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EARLY AGE BEHAVIOUR OF SELF COMPACTING CONCRETE WITH POLYPROPYLENE FIBERS AND CARBON NANOFIBERS

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Summary:

An experimental study on the performance of a self compacting concrete (SCC) with fibers, during the cement hydration process and the evolution in to a rigid material, was carried out. The aim of this study was to investigate the improvement of the hardened performance due to the use of fibers and the early age cracking control ability of fibers at two different scales: on a nano-structural level with the addition of carbon nano-fibers (CNF) and on a micro-structural level with the addition of polypropylene micro-fibers. The inclusion of nanofibers increased the flexural strength of cement based pastes although some problems concerning early age cracking emerged.

As any other concrete, SCC can exhibit problems during the very early ages after mixing, when cement hydration occurs, as shrinkage and early cracking. Although these problems arise during early ages, their effect can seriously compromise the long term performance and durability of the material.

SCC samples with and without fibers were monitored through its hardening process using several experimental techniques and considering the effect of water evaporation due to wind exposition during the setting process. The effect of the fibers and different SCC components, as nanosilica, and a high range water reducing admixture (HRWRA), was analysed. Besides, the ability of the applied components to avoid the stress development during the early ages was also evaluated. The influence of the fibers and additional components on the early age shrinkage was assessed, identifying the relationship between shrinkage and early age cracking.

1 INTRODUCTION

Many studies have been published searching for an effective method to mitigate early age cracking in concrete. Beyond limiting the water loss from the exposed surfaces of the concrete members during early ages, other possible ways are controlling the environmental conditions or modifying the concrete composition [1].

The addition of fibers is an effective way to fulfill that goal. The material, length and orientation determine the cracking control ability of the fibers. Fibers efficiency bears on their ability to distribute, absorb and transmit the stresses, as fibers counteract cracking growth. Furthermore, in the hardened state under the effect of stress, the fibers sew the cracks, allowing larger fracture stresses [2-3].

In order to asses the effect of fibers on the early age behaviour of a self compacting concrete (SCC), an experimental study was carried out, using fibers in two different scales: on a nano-structural level, using *carbon nano-fibers (*CNF) and on a microstructural level, adding polypropylene (PP) micro-fibers.

The mechanical properties of CNF, as Young modulus and tensile strength, make this type of fibers good choice for nano-scale reinforcement [4]. One of the main problems concerning to the addition of CNF is their dispersion into the fresh concrete. When the CNF are prepared as a stable water suspension, a homogenous distribution in the fresh mixture can be obtained.

PP fibers (19 mm length) were included in a proportion of 900 g/m³, jointly with the CNF. PP fibers produce a multi hybrid fiber reinforcement at different scales, due to their shape, distribution ability and mechanical properties, providing additional performance to the concrete [5].

The use of self compacting concrete (SCC) has become one of the most efficient solutions within the world of the concrete construction [6]. SCC performance in the hardened state is quite similar to ordinary Portland concrete (OPC). However, some studies have pointed out some differences between SCC and OPC behavior in the fresh state and during early ages. An increase of early age shrinkage related to the higher amount of fines in the composition has been described [6-7]. This larger shrinkage, along with other circumstances, can lead to an increase on early age cracking risk.

Concrete shrinkage take place at early ages (EA), 24-48 hours, and is related to three main causes: drying, thermal and autogenous phenomena [8-9]. The main effect of EA shrinkage is early cracking, which produces a permanent damage to the material that can compromise durability. However, the relationship between shrinkage and cracking is not entirely clear, because larger shrinkage do not mean larger cracking [10]. Shrinkage is an outcome of the hydration process, the environmental effects and the stiffening of young concrete. Depending on the boundary conditions of the concrete members, shrinkage can produce stresses in the material during the hardening process. If the material is not able to absorb the stresses generated, the material cracks [10-11].

The volumetric variation is closely related to the role of water in the concrete composition and its interchange with the environment [12-13]. Two main groups of factors can be identified: the internal factors, such as physical-chemical reactions that occur during concrete setting and hardening, and the external factors, which involve water evaporation due to environmental or climatic conditions or water subtraction by the formwork or the dry components.

The aim of the experimental study was to monitor the changes on the SCC behavior during the early ages when fibers and nano-additions were used. The experimental setup consisted of several tests to determine measured horizontal shrinkage, the state evolution from plastic to rigid behaviour, through ultrasonic pulse velocity, the temperature variation of the sample, as well as surface cracking of a double-restrained slab sample.

In order to identify the effects of composition changes, the tests emulated environmental conditions. The tests were performed with a constant airflow of 3m/s, in order to increase the potential for cracking of the sample, due to water loss from the exposed surface (larger evaporation and shrinkage).

2 EXPERIMENTAL PROGRAM

2.1 Materials

A reference composition of a OPC (HREF) and six types of SCC (Inclusion of a HRWRA, cement replaced by filler, the incorporation of additives and nanoadditions, and the addition of carbon nanofibers and polypropylene fibers) were studied. Fibers were added in order to improve tensile behaviour and to control cracking at different scales. The J ring test was used to evaluate the flowing and passing ability of all mixtures. The coarse aggregate distribution was uniform and no segregation of the samples was observed. The spread diameter of the samples corresponded to SCC

Samples of the pastes (without aggregates) were also manufactured.

	HREF	HREF G	HREF GAIA	HCA	HCAGAIA	HCAGAIA CNF	HCAGAIA CNF FPP
Cement CEM I 42,5 R	700	700	700	350	350	350	350
Limestone Filler (Betocarb P1-DA)	-	-	-	350	350	350	350
Water (*)	207	201.75	206.1	204	206	206.5	215
HRWRA (Glenium ACE425)	-	10.5	-	5.25	-	-	-
HRWRA + Nano-silica (Ulmix NS SCC)	-	-	7	-	3.5	3.5	3.5
CNF (Carbon-Nano-Fibers)	-	-	-	-	-	58.3	58.3
FPP (Polipropylene fibers)	-	-	-	-	-	-	0.9
Coarse aggregate (4-20 mm)	790	790	790	790	790	790	790
Sand (0-4 mm)	693	693	691	691	691	691	683

W/c (**)	0.36	0.36	0.36	0.71	0.71	0.71	0.71
W/fines (cement + additions) (**)	0.36	0.36	0.36	0.36	0.36	0.36	0.36
djf(***) mm	-	875	820	815	825	765	725
CbE(***) %		37%	95%	28%	62%	94%	85%

(*) Liquid water added.
(**)The amount of water included in the sand and minor components (HRWRA & CNF) was also taken into account.
(***)UNE 83362 :2007- SCC Characterization of flowability through rebars. Slump-flow test with J-ring.

Table 1 : SCC compositions considered in the study.

The cement type was a CEM I 42.5 R type, supplied by Portland Cement Valderrivas Company [14]. In all the mixtures of this study, the water to fines ratio (w/f) remained constant at 0.36.
A conventional high range water reducing admixture (HRWRA) (425 Glenium ® ACE produced by BASF, polycarboxylate-based) was added to the reference concrete (HREFG). Another superplasticizer additive (HRWRA) combined with nanosilica (NS). (NS Ulmix ® developed by Ulmen SCC), in a amount of 1% on with regard to cement weight, was considered (HREFGAIA). Both mixtures presented a self compacting ability.

In both SCC reference mixtures, 50% of the cement was substituted by limestone filler (P1-DA Betocarb ®, produced by Omya Clariana SL), obtaining two conventional SCC mixtures (HCA and HCAGAIA, respectively).

Carbon nanofibers (CNF) prepared in an aqueous suspension (GANF ® provided by Grupo Antolin Company) at 0.5% of cement weight and polypropylene fibers (900g/ m³ of 19 mm length produced by Grace) were also added to the second conventional SCC mixture with limestone filler (named HCAGAIACNF and HCAGAIACNF FPP, respectively).

2.2 Experimental method

The characterization tests were done in both the fresh and the hardened states of the concrete mixtures of this study. The early age performance was monitored with a multi-test setup during the first 24 hours after mixing, which produces a good indication of the physical and mechanical evolution of the samples during this period [12, 15 -16].

The mixing process was as follows: the dry components were mixed first and the water was added afterwards. At last the additives (HRWRA) and fibers were incorporated subsequently. Average mixing times did not exceed five minutes. Cubic samples (100 x 100 x 100 mm) were manufactured and water cured. The samples were tested in the hardened state at 1, 7 and 28 days. Compressive strength, apparent density, porosity and ultrasonic young modules were measured. Flexural strength was measured on prismatic paste samples (40 x 40 x 160 mm).

At early ages, temperature, ultrasonic pulse velocity (UPV), water evaporation (mass loss) and shrinkage were tested.

For the Temperature and Ultrasonic pulse velocity was used a plastic mold (150 x 100 x 70) [17], in the longitudinal axis on each face was made a 50 mm diameter hole, in order to place the ultrasonic transducers in direct contact with the fresh concrete. A TESTO thermometer and a ultrasonic PUNDIT PLUS equipment (54 kHz transducers placed at 140 mm), was used during the first 24 hours after the placement of the material. The test setup is shown in Figure 1. [17].

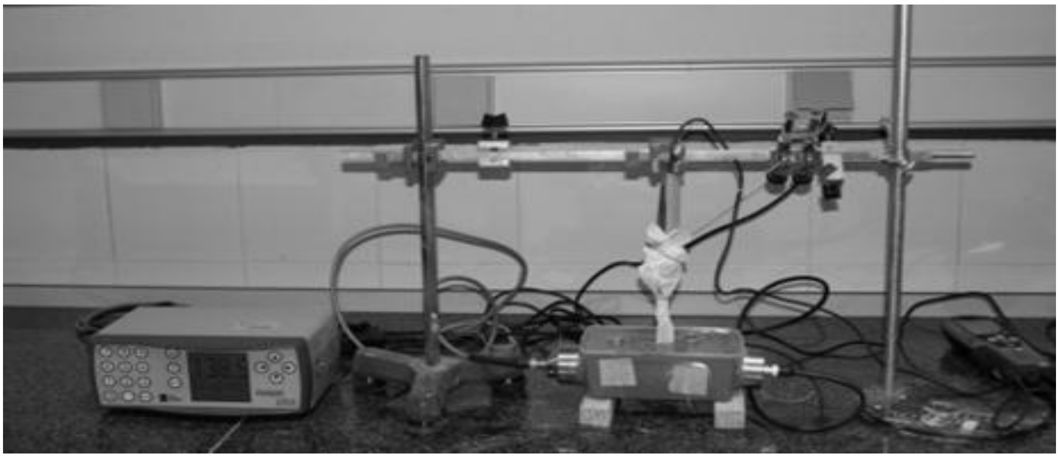

Figure 1: Ultrasonic and temperature monitoring setup.

Free shrinkage and mass loss were monitored on the same sample, as it can be observed in Figure 2. The sample was placed in a 500 x 100 x 50 mm metal tray connected to a LVDT displacement electronic measurement. To maximize water evaporation and shrinkage of the sample an air flow of 3 m/s during the first 6 hours was applied, wich is a common test condition, [11,15].

This early age multi-test monitoring procedure has been previously described [10], as the methodology used to generate a volumetric potential variation. Considering the same environmental conditions (air flow of 3 m/s during the first 6 hours) cracking risk was assessed with samples placed in a 600 x 900 x 50 mm mold with restraining anchorage in both directions of the plane with multiple galvanized steel U shaped pieces arranged in the mold base in Figure 3a, (Kraai slab modified test)

[18]. The slabs were demolded after 24 hours and then the fraction of cracked to the total slab area was measured, taking into account the length and width of the cracks. Figure 3b presents the cracking slab test procedure.

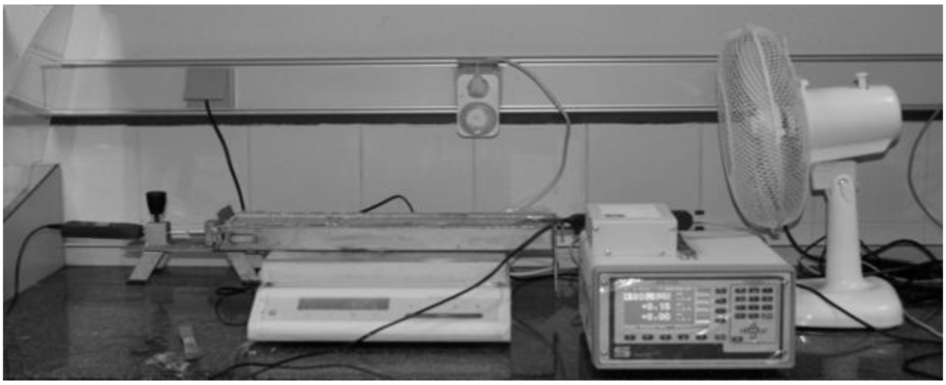

Figure 2: Free shrinkage test setup (air flow speed 3m/s).

Figure 3a: Kraai Slab Test

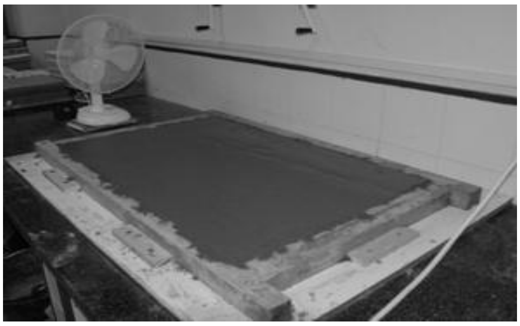

Figure 3b Kraai Slab test

3. EXPERIMENTAL RESULTS AND ANALYSIS

The mixtures of this study, with and without fibers, were tested at early ages and in the hardened state.

3.1 Hardened performance and mechanical characterization

Compressive and flexural strength were tested on paste samples of the concrete compositions without aggregates (40 x 40 x 160 mm specimens). Figure 4 shows the compressive and flexural strength experimental results at 1, 7 and 28 days. It can be observed that the flexural strength measure at 1 day of the compositions with nanosilica and CNF were higher than the SCC composition with a conventional HRWRA. At 28 days, the flexural strength results of the composition with CNF was a 30% higher than the SCC compositions.

When comparing the compressive strength of SCC compositions with limestone filler, it can be observed that the composition with the HRWRA that includes nanosilica showed higher values and that at 7 days reached 90% of the compressive strength at 28 days. The evolution of the compressive strength of the composition with CNF was slower but, at 28 days, the compressive strength was slightly higher when CNF was used.

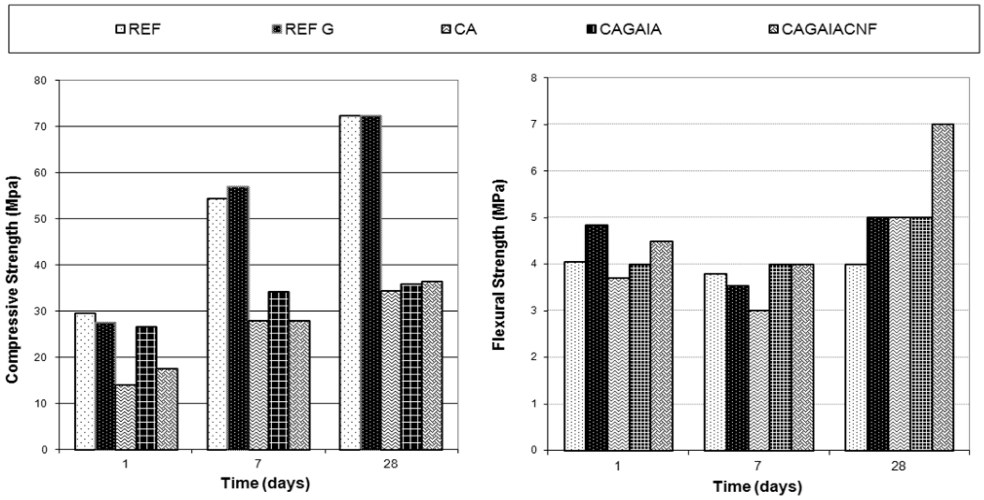

Figure 4: Evolution of compressive and flexural strength of paste samples (compositions of this study without aggregates).

The compressive strength of concrete samples was tested using 100 x 100 x 100 mm specimens, at 1, 7 and 28 days. The experimental results obtained are shown in Figure 5. It can be observed that the compositions with limestone filler presented strength values lower than the reference mixtures, due to the substitution of 50 % of the cement in the compositions. The compositions which included the HRWRA with nanosilica (GAIA) showed compressive strength values at 1 day lower than those compositions with the conventional HRWRA. Although the difference was reduced at 28 days. Mixtures containing CNF had similar compressive strengths compared to the SCC-mixtures. The addition of PPF did not affect the compressive strength of the compositions [19].

A slight delay on the evolution of the compressive strength was observed for the compositions with limestone filler.

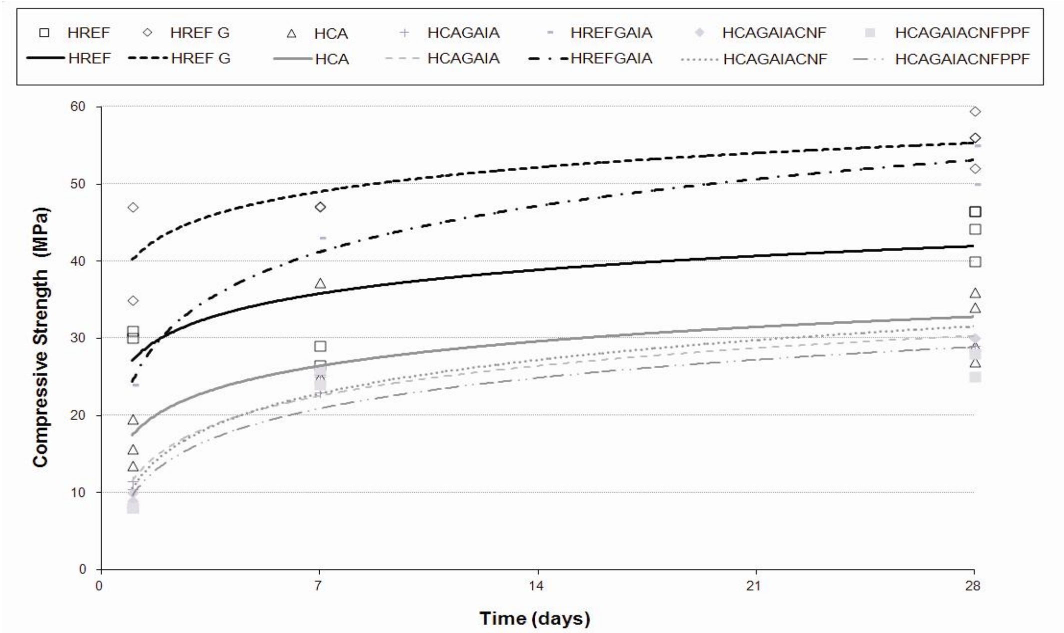

Figure 5: Concrete compressive strength

3.2 Early age performance and microstructural analysis

3.2.1 Early age cracking

Double restrained slab specimens were tested of all the compositions of this study. The cracks were measured at 7 days (the slabs were demolded at 24 hours). The slabs were divided into quadrants in order to facilitate the readings and the cracks were measured and ink marked. The Kraai slab surface of the concrete composition with CNF (HCA-Gaia1%- CNF0,5%) can be seen in Figure 6.

Figure 6. Kraai slab.

Figure 7 summarizes the maximum crack length and the cracked area (in % with regard to the sample that showed the maximum cracked area) of the compositions under study. The cracked of the reference composition was very small, while the SCC compositions had larger crack length and cracked area. This fact can be related to the influence of the larger transition zone (due to the larger amount of fine inert particles) in the cracking and fracture generation. [6-7]

The addition of CNF did not control concrete cracking at early ages. The SCC composition with the conventional HRWRA (HCA) had the largest cracked area, with a value of 186.1 mm^2/m^2, and was considered as 100%. According to this value, the SCC composition with CNF (HCAGAIACNF) only reduced around 15 % of cracked area. According to this experimental result, it can be concluded that, for the compositions under study, the use of CNF alone is not an effective measure for early age cracking control. Moreover, the addition of CNF increased the cracked area 80 % with regard to the composition without CNF.

The addition of a low amount of PPF (0.1 % of volumetric fraction, as PP density was 0.9 g/cm^3) to the composition with CNF eliminated cracking at early ages.

Figure 7 also presents the maximum cracking length measured on the tested slabs, taking again the maximum crack length of the conventional SCC mixture (HCA) as 100%. It can be observed that although HCA and HCAGAIACNF had similar values of cracked area (100% and 85.68%, respectively), the cracks appear in different ways. In the first case (HCA) the cracks were longer and less, while in the second case there were more cracks, they are shorter and have a smaller width (100% and 15%).

As a result of the cracking measurements, it can be pointed out that, although the cracking phenomena can be considered to begin at a nano-structural level, the fiber ability of cracking control was achieved at a microstructural level. One possible reason can be found in the stiffness ratio between the fiber and the concrete matrix at early ages, as CNF are stiffer than PPF [3, 10].

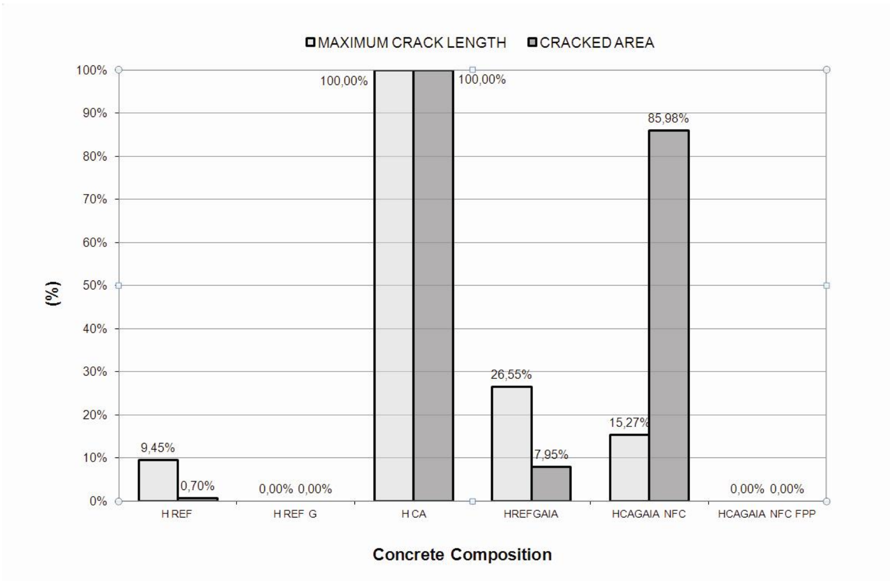

Figure 7: Cracked area and maximum crack length (Kraai slab modified test).

3.2.2 UPV, temperature and shrinkage

In order to better understand the phenomena involved in early age cracking, the evolution of several parameters were monitored during the first 24 hours. Ultrasonic pulse velocity (UPV), temperature profile and free shrinkage were measured and the experimental results are summarised in Figure 8. The UPV results of the sample showed a direct relationship with the increasing temperature of the same sample. Once the minimum temperature was registered, the wave velocity also increased, with different values for each composition under study.

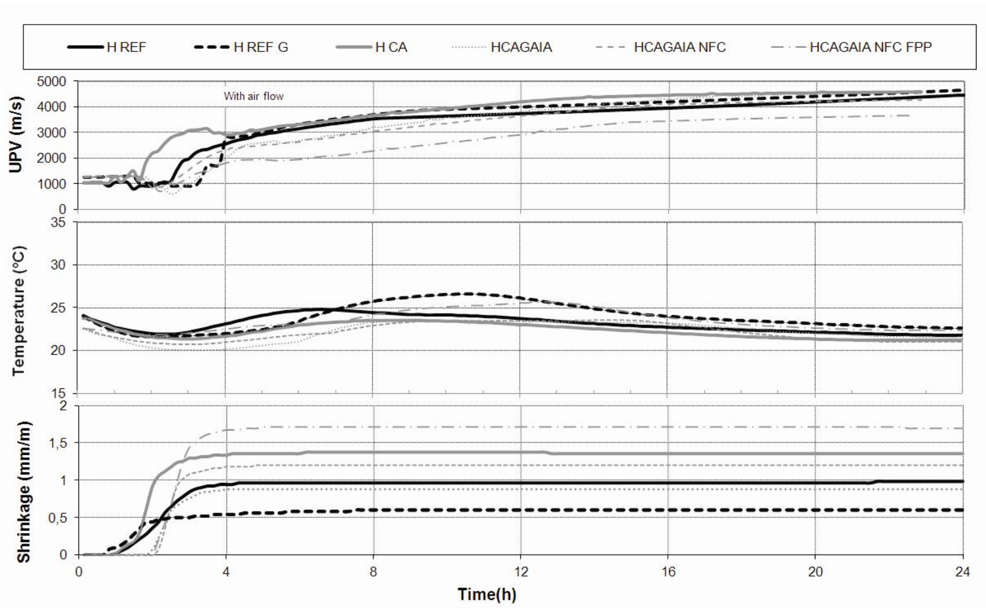

Figure 8: Ultrasonic pulse velocity, temperature, shrinkage.

In a first stage, UPV had unstable values between 1000 and 1400 m/s, during the first 2-3 hours. Then the speed increased dramatically during the next 2 hours At last, the UPV increased at a sustainable rate, reaching stable values at around 24 hours.

It can be observed that the reference compositions with HRWRA present a longer dormant period. This effect is directly related to the action of the additive. The only case when the UPV began to change more quickly is the one with 50% of limestone filler, which coincides with the highest minimum temperature for all dosages during this same period [20].

Free shrinkage occurred between 1 and 2 hours before reaching the minimum temperature and coincided with the initial changes in the UPV measurement. Shrinkage began long before the occurrence of the phenomena that determine a change in the pseudo rigid structure of the mixture (UPV strong increase). Maximum shrinkage was reached before 3 hours in all the compositions under study. The largest shrinkage values were recorded for the mixture that contain CNF and PPF.

3.2.3 Reaction degree

A reaction degree (R_d) was calculated from the temperature profile of the sample during the first 24 hours [21]. The reaction degree is proportional to the integration of the temperature difference between the sample and the environment, considering the fraction of heat released at each moment with regard to the total heat released during the first 24 hours. This parameter allows a comparison among the different compositions considered, relating events to reaction evolution instead of time, as the compositions evolve in a different way with regard to time. Figure 9 presents the Rd values for the compositions considered in this study.

When R_d is compared to free shrinkage it was observed that the SCC compositions with limestone filler, CNF and NS presented larger R_d when the highest values of shrinkage were recorded. This mixtures began to shrink around 3 hours after the casting, coinciding with the values of R_d of 0.1. All important events occur when the degree of reaction is around 0.2. It can be concluded that the critical moment (CM) occurs during this short period of time.

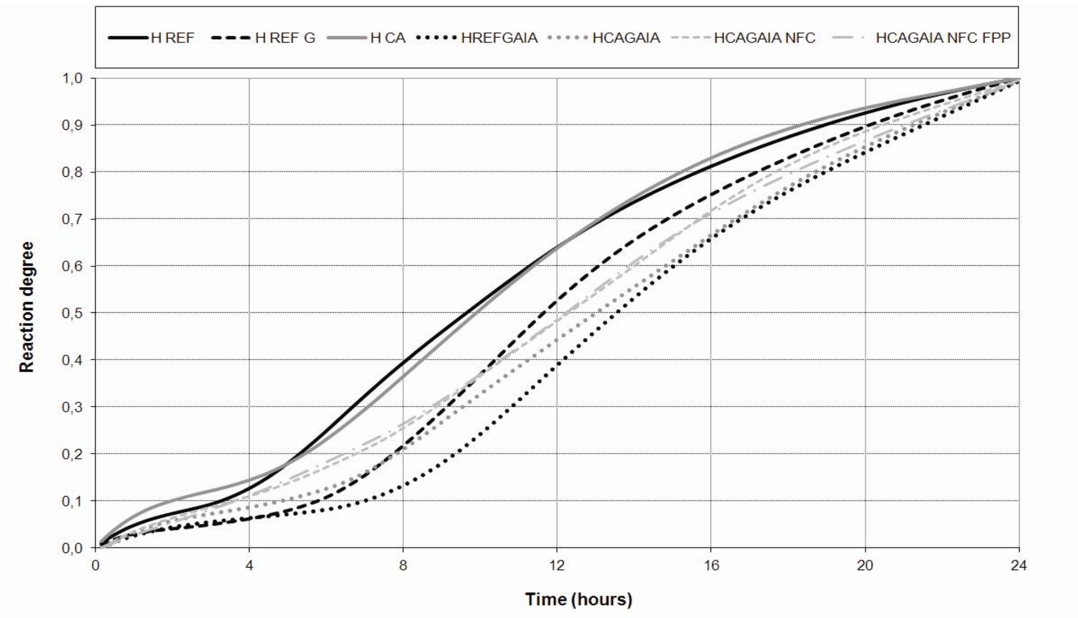

Figure 9: Reaction degree

3.2.4 Scanning electronic microscopy

To asses the distribution of the CNF in the concrete matrix, a scanning electronic microscopy (SEM) analysis of SCC samples was done. Figure 10 shows three images obtained with SEM of a SCC with a HRWRA with nanosilica (a), a reference mixture with a HRWRA with nanosilica and CNF (b) and a SCC mixture with CNF (c). The image (a) of Figure 10, shows a good distribution of the NS on the sample, producing a homogeneous concrete matrix. In images (b) and (c) of Figure 10 the CNF are clearly visible. CNF are homogeneously distributed and connected to the cement paste matrix, acting as bridges through the micropores and microcracks.

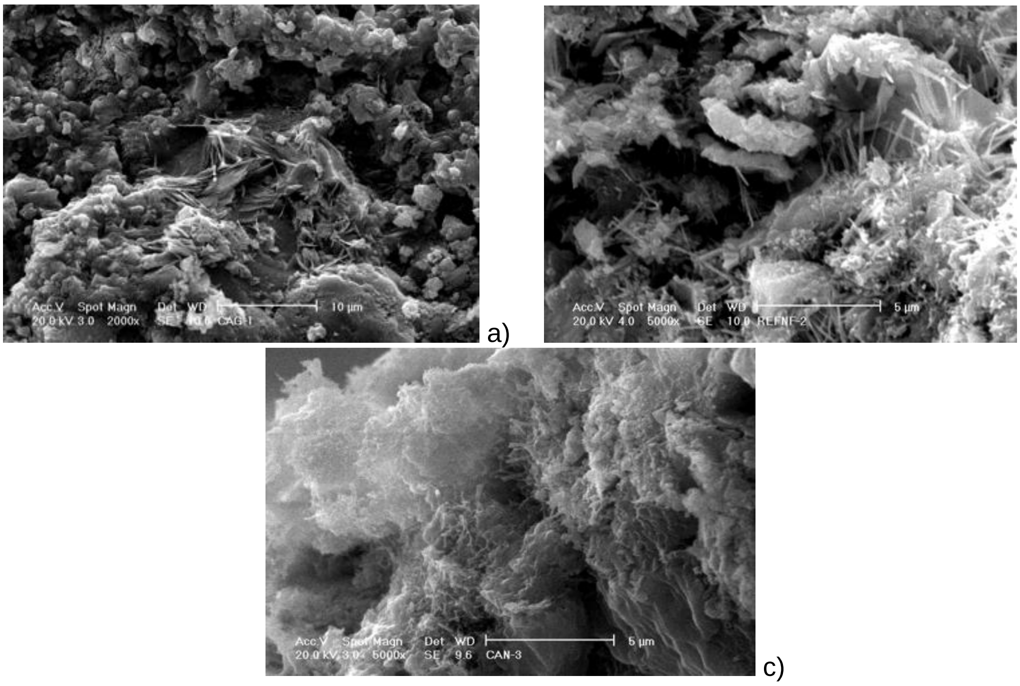

Figure 10: SEM a) CAGAIA; b) REFGAIA CNF ;c) CAGAIA CNF

4. CONCLUSIONS

An experimental study on the effects of carbon nano fibers (CNF) during early ages and in the hardened state of SCC was carried out. According to the experimental results, the use of CNF can improve the mechanical performance of SCC in the hardened state. For the compositions under study, the flexural strength can increase up to 30 %.

The addition of CNF did not ensure cracking control at early ages. For the compositions analyzed, the use of CNF produced larger cracked area. The particle size affecting the relationship of fines in the mix, caused an increase in the amount of paste in dosage, facilitating the appearance of cracks. The use of low amounts (0.1% volumetric fractions) of polypropylene microfibers can solve the cracking problem.

The phenomenon of early age drying cracking can be related to early age shrinkage. However, larger shrinkage values did not imply directly larger cracking. For the SCC compositions under study, it was found that the moment is more important in which a phenomenon that activates the cracking occurs than the magnitude of the phenomenon itself.

A combination of several parameters was used to generate a map of early age concrete features, able to identify the development of the microstructure of cementitious materials and to relate those parameters simultaneously in SCC.

Three stages of the UPV evolution were defined for the SCC compositions under study (although they were very fluid concretes) as the solid network developed. They start from a suspension system, then a solid phase to the complete consolidation of the material. The experimental data produced an identification profile of the evolution of the composition features.

Early age cracking of the SCC compositions under study responded to a multivariate model where the phenomenon depends on the simultaneity of several variables during the stiffening process. Thus, it can range from no impairment to maximum cracking of the material.

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