{-12} m²/s. For series 2 (OPC-FA-L), CEM 10 [55OPC 10FA 35L] concrete does not satisfy the criterion either. The difference observed between CEM I 52.5 R HES concretes of the two series could be due to the difference in the concrete grading curve, as seen in Fig. 1. Series 2 concretes contain more fines, which could lead to a denser material and thus the diffusion could be more hindered.

In series 1, the resistance to chloride penetration seems to be improved by the presence of slag, while the presence of limestone filler seems to not have an influence on it. CEM III/A 42.5 N LA [59OPC 41S], CEM 1 [65OPC 30S 5L] and CEM 3 [45OPC 30S 25L] concretes show low diffusion coefficients compared to CEM I 52.5 R HES concrete. This positive effect of slag compared to clinker is explained by a higher chloride binding capacity with formation of Friedel's salts [23]. Besides, the C-S-H produced by reaction of slag and characterized by a lower Ca/Si ratio have a higher chloride adsorption [12, 22]. Kayali et al. [23] have also highlighted the role of hydrotalcite, a significant hydration product of slag blends, in chloride binding. In series 2, the presence of fly ash also seems to have a beneficial effect against chloride penetration. CEM II/B-V 32.5 R [78OPC 22FA], CEM 1 [65OPC 30FA 5L], CEM 3 [45OPC 30FA 25L] and CEM 6 [50OPC 20FA 30L] concretes show a better resistance to chloride penetration than CEM I 52.5 R HES concrete. This is also due to a higher chloride binding capacity of fly ash compared to Portland cement [24]. When comparing OPC-S-L and OPC-FA-L cements according to the European standard, it seems that fly ash performs better than slag regarding resistance to chloride diffusion, which is in contradictory of what Ytterdal [24] found. But Ytterdal compared mortars with 30% replacement of fly ash and mortars with 50% replacement of blast-furnace slag. When comparing the two test methods, EN 12390-11 gives significantly higher values, which could be due to the different procedure test (saturation under vacuum and immersion during 91 days). The standard deviations are particularly higher too for this method. It is thus always important to consider the test method while discussing about diffusion coefficients.

3.4 Sulfate attack

The results of the linear expansion of mortars immersed in a sulfate solution for one year are presented in Fig. 8 and Fig. 9 for series 1 and series 2 respectively. In the latter, a sulfate resistant cement CEM I 52.5 R LA – SR 3 has also been tested.

The comparative approach requires that the linear variation (*L*) at 365 days should be inferior or equals to 1.2 times the one of the reference mortar: $L_{365} \leq 1.2\ L_{365,ref}$ or that $L_{365} \leq 0.05\%$. In series 1, mortars made with CEM III/A 42.5 N LA [59OPC 41S], CEM 1 [65OPC 30S 5L], CEM 3 [45OPC 30S 25L] and CEM 6 [50OPC 20S 30L] present a linear deformation inferior to 0.02%. This beneficial effect of slag on sulfate resistance is due to lower content of tricalcium aluminate C_3A, which is the main expansive reactive material [5, 12], and due to the low aluminum content of slag [12, 14]. Besides, Courard and Michel [5] noticed that the sulfate resistance of mixtures containing slag was not influenced by limestone fillers, contrary to Portland cement mixtures, for which the relative deformation increases with limestone filler content, up to 15%. Hossack and Thomas [6] observed that limestone content in combination with fly ash or slag had little to no effect on sulfate resistance. In series 2, it appears that the mortar made with CEM I 52.5 R LA – SR 3 performs better than the CEM I 52.5 R HES but shows an expansion of about 1% after one year. Again, mortars made with CEM II/B-V 32.5 R [78OPC 22FA], CEM 1 [65OPC 30FA 5L], CEM 3 [45OPC 30FA 25L] and CEM 6 [50OPC 20FA 30L] present a linear deformation inferior or equals to 0.1%. When comparing the two series, it seems that fly ash is even more favourable to resistance to sulfate attack than blast-furnace slag. This has also been observed by Hossack and Thomas [6]. They found that the intensity of ettringite peaks was greater in fly ash mortar bars than slag mortar bars, which may indicate a lower permeability and thus a less resistance to sulfate. The risk of thaumasite formation for sulfate attacks at 5°C on OPC-S-L cements has been previously discussed by Rondeux et al. [25]. The beneficial effect of slag to prevent damage due to sulfate attacks at low temperature has not been observed for OPC-FA-L cements.

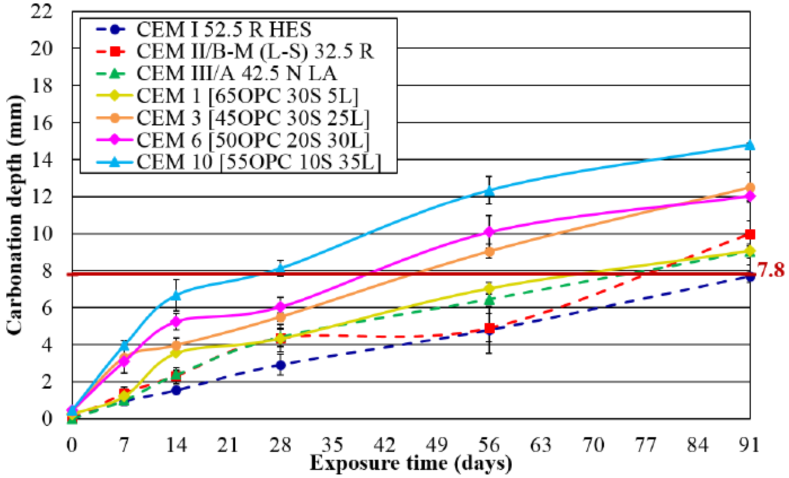

Figure 2: Carbonation depths for T(0.55) concretes series 1 (OPC-S-L) – Criteria at 56 days: 7.8 mm with reference CEM III/A 42.5 N LA.

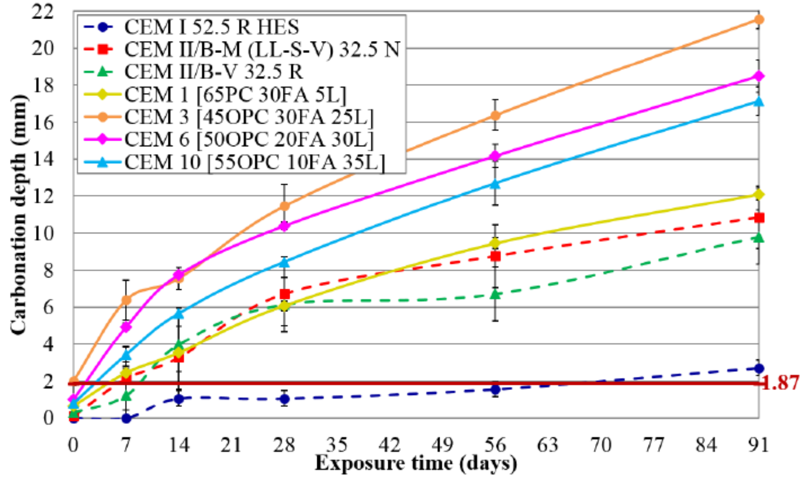

Figure 3: Carbonation depths for T(0.55) concretes series 2 (OPC-FA-L) – Criteria at 56 days: 1.87 mm with reference CEM I 52.5 R HES.

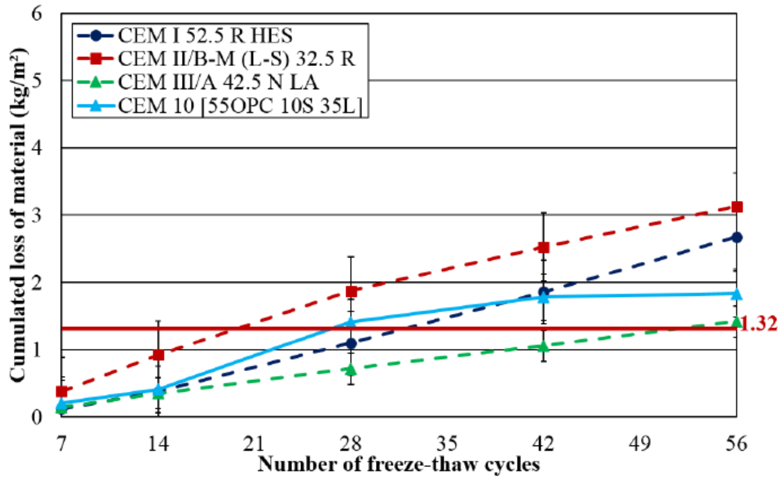

Figure 4: Cumulated loss of material for T(0.45) concretes series 1 (OPC-S-L) – Criteria at 28 days: 1.32 kg/m² with reference CEM I 52.5 R HES.

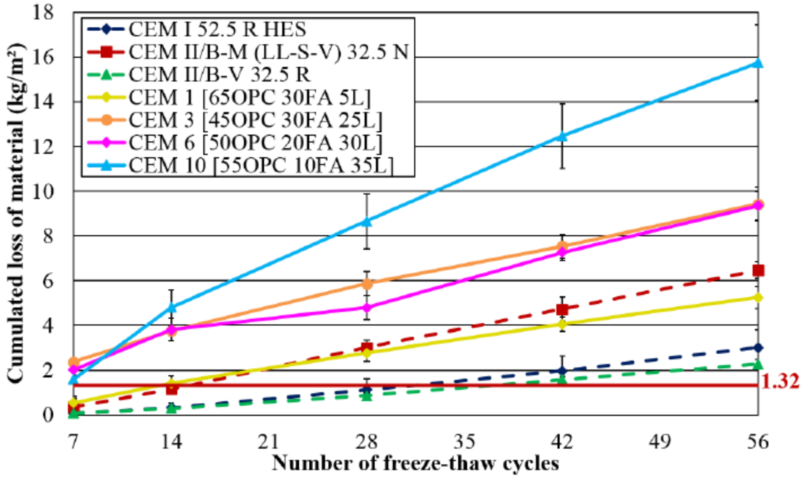

Figure 5: Cumulated loss of material for T(0.45) concretes series 2 (OPC-FA-L) – Criteria at 28 days: 1.32 kg/m² with reference CEM I 52.5 R HES.

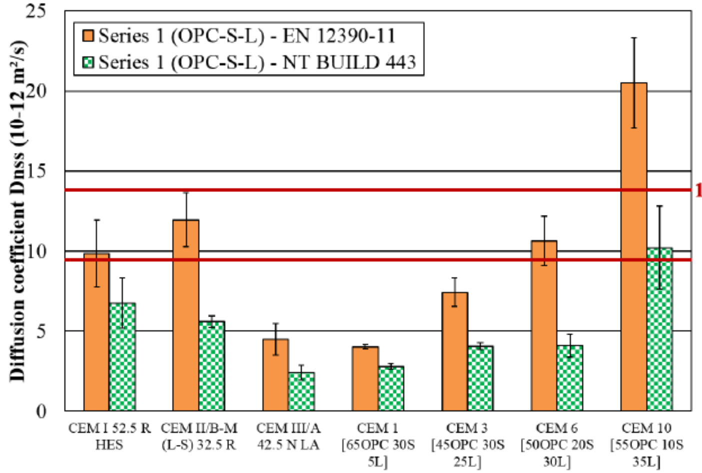

Figure 6: Diffusion coefficient for T(0.45) concretes series 1 (OPC-S-L) – Criteria: 13.8 10^{-12} m²/s (EN) and 9.5 10^{-12} m²/s (NT) with reference CEM I 52.5 R HES.

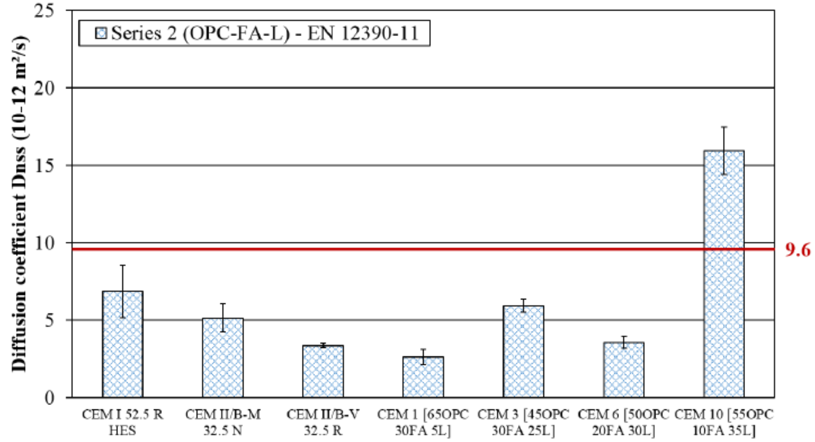

Figure 7: Diffusion coefficient for T(0.45) concretes series 2 (OPC-FA-L cements) – Criteria: 9.6 10^{-12} m²/s with reference CEM I 52.5 R HES.

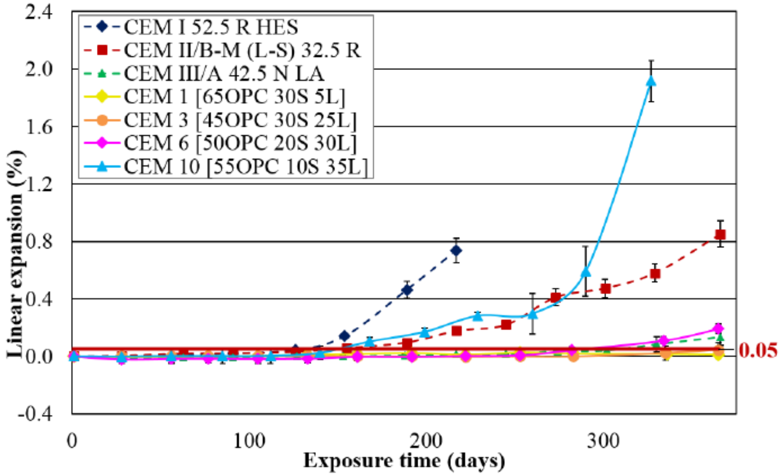

Figure 8: Linear deformation of mortars series 1 (OPC-S-L) – Criteria at 365 days: 0.05%.

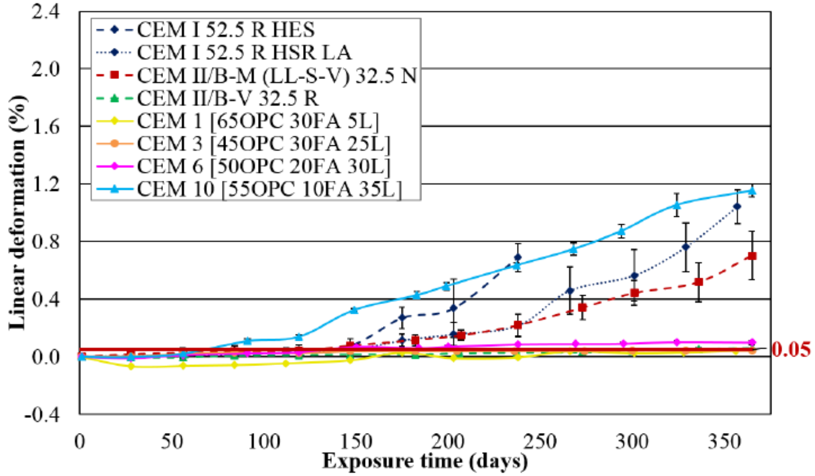

Figure 9: Linear deformation of mortars of the series 2 (OPC-V-L) – Criteria at 365 days: 0.05%.

4. Conclusions

The effects of ternary cements made with blast-furnace slag or fly ash (with a content up to 30%) and limestone filler (with a content up to 35%), on the durability of concretes were investigated. The following conclusions, for the tests conditions and materials of this research, can be drawn:

- Blast-furnace slag and fly ash have a beneficial effect on chloride diffusion and sulfate attack thanks to a higher chloride binding capacity and a lower C_3A content;
- Limestone filler, coupled with blast-furnace slag or fly ash, has no significant influence on chloride diffusion and sulfate attack resistance (at 20°C), but well a negative effect on freeze-thaw with de-icing salts resistance;
- Carbonation resistance decreases with decreasing clinker content and increasing limestone filler content;
- Ternary OPC-S-L cements seem in general to contribute to a better durability performance than OPC-FA-L cements;
- Except for chloride diffusion and sulfate attack, most of the tested ternary cements did not satisfy the requirements of the comparative approach.

This study shows that despite the fact that ternary cements with high amount of limestone and slag or fly ash meet the requirements of European standard EN 197-1 in terms of initial setting time, soundness and compressive strength, these cements could not be used in concrete in every environment. It is necessary to always assess the suitability for use of new composite cements for concrete. An adjustment of the concrete mix (cement content, w/c, grading curve…) or special precautions (thicker covering…) shall be necessary to use these ternary cements in specific environments or applications. Finally, it should be noted that a long curing period is highly recommended for these composite cements.

Acknowledgements

The authors would like to acknowledge the Wallonia Government (Belgium) for the financial support as well as the National Centre for Scientific and Technical Research for the Cement Industry for the production and characterization of the ternary cements.

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