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Early age monitoring of self-compacting concrete with mineral additions

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HIGHLIGHTS

- SCC with limestone filler and several active mineral additions were investigated at early ages.
- Temperature, UPV, capillary pressure, drying shrinkage and cracking were monitored for 24 h.
- Mineral additions brought forward the microstructure changes regarding reaction.
- Mineral additions on SCC increased cracking risks at early ages.
- Largest cracking risk if fast UPV increase occurred after drying shrinkage stabilized.

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ABSTRACT

An experimental program on SCC at early age (EA), combining limestone filler with several active mineral additions (AMA), metakaolin (MC), microsilica (MS) and nanosilica (NS), was carried out to assess the temporal relations among hydration, microstructure changes and drying shrinkage and their influence on early age cracking risks. The relationships between cement reaction and microstructural changes were investigated by monitoring temperature and ultrasonic pulse velocity (UPV). The risk of early age cracking due to drying shrinkage was evaluated, monitoring water evaporation, capillary pressure and shrinkage and measuring cracking on double restrained slabs subjected to an air flow of 3 m/s during the first 6 h. The reaction process, the microstructural evolution and the physical effects of water displacement and evaporation could be followed. For a better comparison among the different compositions, the measured early age parameters were related to a reaction index ($I_{r,24}$), defined as the fraction of heat produced (accumulated plus released) with regard to the total heat at 24 h. Several relationships among the EA parameters were identified. Cracking risk due to drying shrinkage at EA increased when some events related to the early age parameters occurred simultaneously.

1. Introduction

Self-compacting concrete (SCC) can be considered an energy efficient material; because it reduces on-site working, it does not require any compacting energy and it includes large amounts of low embodied energy components, as mineral additions (MA). SCC compositions are typically characterized by containing a large amount of fine particles, further than the cement used in conventional concretes. The combination of fillers with low amounts of active mineral additions (AMA) is an innovative approach to improve SCC mechanical hardened performance without a significant increase of cement content [1]. The use of fillers produces changes in the material performance both the fresh state behavior, as mineral additions modify the workability and rheology, the setting process and the hardened microstructure [2]. These differences do not only modify the material performance but can also compromise durability [3–4]. Among the variables that can affect the cement hydration, the nature and particle size of MA can be highlighted [2,5–10].

Most changes in the microstructure occur during the hydration process, during the first 12–24 h. This period of time is usually known as early age (EA), which comprises the evolution from a liquid dispersion/solution with a plastic behavior (fresh state) into a pseudo-rigid solid (hardened state). Early age (Fig. 1) is nowadays considered as a key point of the cement based materials evolution, because it enables the material performance in the hardened state and the long term [11].

A proper characterization of cement based materials at early ages requires experimental techniques different to the ones commonly used to evaluate fresh or hardened concrete. The simultaneous

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monitoring of several parameters, further than just determining setting time, has been proposed to assess the material evolution during early ages [1,12–13].

During EA, several main microstructural changes occur due to cement hydration (Fig. 2). The hydration products crystallize on the surface of the solid particles in contact with water, bridging the solid particles (grain connection) and filling the gaps among them (pore filling), previously occupied by unreacted water [14]. The water movements create capillary channels, through which the water can bleed to the upper surface due to the hydrostatic pressure and the weight of the solid particles. During cement hydration, the amount of free water is reduced and, therefore, the hydrostatic pressure is also reduced [15]. Besides, the solid grains begin to get interconnected, trespassing their own weight to the incipient solid frame and the capillary net consolidates. The previously bled water is then reabsorbed and available for cement hydration. As the water available gets consumed and the surface of the solid particles gets saturated, the reaction decelerates and the material cools down.

Therefore, the microstructural changes are related to the reaction development and are mainly governed by the presence of water and water movements inside the material. The hydration process is usually studied monitoring calorimetric parameters, as temperature or heat released or accumulated by a sample [2,5,12,16], because cement hydration is an exothermic reaction. However, calorimetric techniques, as isothermal and adiabatic calorimetry, modify the temperature of the samples and can distort the experimental results of the hydration process [1]. The microstructure development can be monitored by ultrasonic pulse velocity (UPV) through samples under defined environmental conditions [1,12,14,17]. The combination of both parameters allows the identification of the main changes of the material during early ages.

The environmental conditions greatly affect the water displacements and evaporation during EA and affect the microstructure evolution and the prospective hardened performance. Temperature can modify the speed of the reaction and, jointly with relative humidity and wind velocity, can force water evaporation and drying shrinkage. The typical consequence of the environmental influence on early age concrete performance, further than reaction speed, is cracking due to drying shrinkage [18–20].

Cracking happens when a concrete member simultaneously has its displacement restrained, suffers a dimensional change, has a certain stiffness and the (tensional) stress overcomes the mechanical capacity of the member. In the case of early age cracking due to drying shrinkage, the dimensional change is related to water evaporation [20] and the formation of menisci inside the (pseudo-solid) pore structure and the stiffness and mechanical capacity only develops when a certain degree of interconnected (pseudo-rigid) microstructure is achieved. Early age stiffness has been described to be related to a mechanism of grain interconnection while strength development depends on pore filling by hydration products [14]. Therefore, cracking due to early age drying shrinkage is linked to reaction development, microstructure evolution and environmental effects.

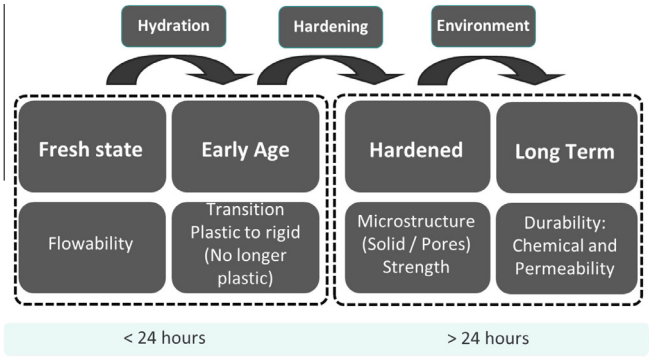

Fig. 1. Cement based materials evolution.

As far as shrinkage depends on paste hydration and water evaporation, SCC is especially susceptible to crack at early age, because SCC usually has a larger amount of paste and water than any conventional concrete. The use of admixtures can also modify EA behavior [21]. However, SCC has been described to show similar cracking risks than conventional concretes at early ages [3]. MA have been reported to modify the material evolution at early ages through two main mechanisms: facilitating the nucleation of hydrated cement products, as fillers and non-active MA [2,5,8], and reacting with portlandite due to pozzolanic activity, as AMA [1], producing calcium silicate hydrates (CSH) [6,9,22] and aluminates [7]. In any case, early age cracking of SCC is still an issue to be considered, as far as it can compromise durability because it would enlarge permeability [23].

2. Experimental program

An experimental program on SCC with several AMA was carried out. Limestone filler (CA), microsilica (MS), nanosilica (NS) and metakaolin (MC) were considered. The aim of the study was to assess the influence of SCC composition at early ages. The hydration process, the microstructure evolution, the heat and water interchanges with the environment, the capillary pressure and the free drying shrinkage were simultaneously monitored. The cracking risks linked to early age drying shrinkage were also considered.

2.1. Materials and mix proportions

Six concrete compositions were tested, which are summarized in Table 1. A reference concrete, without a high range water reducing admixture (HRWRA) and a dry consistency, was considered (named HREF). This reference composition was modified including a HRWRA, Glenium® ACE 425 manufactured by BASF, and achieving a self-compacting ability (HREFG). Afterwards, 50% of the cement was substituted by limestone filler, Betocarb® P1-DA (85 ± 5% under 63 μ), supplied by Omya Clariana SL, producing a conventional SCC with filler (HCA). Three active mineral additions (AMA) were added to this composition, substituting 5–10% of the limestone filler: a densified microsilica (MS), Meyco MS 610, and an amorphous nanosilica suspension (NS), Meyco MS 685, both supplied by BASF Construction Chemicals España S.L., and a metakaolin (MK), Optipozz™ (average diameter 1.35 μ), manufactured by Burgess Pigment Company and supplied by Omya Clariana S.L. Accordingly, the SCC compositions were labeled HCAMS, HCANS and HCAMC. The water to fines ratio (w/f) remained at 0.36 for all the compositions and the amount of paste was constant in all cases. The nominal chemical compositions of the cement, the filler and the MA have been previously described [1].

Table 1 also records the slump of HREF composition (dry consistency) and the average J-ring spread flow diameter (djf) and the blocking coefficient (CbE) of the SCC compositions, measured according to EN 10250. No segregation or bleeding was observed on the fresh SCC samples.

2.2. Early age measurements

The internal temperature and p-wave ultrasonic pulse velocity (UPV) was monitored on 150 × 100 × 70 mm concrete samples at laboratory conditions (20 ± 2 °C, 50 ± 10% relative humidity) during 24 h. The samples were cast in a plastic mold and the upper surface of the samples was exposed to the ambient. Although the direct temperature measurement is less accurate than other techniques (isothermal and semi-adiabatic calorimetry), this procedure does not modify the thermal profile of the samples, which could alter the reaction speed [2,17]. Capillary pressure was also monitored with pressure measurement devices connected to a computer. Four capillary pressure devices were placed in each sample: two at 15 mm and other two at 35 mm depth. The results obtained did not vary with the depths considered, and an average value was considered for comparison.

A free shrinkage apparatus with internal dimensions of 500 × 100 × 50 mm, subjected to an air flow of 3 m/s during 6 h, was used to simultaneously monitor free drying shrinkage and evaporation. The evaporation rate was calculated, measuring the mass loss of the samples by placing the shrinkage apparatus on a continuous recording scale.

Early age cracking risk due to drying shrinkage was assessed on two types of slabs: 400 × 400 × 40 mm and 600 × 900 × 50 mm modified Kraai slab test [3]. U-shaped steel pieces were attached inside the molds in order to achieve a double displacement restraint. The slabs were subjected to an air flow of 3 m/s to force cracking due to drying shrinkage. The experimental setup of shrinkage, evaporation and cracking tests have been previously described [1].