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Quantification and Qualification of High Lime Fly Ash by Efficiency Factor: Mechanical and Durability Aspects

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Abstract

This paper presents a study of the cementing efficiency of high lime fly ash with regards to mechanical and durability properties. The investigated variables were the rate of the incorporation of fly ash, the cement type, the water/cement ratio, and the curing age of the mix. An extensive experimental campaign was conducted in order to determine the compressive strength and chlorides penetration. A test which simulates the penetration of chloride ion in concrete (multi-regime method) has been conducted, in order to determine the chloride diffusion coefficient in a non-steady-state. Two cementing efficiency factors were determined; (i) in terms of the compressive strength, (ii) in terms of the chlorides diffusion coefficient. Both of them have been determined in relation to the water/cement ratio. The result shows that the cementing efficiency is strongly influenced by the water/cement ratio. Concerning durability, greater efficiency values than those observed in relation to the compressive strength have been found.

Keywords: high lime fly ash, cementing efficiency, k value, durability, chloride diffusion.

1 Introduction

Fly ash has been used in mortars for many years. Initially, it was chosen because it was economical, and great efforts have been made to develop it and increase its use [1]. However, Neville [2] states that "the importance of fly ash should not be exaggerated: in this moment is not an economic substitute for cement, or an extension in the mix. However, the fly ash gives important advantages to the concrete, and it is therefore essential to understand the role and influence of fly ash."

Although the above is true, we must not ignore the fact that fly ash is a residue of the process of burning coal in power plants, and is still produced in large quantities. According to several studies, coal will remain a major source of energy worldwide [3]. Fossil fuels in general are expected to remain the main source of energy until 2030 [4]. In 2005, the amount of waste produced by burning coal to generate electricity amounted to 65 million tons in the EU-15, and it is estimated that the EU-27 total production was close to 95 million tons.

In addition to the possible economic benefits of using fly ash, the inclusion of this material in cement-based products reduces the pollution caused by the cement industry and, of course, by concrete. A study in Denmark suggests that current knowledge and experience can be used to produce concrete with a low environmental impact in two ways: the concrete mix design can be modified to create a cement with a lower environmental impact by minimizing the content of cement and cementitious materials and replacing cement by additions; and good environmental management can be applied in the production of cement and concrete [5].

The above information provides the basis for promoting the study and development of alternatives to minimize the environmental impact of cement and concrete. In Europe, for example, the common cements may contain cement clinker, ordinary Portland cement (CEM I) and up to 8 secondary constituents, including high and low lime fly ash, natural pozzolan and silica fume.

The European standard EN 206 (2004) states that when silica fume or fly ash are combined with CEM I, they are referred to as "additions" and qualified as part of the cement content, using the concept of cementing efficiency or the k value, which takes into account their cementing behavior. However, the k value that is established for additions is arbitrary and not supported by test methods [6].

In view of the above, a simple system needs to be devised using existing testing techniques to evaluate the performance of additions in relation to the cement used to manufacture concrete. This paper presents a method for determining the efficiency of fly ash in cement mortars, which can be used to redesign mixes to attain an equivalent strength.

2 Theoretical Basis

Recently, there has been growing interest in determining the efficiency factor or k value of mineral additions to concrete. The efficiency factor has been defined as the part of the supplementary cementing material in pozzolanic concrete (or mortar) that can be considered as equivalent to Portland cement and that has same properties as concrete without any additions[7, 8, 9] .

The study of the efficiency factor goes back to the 1960s, when a paper by Smith [10] showed that strength is not necessarily lost at early ages when fly ash is used in concrete. Smith assumed that the main factors affecting compressive strength are the water/cement (W/C) ratio and the cement type. His theory was as follows: as two concretes that have the same strength at a given age can be produced by modifying the W/C ratio of different cements, then the same approach can be used for concrete that contains fly ash.

The mathematical approach (Eq.1) used was as follows:

$$\frac{W}{C} = \frac{W}{C' + kA} = \frac{W}{C'} \quad \frac{1}{1 + \frac{kA}{C'}}$$

Where (W/C) is the water/cement ratio of control concrete, C' is the amount of cement in concrete with fly ash, A is the amount of fly ash and k is the cementing efficiency of the fly ash. Smith found that a k value of 0.25 can be used for concrete at the ages of 7 and 28 days, regardless of the W/C ratio used.

Smith's methodology was used in the United Kingdom. However, many weaknesses were found in its implementation [1]. Similarly, Gopalan et al., [11] suggested that the k value varies significantly depending on the curing period, the resistance of the mix and the type of fly ash.

It should be noted that most of the methods proposed for the determination of the cementing efficiency factor “k” are based on compressive strength, and do not take into account the water/cement ratio, a variable that has a strong influence on the calculation of this value.

Evidently, knowledge of the durability function is not as developed as that of the compressive strength, in which more factors can influence than those involved in the function of resistance. So far, the research undertaken to generate concrete with similar lasting properties based on the determination of the cementing efficiency of admixtures has been very limited.

Regarding durability, a few studies have been conducted on carbonation resistance and chloride ion penetration taking into account the cementing efficiency of admixtures. Papadakis and Tsimas [7, 8, 9] are of the few researches in the field that were able to observe a lower chloride content in concretes with admixtures after having replaced aggregate or cement with pozzolans; obtaining particularly high "k" values (k = 2 for high-lime fly ash, and k = 3 for low-lime fly ash) compared to the values of compressive strength (k = 0.2 – 0.3).

3 Model used to evaluate the cementing efficiency of the durability

Active admixtures (Type II) contribute in concrete with the formation of a series of hydrated compounds that alter properties such as strength or penetration of aggressive compounds, among others [1, 12]. Thus, the efficiency factor "k" attempts to account for this contribution.

Asserting that two concretes can be designed using a specific chloride diffusion coefficient, with and without admixtures, a relationship can be established for both with regard to chloride diffusion and water/cement ratio, applying the cementing efficiency factor. This can be expressed graphically with following Figure (Fig. 1).

Following the same approach, we can obtain two types of concretes, one with fly ash and one without, both with the same chloride diffusion coefficient but different water/cement ratios. Using equation 2, we can relate these concretes through the cementing efficiency factor of durability (kd).

$$Dc \;\; (addition) = f \quad \frac{w}{c \;\; + k_d * A} \quad = Dc_0(control) = f \quad \frac{w}{c_0}$$

Where DCφ is the diffusion coefficient of fly ash concrete, DC0 the diffusion coefficient of control concrete, w the amount of water, cφ the amount of cement in the fly ash concrete, C0 the amount of cement in the control concrete, and A is the amount of fly ash used. With the equation above, we can gauge the value of "kd", as we assume the following equation (3) to be true:

$$\frac{w}{c_0} = \frac{w}{c \;\; + k_d * A}$$

From which we can explain the concept of the cementing efficiency factor of durability as show in equation 4:

$$k_d = \frac{1}{\;} \quad \frac{\omega}{\omega_0} \quad 1$$

Where ω_φ is the water/cement ratio of concrete with fly ash, ω_0 the water/cement ratio the control mix, and φ the percentage of fly ash in the mixture. Because the cementing efficiency factor of the admixtures is determined, necessarily, by the curve that exemplifies the behaviour of the chloride diffusion coefficient in relation to the water/cement ratio, a potential correlation was used, taking into account the results observed in previous studies [13, 14, 15]. The mathematical expression is shown in equation 5:

$$DC_c = b + \left(\frac{w}{c}\right)^m$$

Where DCc is the chloride diffusion coefficient, and b and m are constants.

4 Experimental approach

The study of the cementing efficiency of fly ash in concretes and its relationship to chloride diffusion was developed in three stages: (i) the study of compressive strength development in concrete with and without fly ash, (ii) the determination of chloride diffusion coefficients, and finally, (iii) the determination of the cementing efficiency values concerning durability, in this case, based on chloride diffusion.

4.1 Materials, measurement and testing.

Throughout the experimental campaign two types of cements were used, (CEM I 42.5R and 52.5R), one high lime fly ash, three fly ash replacement percentage rates per cement (0%, 25% and 43%), two water/binder ratios (0.45 and 0.60 cement + fly ash) and three curing ages (7, 28 and 90 days).

The physical and chemical properties of the biding materials used (CEM I 42.5R and 52.5R and fly ash) are shown in Table 1. The determination of the chemical composition was performed through X-ray fluorescence tests (XRF, Philips PW-1400 model) and mineralogical composition by way of X-ray diffraction tests (XRD - Siemens D-500 Cu diffractometer).

The main phases present in fly ash are amorphous compounds of aluminium silicates, mullite ($Al_6Si_2O_{13}$), quartz (SiO_2), magnetite (Fe_3O_4), anhydrite ($CaSO_4$), ettringite ($3CaO\ Al_2O_3\ 3CaSO_4\ 32H_2O$), hematite ($Fe_2O_3$), and lime ($CaO$). The fly ash can be classified as type C, as contents of silica, alumina and iron exceed 70% and it presents a high content of calcium oxide (27%).

Table 2 shows some physical properties of the binding materials. It can be observed that high lime fly ash has average particle size (26 μm). The trial was conducted in wet (with ethanol as liquid medium) by laser scattering (LS 13 320-Beckman Coulter). The determination of the shape of the particles was performed using scanning electron microscopy (SEM).

The design of the concrete mix consisted in finding the minimum amount of void space in the mix with different aggregate proportions [16] and directly replacing cement with an equal weighting amount of fly ash. The dosages used in every mix for compressive strength and durability tests are shown in table 3. The control mixes maintained a water/binder ratio of 0.45 and 0.6, respectively called A0 and B0. The dosages were prepared with two types of cement, CEM I 42.5R (L) and CEM I 52.5R (M).

All aggregate was used in a saturated surface dry condition. The concrete samples were fabricated in cylindrical steel test tubes with a 100 mm in diameter and 200 mm in height using manual compaction. The molds were covered during the first 24 hours to minimize water evaporation. Afterwards, the samples were placed in a humidity chamber (97 ± 2%) until the respective test dates.

The compressive strength test was performed according to the UNE EN 12390-3 [17]. To determine the chloride diffusion coefficients a chloride migration test was performed according to UNE 83987 standard [18]. We used concrete disks 75 mm in diameter, extracted from the middle third of the original cylindrical specimens. To determine the Dns, the device used is the classical two compartments cell, where one of the chambers is filled with a chloride solution while the other contains a free chloride solution. Periodically along the experiment, Cl- concentration in both compartments has to be monitored in order to determine the flux of chlorides throughout the specimen. Distilled water was introduced in the compartment where the anode was located (anolyte) in all the cases. This neutral solution avoids chlorine evolution in the anode by inducing its own corrosion. The voltage applied was of 12 V DC, although the real voltage drop across the specimen was measured. The electrical field was switched off and after waiting for 5 s, conductivity of the anodic solution was measured by introducing an electrode of conductivity in the compartment. Values of conductivity were referenced to a temperature of 25 ºC, by considering an increase of 2% in the conductivity of the solution when temperature increases a degree centigrade. Once the conductivity was recorded, the electrical field was switched on again to continue the test.

5 Results and discussion

5.1 Compressive strength.

Results of the compressive strength test are shown in Figures 2 and 3 for concretes made with CEM I 42.5R y CEM I 52.5R. For example, a concrete designated L II A 25, references a concrete fabricated with CEM I 42.5R, high lime fly ash, water/binder ratio of 0.45, and a 25% of cement replacement with fly ash.

The results show that the compressive strength of all concrete mixes with fly ash experiences a loss of initial strength proportional to the percentage of replacement.

In terms of average loss of strength at an early age, mixes with fly ash reach the values of 20%. With this data, it can be suggested that the influence of fly ash at the early age of 7 days is little and is related to the specific characteristics of the type of fly ash used. The influence, at this age, of the cementing properties of a fly ash with high lime content should also be noted.

At 28 days, the loss of strength in mixed with fly ash is 17%. Finally, after 90 days of hydration, the loss of strength in concretes with fly ash, compared to that of the control mix, experience a decline in strength loss to11%.

From the results above, it can be posed that at an early age fly ash acts more like filler, as it has little influence on the improvement of strength and the interfacial transition zone. Nonetheless, with an increased time of hydration, compared with the control mixes, significant improvements in strength can be observed in concretes with fly ash.

High lime fly ash reacts more rapidly than low-lime fly ash, but according to the results, concretes made with high lime fly ash do not show a significant increase in strength over time.

The effect of packaging depends on the ash and cement used. According to Monteiro, the best results are obtained with a Portland cement of relatively coarse particle size and a fine fly ash [19].

Packaging has a beneficial effect on strength by reducing the volume of air trapped in the concrete. Yet, the most important contribution of packaging is the volume reduction of the large capillary pores. On the other hand, the coarser particles of mineral admixtures can be also considered micro-aggregates, which improve the density of the hydrated cement paste in a manner similar to that of the anhydrous cement particles. This is also beneficial in terms of strength, resistance to crack propagation and stiffness. Finally, the resulting system of capillary pores is able to retain more water than may be available for hydration in the long term.

5.2 Chloride diffusion coefficient of concrete.

The behavior of chloride diffusion coefficient (CDC -for non-steady state) of concrete was studied at the age of 28 and 90 days. Figure 4 and 5 show the behavior of concrete made with CEM I 42.5R and 52.5R with replacement rates of 25% and 43%. The curves shown are given in terms of water/binder ratio.

The study of the chloride diffusion coefficient is of great importance, as it is capable of providing an estimate of how long it will take for corrosion to appear in the re-bars of reinforced concrete. As the most important way of protecting from deterioration is by preventing chlorides from reaching the reinforcing steel. This is because the chloride ion needs to move from the surface of the concrete into the rebar and build up enough quantity for the corrosion to start. Thus, it is this ion transfer rate that controls the rate of deterioration, at least the initial stage [20, 21].

According the results obtained, control mixes made with cement CEM I 42.5R present higher chloride diffusion values than concretes made with CEM I 52.5R (fig. 5). However, at the age of 90 days, concretes with CEM I 52.5R have values slightly greater than concretes with CEM I 42.5R when it comes to the diffusion coefficient (non-steady-state) for water/binder ratios 0.45 and 0.60.

Another variable that has a significant influence in the chloride diffusion coefficient is the water/binder ratio. As it can be observed, an increase in the water/binder ratio results in a severe increase in the diffusion coefficients. This is due to the fact that an increase in the water/binder ratio contributes to a greater number of pores and, above all, a greater connectivity between them.

Age also exerts a strong influence on diffusion of chloride. A reduction in the diffusion coefficient for non-steady-state can be observed when moving from 28 to 90 days of curing. This reduction is a result of the pore refinement caused by advance of cement hydration, and the increasing amount of available gel that can serve a place for chloride to bind.

The behaviour of concretes mixed with high lime fly ash must be done differentiating the type of cement used. In the case of concretes with CEM I 42.5R, the diffusion coefficients, for both 28 and 90 days, are equal or smaller than those of the control concretes. This situation can be due to the double nature (hydraulic and pozzolanic) of the fly ash, which generates reaction products (CSH) at early ages that aid the physical fixation of chloride and also have the refining effect on the pores (Fig. 4).

The behaviour is slightly more complex when using high lime fly ash and CEM I 52.5R because this type of cement produces a lower porosity, and to observe the effect of fly ash it must be sufficiently efficient in both physical and chemical terms. That is, physically, sufficiently small in relation to cement so it can a significant filling effect, and chemically, sufficiently reactive with Portlandite. That is why its addition generates its better results when used in higher quantities (43%), with values equal to or lower than those of the control concrete, especially at low water/binder ratios. When used in smaller percentages (25%), it generates higher diffusion values than in the control concretes (Fig. 5).

5.3 Chloride diffusion and cementing efficiency of high lime fly ash.

As discussed in above, the curve that shows the behaviour of chloride diffusion in relation to the water/cement ratio was used to determine the cementing efficiency of durability (Fig. 1).Taking into account results obtained in previous studies, a potential correlation was used [22, 23, 15], the mathematic expression is shown as equation 4.

In the experiment, two water/cement ratios were used, (maintaining all other variables constant) to determine the constants “b” and “m” for each age. The values were determined by means of a regression analysis (potential estimation), afterwards, the respective curves were traced. Water/cement ratios ranging from 0.40 to 0.80 were used.

Based on these curves, the values of “kd” were established for every type of fly ash, replacement percentage, cement type and curing age. The mathematical expression employed to determine the value of the cementing efficiency factor is showed as equation 3. This equation must be used to find the efficiency value, where the same chloride diffusion coefficient must be applied for both the control mix and the one with fly ash. With this procedure, the value of “kd” and its variation, in relation to other variables, was obtained.

The results obtained are expressed graphically in Figures 6 and 7. Analyzed according to the type of cement used, where values of the cementing efficiency are calculated based on the diffusion coefficient for non-steady-states.

The water/cement ratio plays a very important role in the efficiency values in terms of chloride diffusion. An increase in the water/cement ratio results in a decline of efficiency values. This increase causes a rise in porosity and connectivity of pores, as well as a substantial increase of the interfacial transition zone, impairing the contributions of fly ash.

The efficiency of high lime fly ash, at 28 days of age, gives values equal to or lower than one, thus showing its binding nature and producing a rise of “kd” when increasing their content in concrete (Fig. 6 y 7). By extending the time of hydration, there is a rise in the cementing efficiency when high lime fly ash is used with CEM I 42.5R, whereas, there is a fall when used with CEM I 52.5R. This may be because the control concrete with CEM I 52.5R generates more significant improvements than those generated by fly ash.

Whereas when using CEM I 52.5R at 90 days the efficiency values are significantly lower than one (Fig. 7). It can also be observed that an increase of fly ash content in concrete is accompanied by a rise in the cementing efficiency values, regardless of the type of cement used.

6 Conclusions

With the knowledge gained from existing literature, along with an extensive experimental campaign, it can be argued that the methodology used to determine the cementing efficiency coefficient is valid to obtain fly ash concretes with equivalent lasting properties to the control concrete.

In the practice, the studied concept of cementing efficiency of fly ash can be applied to predict the performance of concrete with mineral additions. The efficiency factor is defined by the fraction of fly ash, in mortar or concrete with admixtures, that can be considered equivalent to the behaviour of Portland cement in terms of compressive strength and chloride diffusion.

Overall, in relation to the studied variables, the cementing efficiency of fly ash behaves the following way:

- The cementing efficiency depends on the Portland-fly ash couple used.

- High-lime fly ash produces higher efficiency values at a young age, but, at more mature age, low-lime fly ash presents higher values.

- By increasing the water/cement ratio, there is a decline in cementing efficiency.

- By increasing hydration time, the cementing efficiency rises.

- By increasing the percentage of fly ash in materials, the value of cementing efficiency increases as well.

In general, it can also be observed that the chloride diffusion efficiency values are greater than 0.5, demonstrating that fly ash has a greater influence in durability than in strength, considering that the values of strength efficiency can sometimes be lower than cero (0), which indicates the detriment of a property.

Acknowledgements

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Table 1. Chemical composition of binding materials used

	Fe_2O_3	MnO	TiO_2	CaO	K_2O	P_2O_5	SiO_2	Al_2O_3	MgO	Na_2O	SO_3	LOI
CEM I 42.5R	2.55	0.03	0.25	61.06	0.71	0.10	19.90	5.17	1.69	0.00	3.5	3.87%
CEM I 52.5R	2.21	0.03	0.21	61.21	0.75	0.07	19.33	5.07	1.86	0.04	3.5	3.94%
HLFA	6.21	0.05	0.71	27.39	1.55	0.36	38.71	16.46	1.34	0.22	3.3	10.17%

Table 2. Physical properties of binding materials

	Specific gravity (g/cm^3)	Average particle size (μm)	Particle shape
CEM I 42.5R	3.09	10.20	Irregular
CEM I 52.5R	3.09	7.42	Irregular
HLFA	2.55	9.66	Spherical -Prismatic

Table 3. Dosage of concretes with w/b = 0.45 and 0.60

	Material quantity (kg/m^3)					
Material	A 0	A 25	A 43	B 0	B 25	B 43
Cement	378	302.4	264.3	283.3	226.6	198.1
Water	170	170	170	170	170	170
Fly ash*	0 %	25 %	43 %	0 %	25 %	43 %
Fly ash	0	99.4	149.1	0	74	111.3
w/c	0.45	0.56	0.64	0.60	0.75	0.86
w/binder	0.45	0.45	0.45	0.60	0.60	0.60
Coarse A.	675.3	675.3	675.3	695.3	695.3	695.3
Medium A.	450.2	450.2	450.2	463.6	463.6	463.6
Fine A.	750.3	750.3	750.3	772.6	772.6	772.6
Additive	2.1	1.5	1.2	1.13	0.7	0.4
Total	2426	2425.3	2425	2386	2385.5	2385.2

Fig. 1 Relationship between chloride diffusion coefficient and water/cement ratio

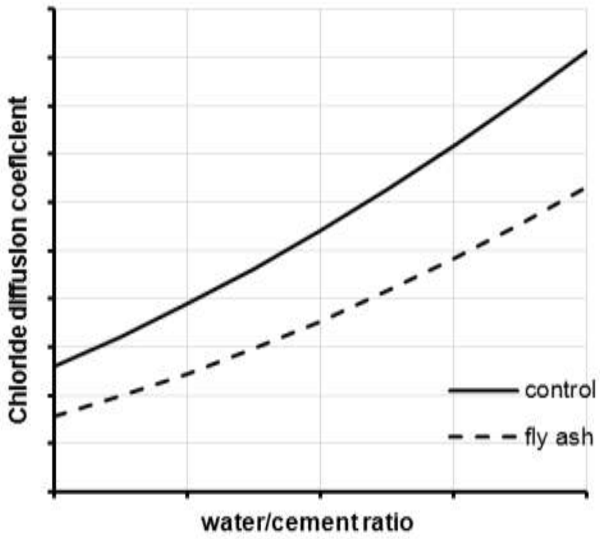


Fig.2 Evolution of compressive strength of concretes with CEM I 42.5R and high lime fly ash

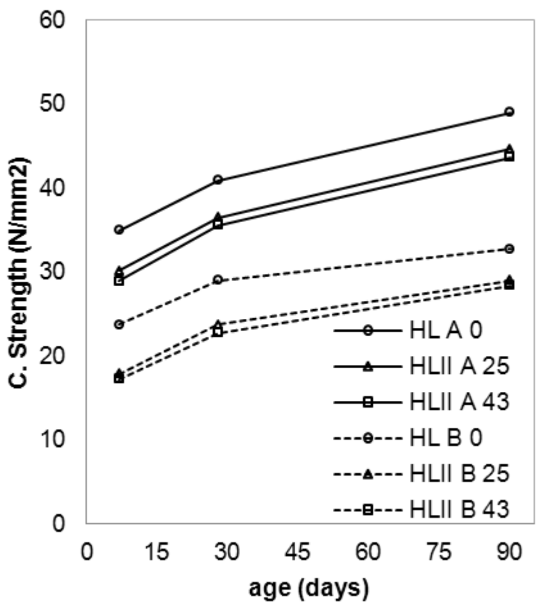


Fig.3 Evolution of compressive strength of concretes with CEM I 52.5R and high lime fly ash

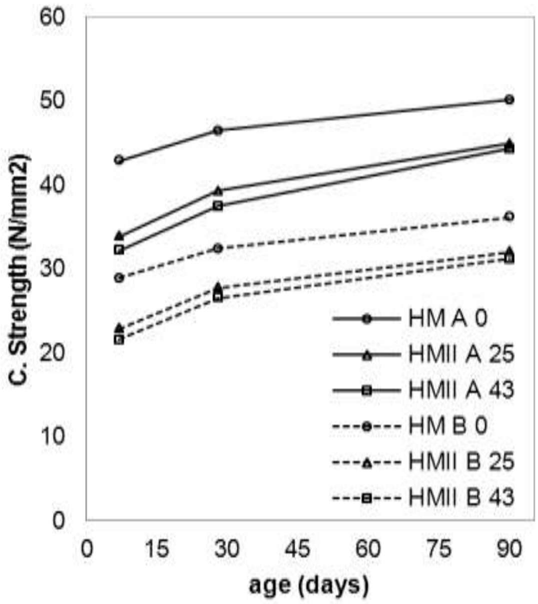


Fig. 4 Evolution of CDC of concretes with CEM I 42.5R and hig lime fly ash

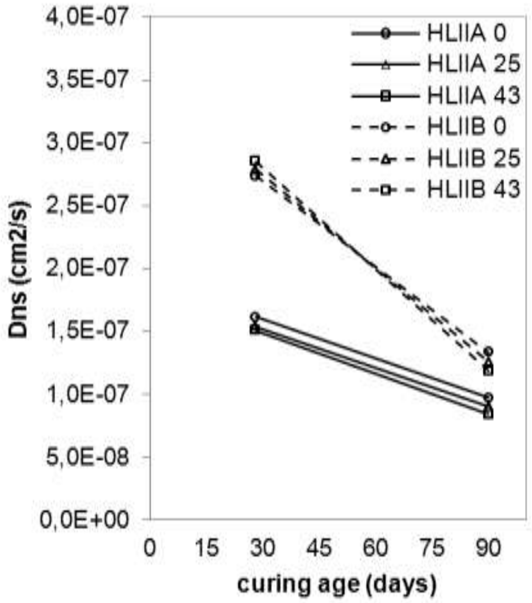


Fig. 5 Evolution of CDC of concretes with CEM I 52.5R and hig lime fly ash

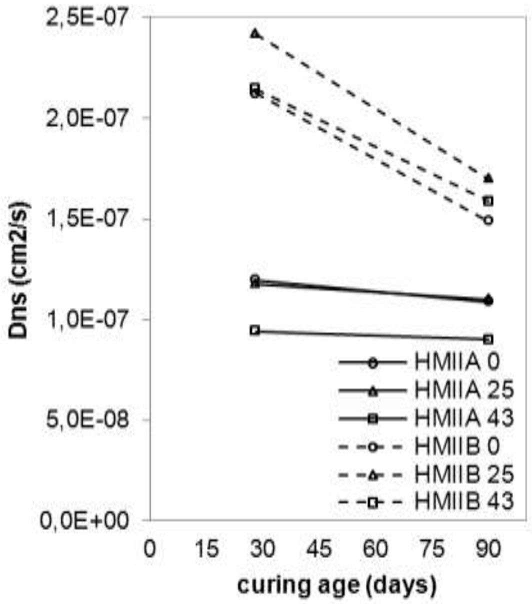


Fig. 6 Behavior of “kd” factor in concretes with CEM I 42.5R and high lime fly ash in relation to fly ash content, age and w/c ratio; a. a/c = 0.40; b. a/c = 0.525; c. a/c = 0.65.

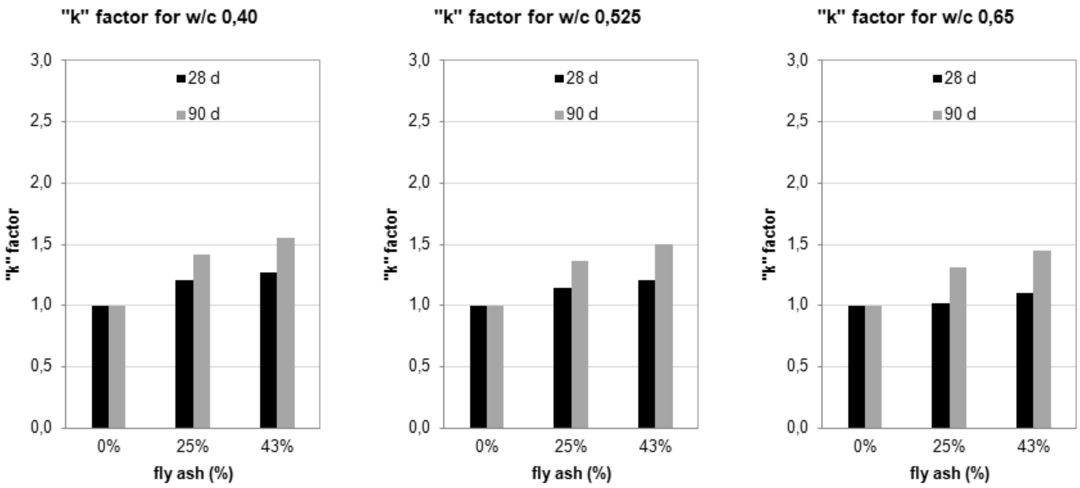
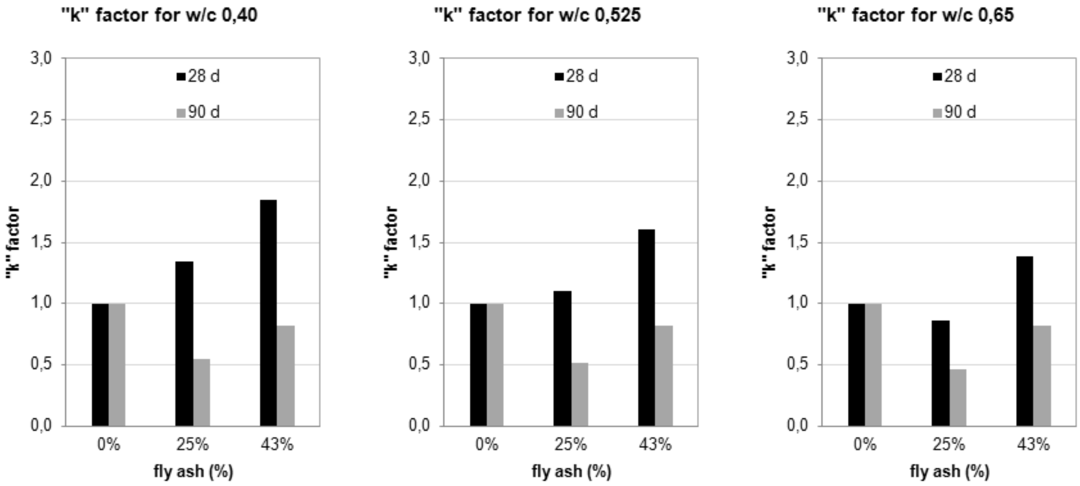


Fig. 7 Behavior of “kd” factor in concretes with CEM I 52.5R and high lime fly ash in relation to fly ash content, age and w/c ratio; a. a/c = 0.40; b. a/c = 0.525; c. a/c = 0.65


"k" factor for w/c 0,40
"k" factor for w/c 0,525
"k" factor for w/c 0,65
28 d
90 d
"k" factor
fly ash (%)
0%
25%
43%
0,0
0,5
1,0
1,5
2,0
2,5
3,0