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THE INFLUENCE OF FLY ASH AS SUBSTITUTE OF CEMENT IN THE DURABILITY OF CONCRETE

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Synopsis

Limitation of transport capacity through the concrete is one of the key points in the improvement of the material's durability. The use of fly ash as an admixture to concrete is widely extended; a general consensus has been established due to the pore size reduction produced by the ashes. Nevertheless, the importance of the micro-structural and composition changes in mechanical and durable properties is not well defined. In the present study the use of fly ash has been considered as substitute of cement in the design limits. The concrete mechanical properties and its porous structure were evaluated. The tests included porosimetry and water permeability tests. In order to characterize the hydration products and its evolution with time TG and DTA analysis were performed. This work studies the fly ash concrete hydration process, their influence in the porous distribution, and the mechanical and durable properties of the material.

Keywords: durability; fly ash; permeability; porosimetry; thermal analysis

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INTRODUCTION

In the last decades, important efforts have been made to improve the quality and durability of concrete. Numerous, researchers[1,2] as Pihlajavaara and Naik have found that pozzolans can improve concrete properties. The main improvements are lower heat liberation, the lime consuming, and a pore refinement process. Pozzolans as fly ash offer additional advantages as a lower cement dosage, less pollution associated with the production of cement, and a better solution for the ashes disposal problem.

Concrete is a cost-effective construction material. But, lifetime of infrastructures is limited by the decay of building materials. According to estimates made by Davies and Jones[3] in 1996, the repair cost of roads and maritime structures in Europe was over £ 1 billion annually. The amount is larger in the United States, where in 2002 the corrosion cost of its infrastructure was estimated to be 22.6 billion dollars annually[4], where such costs may increase at a rate of $500 million each year[5].

The Spanish Code of Structural Concrete[6] (EHE-98) defines the durability of concrete as "its capacity to withstand, during its projected useful life, the physical and chemical conditions that it is exposed to, and may cause its degradation as a consequence of effects other than the loads and strains considered in the structural analysis." On the same way, the ACI Committee 201 on durability of concrete defined the durability of portland cement concrete as its ability to resist weathering action, chemical attack, abrasion, or any other form of deterioration[7]. The *Comité Euro-International du Béton*[8] (CEB) states that the majority of the physical and chemical processes that have an influence in the durability of concrete structures are conditioned by the transportation of liquids through its pores and cracks. The pore structure formed may be classified by its size and distribution. According to the International Union of Pure and Applied Chemistry (IUPAC), pores are classified in: *micro-pores* ($Ø < 2$ nm), *meso-pores* ($2 \text{ nm} < Ø < 50$ nm) y *macro-pores* ($Ø > 50$ nm). Those that affect the durability of concrete are the *meso-pores* and *macro-pores*, especially in the case of interconnected pores and open porosity, which facilitates the transport of soluble substances in liquid or gaseous forms.

Portland cement hydration and fly ashes reaction

There are two principal reactions in concretes with fly ashes. The first one produces a C-S-H gel ($C_3S_2H_4$) and calcium hydroxide (Portlandite) due to the hydration of portland cement, equation (1). In the second one, the Portlandite (CH) is combined with silicon oxide (SiO_2), aluminum oxide (Al_2O_3), or ($SiO_2 \cdot Al_2O_3$) of the fly ashes (FA) to generate new gels, equations (2, 3, and 4).

$$2C_3S + 7H \rightarrow C_3S_2H_4 + 3CH \quad (1)$$
$$3CH + (SiO_2)\ FA + H \rightarrow CxSyHz \quad (2)$$
$$3CH + (Al_2O_3)\ FA + H \rightarrow CxAyHz \quad (3)$$

$$3CH + (SiO_2 \cdot Al_2O_3)\ FA + H \rightarrow CxSyHz \cdot CxAyHz \quad (4)$$

The variety of chemical compositions and physical properties of the fly ashes make it hard to predict in a general way how the characteristics of concrete will be modified. For this reason, the American Concrete Institute[9] (ACI) recommends the analysis of fly ashes and the realization of tests characterizing concretes with fly ashes.

Thermal analyses (TA) were used in the present study for the determination of pozzolanic activity of the ashes. The techniques are thermogravimetric (TG) and differential thermal analysis (DTA). The TA methods are based in the variation of the mass of a material while varying the temperature. The changes studied while warming the concrete samples with and without fly ashes may be summarized as the loss of mass mainly due to dehydration reactions. In the TG curve the mass variation is shown as a function of the temperature, so that a mass variation may be produced in a certain interval. By studying the fluctuations of the curve, the researchers may determine the variation in the sample's mass. Such variations are typical to the chemical reactions inherent to the process. The DTA curve graphically represents the difference between the sample's temperature and that of its surrounding environment, with the temperature, when it is increased in a determined atmosphere. The closed area between the curve and the baseline is related to the heat of the process. This allows the perception of the proportions of the different compounds present in the concrete.

Durability

Various laboratory and field observations[10] demonstrate that concrete with fly ashes are more permeable than concretes without ashes at an early age, regardless of the calcium content of the ashes and for replacement levels of up to 50%. The tendency inverts after 180 days due to the pozzolanic activity of the ashes.

Researchers Manhoman and Mehta[11] argue that the calcium hydroxide liberated in the hydration process of the cement is water-soluble and could lixiviate from the concrete's mass, leaving vanes that would allow the introduction of fluids. The fly ashes react chemically with the calcium hydroxide liberated to form new C-S-H gels, reducing the possibility of lixiviation. Additionally, the long term reaction of the fly ashes refines the structure of the concrete pores, reducing its permeability to fluids and all compounds contain therein.

Conglomerates composition and fineness

Frayy[12] investigated the pozzolanic reactions and suggested that the reaction of the fly ashes began after 7 days from being mixed, where in the first week the ashes behaved as an inert material within the mixture. Sybertz and Wiens[13] found that the fineness of the ashes accelerated its pozzolanic activity. In a study with type I portland cement and five fly ashes from different origins, the researchers[14] concluded that the fineness of the ashes, and not its chemical composition, have a significant effect in the compression strengths of all the studied mortars.

Professor Fernández Cánovas[15] explains that in the case of the cement fineness, manufacturers may produce cements with similar characteristics by using different dosages of C_3S. Cements with low tricalcium silicate content achieve the same strengths as cements with larger amounts of C_3S by increasing the fineness of the cement. However, finely ground cements present a number of inconveniences, such as an elevated grinding cost, retraction and propensity to fissures, stronger reactions of the cement alkalis with aggregates, and a larger demand for water.

It is reasonable to assume that in cements with fly ashes added, the inclusion of finely grated cements favor the early reaction between the ashes and the liberated portlandite during the cement hydration process[16]. On the other hand, it is known that when the composition of cement is observed, the proportion of silicates (C_3S/C_2S) controls the amount of calcium hydroxide produced, the hydration at an early age, and the development of strengths. Stoichiometric calculations show that the hydration of C_3S produces 2.2 more calcium hydroxide than the same amount of C_2S[17]. Actually the performance of the ashes depends on the cement provided for the construction industry, rather than in the preparation of the cement to complement the ashes. The result is that the specifications of the ashes are designed so that they may adjust to the cements available in the market. To adequately exploit the fly ashes that exist in the cement market today, the cement should be formulated to optimize the performance of the combination in terms of consistency, strength and durability[18].

RESEARCH SIGNIFICANCE

A good number of the studies on blend cement were carried out to judge the properties of fly ash. Studies on the influence of the cement composition in fly ash concrete durability are very scarce in the technical literatures The objective of this research work is to study the influence of the fly ashes, as partial substitute of the portland cement in concrete durability, in accordance to the EHE-98. To achieve these objectives mechanical characterization tests were performed as well as the tests for determination of resistance to water penetration by hydrostatic pressure, mercury intrusion porosimetry and TG/DTA.

EXPERIMENTAL INVESTIGATION

Materials

EHE-98 specifies that the concretes with fly ashes added must be manufactured with CEM I type cement. This work utilized two CEM I 42.5 R cements with different C_3S/C_2S relationships. The first one developed strengths due to the fineness of the grinding, denominated as "F", and the second, due to its composition, denominated "G".

The aggregates employed are siliceous, and they are from the same source. The coarse aggregates used have a maximum diameter of 20 mm, from crushed stones, and its granulometric module is of 7.08. The fine aggregates have a sand equivalent of 76% and a granulometric module of 2.86.

The fly ashes utilized are siliceous, and with a low content of calcium oxide, classified as type V, in accordance to the Spanish Cement Reception Code[19] (RC-03). Additionally, they comply with UNE-EN 450-1:06 standard for their usage as a concrete admixture. They proceed from the thermoelectric power plant of Andorra (Teruel, Spain). The chemical composition of both cements and the fly ashes are presented in **Table 1.** The cements were analyzed by *Instituto Eduardo Torroja de Ciencias de la Construcción* (IETcc). **Table 2** shows the characteristics of the major compounds in the portland cements. They are in accordance with the UNE 80304:06.

Dosages

Bolomey method was used for the concrete mixture proportions. All mixtures, as a fix value, used 350 kg per cubic meter (m^3) of concrete, along with a w/c of 0.65. The dosages of concrete with fly ashes followed the recommendations of UNE 83414:90 EX. Furthermore, following the *Limitations to the contents of water and cement*, EHE-98 (art. 37.3.2) for cements with admixture, c is substituted by c + KF, where K is the coefficient of efficacy of the fly ashes, and F is the content of the admixture. Hence, the w/c is substituted a/(c + KF). The fly ash substitution levels are in accordance to EHE-98. The maximum amount of fly ash allowed by this code is 35%. The ashes efficacy coefficient was K = 0.30. **Table 3** shows the dosages used of each material in the mixtures.

Specimens

The concrete was mixed in a vertical axis planetary mixer, with a capacity of 100 liters. The test specimens were 15 cylindrical samples of 15 cm diameter by 30 cm height. The mixing and curing processes followed the recommendations of ASTM C 192.

Items of investigation

Table 4 enumerates the tests performed and the ages of the samples at the date of the test. The porosity of the samples was measured through Mercury Intrusion Porosimetry (MIP). The technique utilized is standardized by ASTM D4404-84 (2004). A Micrometrics, model Autopore IV 9500 was used for this experiment; operating at pressures of 33.000 psi (228 MPa), covering a pore diameter range from 0.006 to 175 µm. Every sample was conditioned by a preheating and degasification process, where the sample was dried at a temperature of 40 °C, until constant weight and was later degasified with a vacuum pump. The sample mass was 3.5g ±3.

For the TG/DTA analysis, samples were prepared from a 10 mm slice of concrete from the center of the cylindrical samples. Before the heating process, the samples were dried and pulverized until passing the 0.5 mm screen. A Simultaneous Thermal Analyzer by STANTON, model STA 781 with a precision scale of 0.1 µg, was utilized. The dynamic heating ramp varied between 20 and 1,000 °C. The heating rate was 10°C/min, and the cresols utilized were made of platinum (Pt). The reference material is α-alúmina (α-Al_2O_3), and N_2 was used as inert atmosphere.

EXPERIMENTAL RESULTS

Compressive strength

Fig. 1 shows the results of compressive strength for each mixture proportions and cements employed. The values presented correspond to the average value of the three samples tested.

The samples mixed with the "F" cement present better compressive strength results than the samples made out of the "G" cement, under the same conditions. This difference is seen at all ages, for all the proportions of cement ash. Furthermore, the addition of ashes between 15% and 35% has no significant effect in the compressive strength for the same type of cement. At the same time, the authors observed that the compressive strengths of concretes with fly ashes are superior to the compressive strengths of concretes without (reference) after 28 days, and particularly at 91 days. However, after 7 days, the compressive strengths of the concretes with ashes are lower than their reference concrete.

Splitting tensile strength

Fig. 2 demonstrates the results of tensile strength (splitting tensile test) for each mixture proportions and cements utilized. The presented values correspond to the average value of the three tested samples.

Those mixed with cement "F" show more tensile strengths than those mixed with G cement. These differences emerge at all ages for all the fly ash/cement. Additionally, the incorporation of ashes between 15% and 35% has no significant impact on the tensile strengths for the same type of cement. In the case of concretes mixed with "F" cement, the addition of ashes does not substantially modify its behavior, where the difference at 91 days is only of 0.1 MPa. However, in the concretes with "G" cement, the improvement was superior under the same conditions. For the tensile strength, the difference between the two cements employed is more notable than the one caused by the addition. Hence, the concretes made with "F" cement and without ashes demonstrated the same or superior tensile strength values than any of the combinations obtained with "G" cement.

Modulus of elasticity

Fig. 3 shows the results of the modulus of elasticity for each mixture proportions and cements used. The presented values correspond to the average value of the three samples tested.

The values measured at 91 days show that the concretes with "G" cement have a larger value of modulus of elasticity than those made with "F" cement. The concretes with fly ashes have larger elasticity values than those without after 28 days, regardless of the cement utilized.

Water permeability and mercury intrusion porosimetry

Fig. 4 shows the results of water penetration under pressure and the average diameter of the pores for each mixture proportions and cements utilized 91 days after mixing. The presented values correspond to the average value of the three samples tested.

The concretes without ashes demonstrate no apparent difference among themselves, hence reference is made to water penetration and average size of the pore. The results demonstrate that the permeability and the average diameter of the pores are reduced with the addition of fly ashes. The concretes made with "G" cement are less permeable than those made with "F" cement. This aspect is confirmed with the smaller average size of the pores, as well as the results of the mercury intrusion tests. **Fig. 5** presents the compressive strength and total porosity as measured by mercury intrusion 91 days after mixing. Concretes with "G" cement have a larger total porosity than those made with "F" cement. This figure demonstrates that the increases in porosity reduce the compressive strength. **Figs. 6 and 7** demonstrate the distribution of pores in the studied samples. Concretes with "G" cement have a larger volume of meso-pores than those with "F" cement. In the case of concretes with fly ashes, the concretes made with "G" cement have a lower volume of macro-pores than the concretes with "F" cement.

Thermogravimetric/Differential Thermal Analysis

Fig. 8 shows an example of the TG/DTA curves for the concrete sample. The figure corresponds to concrete at 28 days. The loss of mass in the zone between 100 and 200ºC is attributed to dehydration of C-S-H gel. The step between 410 and 580ºC is due to decomposition of portlandite. The loss before the portlandite step is due to decomposition of C-S-H and hydrated aluminate phases, but in this zone samples show slight step indications. The absence of steps is probably due to a combination of low crystallinity, the presence of another phase, and the presence of AF_m phases of different compositions in mixture, or solid solution, or both. The zone between 570 and 573ºC is assigned to the crystalline inversion of unreacted quartz, from aggregates in the concrete, and they are present in all samples. The last zone is located between 700 and 900°C, and it belongs to the decarbonatation process. The identification of mineral components and their quantification, using TG/DTA, is difficult due to overlapping. The difficulty level increases in the concrete samples due to the presence of aggregates. On the other hand, the use of concrete allows more realistic results. In the case of the samples with pozzolanic additions it is difficult to distinguish the hydration products due to the hydration of cement (C-S-H gel) to the gel produced during the pozzolanic activity of fly ash. Moreover, the presence of pozzolan particles contributed to accelerate the hydration of cement particles as it can be seen in **Fig. 9.** This shows that, independent of the cement properties, the presence of 15% or 35% of fly ash accelerates the hydration process, and produces an increase of percent weight loss of the dehydration and dehydroxilation products at 28 days. The results obtained from the TG/DTA analysis after 91 days, **Fig. 10**, appear to indicate the transformations of the C-S-H gel phase into other hydrated phases and the pozzolanic activity. These changes have been observed by other researchers[20].

One of the objectives of the present research is to study the pozzolanic activity of the fly ash 15% of substitution respect to 35% of substitution at 28 and 91 days. For this, the mass losses of the zones corresponding to calcium hydroxide (CH) were evaluated, so that researchers could calculate the percentage of fixed lime[21] by the fly ashes, as expressed below:

$$Fixed\_Lime(\%) = \frac{CH_C * C_{(\%)} - CH_P}{CH_C * C_{\%}} 100 \quad (5)$$

Where CH_C is the amount of CH in the concrete without fly ash for a given curing time, CH_P is the amount of CH in the concrete with fly ash at the same age and $C_{\%}$ is the proportion of cement in the concrete. The dehydroxylation of CH is given by the following reaction:

$$Ca(OH)_2 \rightarrow CaO + H_2O \quad (6)$$

The registered loss in the portlandite water zones corresponds to the water freed in this dehydration (H). The evaluation of the mass loss is done in a tangential form, since the baseline before the decomposition is not horizontal. Finally, to obtain the amount of CH present in the mixture, according to the stoichiometry of the reactions, authors made the following calculation:

$$CH = \frac{H}{MW_H} MW_{CH} \tag{7}$$

Where H is the loss due to the water registered in the zone of portlandite, MW is the molecular weight and the subscripts indicate whether it is water (H) or calcium hydroxide (CH). In order to observe the amount of lime fixed by the fly ash, **Fig. 11**, the following expression was used:

$$Incremet(\%) = \frac{\%Fixed\_Lime_{with\_FA} - \%Fixed\_Lime_{without\_FA}}{\%Fixed\_Lime_{without\_FA}} \tag{8}$$

DISCUSSION

The tests performed clearly demonstrate an improvement in the compressive strength for concretes with fly ashes in the cements studied, particularly 91 days after mixing. The improvements in tensile strengths are appreciated as well, even though it is less significant. The values of the modulus of elasticity also exhibit a tendency to stay or increase after 7 days. The mechanical tests indicate that the concretes created with “F” cement show superior compressive strength and tensile strength to those created with “G” cement. It is important to point out that in the splitting tests the combination of fly ash with “G” cement is more effective than with “F” with regards to the sample that did not include any ashes. For the values of the modulus of elasticity the combination of ashes with “G” cement exhibits larger values than the ashes with “G” cement at 91 days.

The different percentages of ashes used demonstrate that there is an optimal value for mechanical behavior in a percentage between 15% and 35%. This fact is explained if a variation factor of the efficiency of the ash as a function of the percentage used is admitted. Addition values close to 15% appear to proportionate high efficiency levels due to a better contact between the ash and the cement’s hydration products, which is essential for the pozzolanic reaction of the ashes. An elevated value of the ashes seems to impair the optimum utilization of the ash since it makes it more difficult for the fly ash and the portlandite to make contact, reducing its efficiency coefficient. Hence, the total value of the porosity, as well as the values of the compressive strength and permeability of water, demonstrates a tendency to stabilization when the ash content is increased.

The behavior towards the permeability of the tested concretes demonstrates a great dependency on the average pore diameter for all the cements studied. For the same or similar construction of the matrix, a reduction in the pore size causes a reduction in the permeability values. The reduction of total porosity in the concretes for every cement studied, along with the inclusion of the different additions of ashes, caused an increase in resistance. However, the concretes mixed with cement “F”, which demonstrated the highest compressive strength values, did not obtain the best results in the pressured water penetration test.

The TG/DTA analysis may be correlated with compressive strength at 28 days. The results demonstrate that the fly ashes and the “G” cements increase the compressive strength in 20% at 28 days. The concretes mixed with fly ashes and “F” cement increases is close to 9%. This coincides with the quantities corresponding to dehydration of the C-S-H gel. Whereas with the “G” cement and fly ashes the dehydration is of 38%, the combination with “F” cement shows an improvement of 27%. Thermal analyses show the development of the pozzolanic activity of concretes at 91 days. It is confirmed that reference concrete with cement “G” reacts more slowly than with cement “F”. Likewise the fly ashes pozzolanic activity is observed by the decrease in the values of dehydroxylation.

The fixed lime evaluation demonstrates that the mixtures with “F” cements and 15% substitution show better pozzolanic activity than mixes with “G” cement under the same conditions. These differences are clear at days 28 and 91. The mixtures with “G” cements and 35% substitution demonstrate better pozzolanic activity than the mixtures with “F” cement under the same conditions.

The relationship C_3S/C_2S in cement “G” is three times higher than in “F”. The direct result is a greater amount of portlandite in these concretes. The results show a reduction of pore diameters, a lower volume of macro-pores and less water penetration depth in the concretes prepared with cement “G”. The pozzolanic reaction has been benefiting from the use of cement “G”. This is possible due to a larger availability portlandite.

CONCLUSIONS

These results allow for a better understanding of the behavior of concretes with fly ashes as a partial substitute of portland cement. From the results obtained, the authors may conclude that:

1. Fly ashes contribute to an improvement in the mechanical properties of concrete at middle-terms (tests at 91 days).
2. Fly ashes contribute to decrease the permeability of concrete at a middle-term. This improvement manifests itself in the modification of the average diameter of the pore and in its size distribution.
3. The C_3S/C_2S relationship may help to explain the behavior of the porous structure and water permeability of fly ash concrete.
4. TG/DTA analyses provide information about the hydration products and pozzolanic activity in concrete and this information may be related with the mechanical properties of concrete at 28 days.
5. The computation of fixed lime demonstrates that a 15% substitution exhibits better properties when using “F” cement rather than “G” cement, allowing for better compressive strength. The inverse situation occurs with a substitution of 35%.
6. The effectiveness of fly ashes is not only determined by its own characteristics. The chemical and physical properties of the cement influence on the effectiveness of ashes and the ideal replacement volume.
7. The behavior of concretes with fly ashes varies as a function of the physical and chemical properties of the cements used, even though these are under the same category of common

cements. The different combinations may enhance various mechanical and physical properties of concretes.

8. High mechanical strengths of concretes with fly ashes do not imply achieving the best lasting or physical properties.

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TABLES AND FIGURES

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Table 1: Chemical composition

Test	Results (%)			Standards
	Holcim (F)	Cemex (G)	Fly Ash	
Loss on ignition	1.39	4.02	1.14	UNE 8015
Insoluble residue	1.07	0.74	---	UNE 196-2
Sulfur trioxide, SO_3	2.96	3.32	0.85	UNE 196-2
Al_2O_3	5.29	6.3	30.02	UNE 80230
Fe_2O_3	2.47	2.71	19.93	UNE 80230

CaO	62.29	63.06	8.09	UNE 80230
MgO	2.42	1.96	1.70	UNE 80230
Free CaO	0.30	1.18	0.10	UNE 80243
SiO_2	20.87	19.12	40.56	UNE 196-2
Na_2O	0.26	0.35	0.22	AES
K_2O	0.95	0.93	1.13	AES
AES: Atomic Emission Spectroscopy				

Table 2: Characteristics of the major compounds

Cements	C_3S	C_2S	C_3A	C_4AF
Cement *F*	56,06	17,56	9,84	7,51
Cement *G*	65,13	5,84	12,12	8,24

Table 3 – Dosages for a m^3 of concrete

Fly ash / Cement	Portland Cement (kg)	Fly Ash (kg)	Fine Aggregates (kg)	Coarse Aggregates (kg)
0,00	350,0	00,0	918,0	780,0
0,15	334,9	52,2	878,5	780,0
0,35	316,7	110,9	830,6	780,0

Table 4 – Tests performed

Tests	Time (days)			Standards
	7	28	91	
Compressive strength	X	X	X	UNE-EN 12390-3:2003
Splitting tensile strength	X	X	X	UNE-EN 12390-6:2001
Modulus of elasticity	X	X	X	UNE 83316:1996
Water penetration	--	--	X	UNE-EN 12390-8:2001
MIP	--	--	X	ASTM D4404-84(2004)
TG/DTA	--	X	X	----

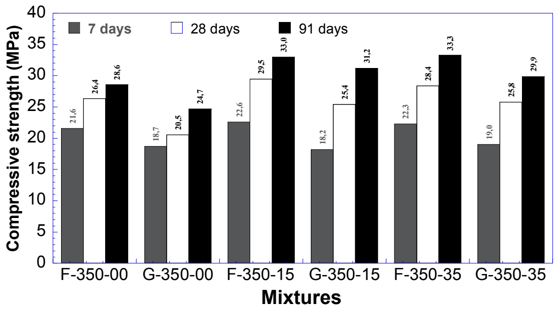

Fig. 1 – Compressive strength

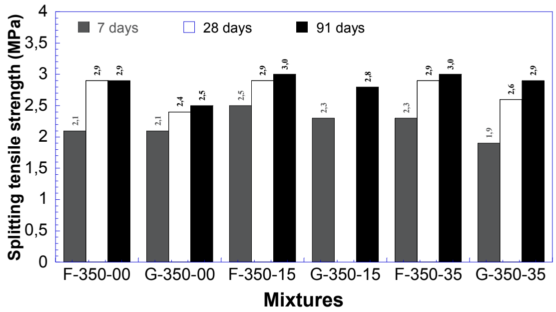

Fig. 2 – Splitting tensile strength

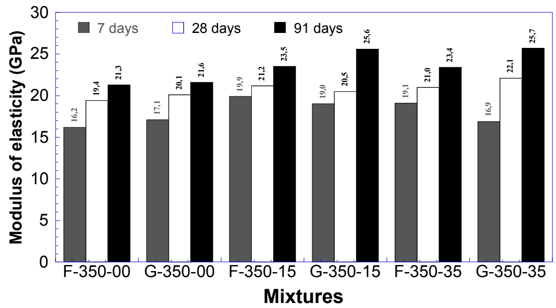

Fig. 3 – Modulus of Elasticity

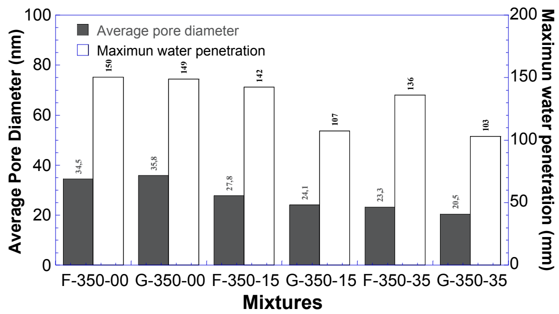

Fig. 4 – Water permeability and MIP average pore diameter (91 days)

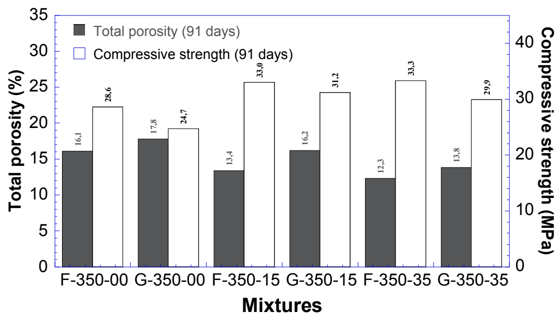

Fig. 5 – Compressive strength and MIP total porosity (91 days)

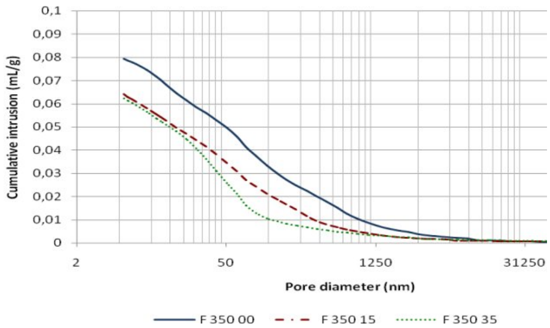

Fig. 6 – MIP pore distribution: cement "F" (91 days)

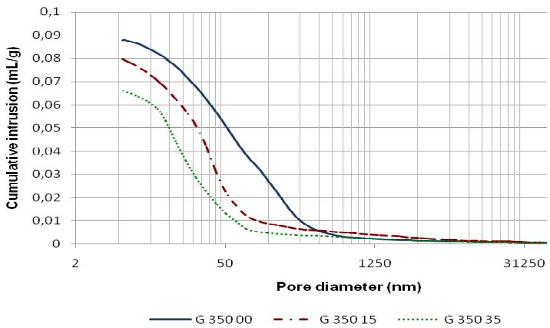

Fig. 7 – MIP pore distribution: cement "G" (91 days)

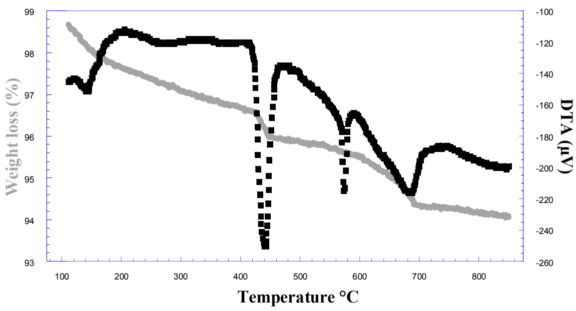

Fig. 8 – TG/DTA sample curves

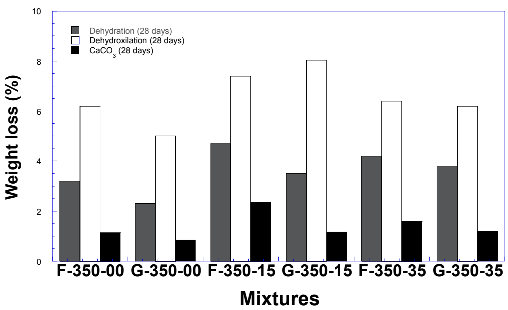

Fig. 9 – Thermogravimetry/Differential Thermal Analysis (28 days)

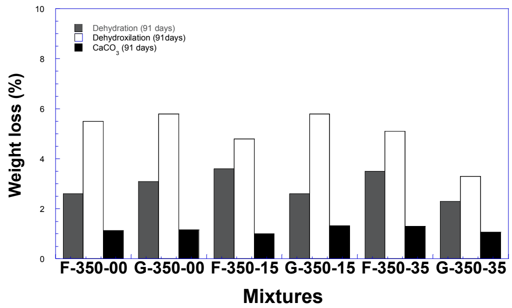

Fig. 10 – Thermogravimetry/Differential Thermal Analysis (91 days)

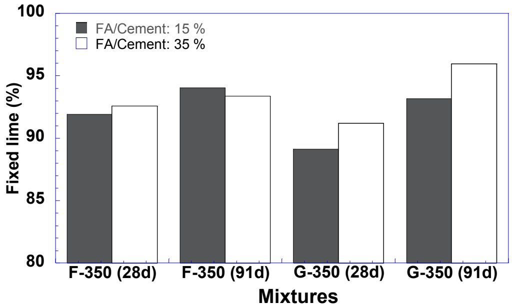

Fig. 11 – Fixed lime evaluation