EXPERIMENTAL STUDIES OF SELF-HEALING CEMENTITIOUS MATERIALS INCORPORATING MINERAL ADMIXTURES

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ABSTRACT

In order to limit the release of CO_2 produced in cement manufacturing, clinker (the major cement component) is often partially replaced by mineral admixtures like blast-furnace slag. The use of mineral admixtures presents different advantages such as the recycling of industrial waste and the improvement of different material properties (workability, sulfate resistance, compressive strength at long term…). Nevertheless, several civil engineering constructions made with blast-furnace slag cement presented cracking at early age due to restrained shrinkage (autogeneous, thermal and drying shrinkage) that affects significantly the material transfer properties and their durability in aggressive environments (chloride, CO_2). Under certain conditions however, it is known that cementitious materials present a self-healing capacity. The purpose of this research work is to study the beneficial influence of the natural self-healing capacity of mortars with blast-furnace slag cement on the lifetime of civil engineering constructions.
A new experimental device has been designed in order to understand the self-healing capacity of mortars at early age. The novel apparatus allows developing micro-cracks in a mortar ring specimen by restrained shrinkage and monitoring the evolution of self-healing by gas permeability and effective diffusivity measurements. The effects of different slag contents (0%, 50%) and curing conditions (air conditioned room at 25±1°C and 45±5% R.H., in tap water) on the self-healing kinetics are studied. Optical measurements in 2D (optical microscope, SEM) and 3D (tomography), chemical analyses of the self-healing products and quantification of the properties of cement with blast-furnace slag (compression and tensile strength, autogenous and drying shrinkage, heat release) complete the study.

1. INTRODUCTION

Modern cementitious materials can nowadays be designed with improved properties (strength, durability…) and lesser environmental negative effects. For example clinker, whose industrial production releases important amounts of CO_2, is sometimes partially replaced with mineral admixtures like blast-furnace slag. But some constructions realized with this new material showed cracking at early age reducing their durability performance. The self-healing capacity of these materials (by activation of the hydration of the anhydrous cement close to crack) could act as a counterpart. The latent hydraulic properties of slag could present interest to improve the self-healing phenomenon.

2. MATERIALS AND METHODS

In this work, two mortar compositions are used: the first using a Portland cement CEMI 52.5 N CP2 NF and the second where 50% of the cement is substituted by blast-furnace slag. The two compositions are defined hereafter as CEMI and 50%BFS and are characterized by a Water/Binder (W/B) ratio equal to 0.50 and 0.52 respectively (the quantity of binder is the same for the two compositions). The sand used has a normalized size.

Two methods are adopted to follow the kinetics of the self-healing phenomenon at early age. The first consists in monitoring the decrease in the cracking size of the mortar samples (diameter Ø = 4cm and height h = 10cm) by means of tomography. A crack characterized by a width equal to about 100 µm was obtained on seven days old specimens by using a splitting test. Specimens were confined with a bi-composant resin reinforced with a glass fiber band to avoid breaking in two parts (method proposed by Van Tittelboom et al., [1]). The analysis by tomography was performed at different ages. Between the different steps, the samples were stored in individual boxes filled with tap water.
The second method consists in following the self-healing phenomenon at early age on a crack due to restrained shrinkage [2] using an optical microscope (measuring the crack width) and performing gas permeability tests. Cylindrical specimens (Ø = 15cm and h = 5cm) were tested and their deformations were restricted by a brass cylinder core located at their center. A crack appeared after about 10 days (~100µm) and was detected by Digital Image Correlation. The samples were submitted to different curing conditions (kept in tap water and in an air-conditioned room at 25±1°C and 45±5% R.H). The self-healing phenomenon was monitored at 7, 14, 21 and 28 days after the crack formation. Before the gas permeability tests, the samples stocked in tap water were dried during 24 h at 35°C to allow the gas flow through the crack.
Moreover, and in parallel of the above study, the compression and tensile strengths, the autogenous and drying shrinkage of the two mortar compositions were characterized on prismatic samples (4cmx4cmx16cm). Semi-adiabatic calorimetry tests were also realized in order to assess the hydration rate.

3. RESULTS

The tomography allows observing in 3D the evolution of the crack characteristics (density, length, opening, percolation…, see Figure 1), for a crack larger than the voxel size (here about 20 µm). To quantify the cracking density, a specific program was developed in Matlab. It analyses the gray level of each radiography picture (256 gray levels), the quantity and the size of the voxels and allows determining the empty volume. In figure 1, we can clearly see that the crack and porosity have the same gray levels. To distinguish these two types of voids, the program was adapted to only determine the connected (percolated) voxels with the same gray scale using a "burning" algorithm. An example is presented in figure 2 for the 50%BFS specimen just after cracking and 28 days later. We note the decrease in the crack width. Moreover, the crack does not follow the same pattern as of the previous image. This is evidence of the self-healing phenomenon acting in this sample.

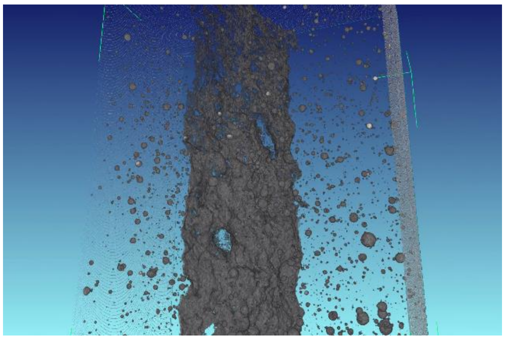

Figure 1: 3D reconstruction by tomography: Crack and porosity

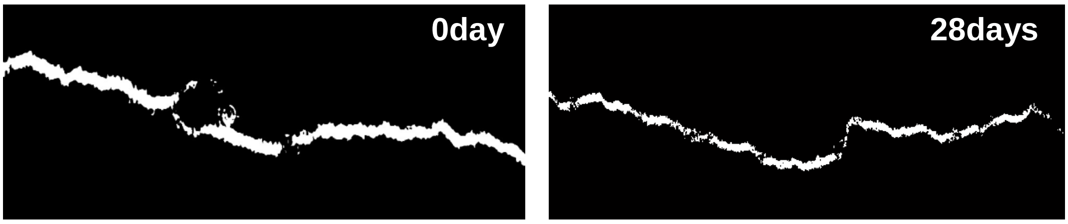

Figure 2: Evolution of the crack size on the 50%BFS sample just after cracking and 28 days later (percolated cluster in 3D)

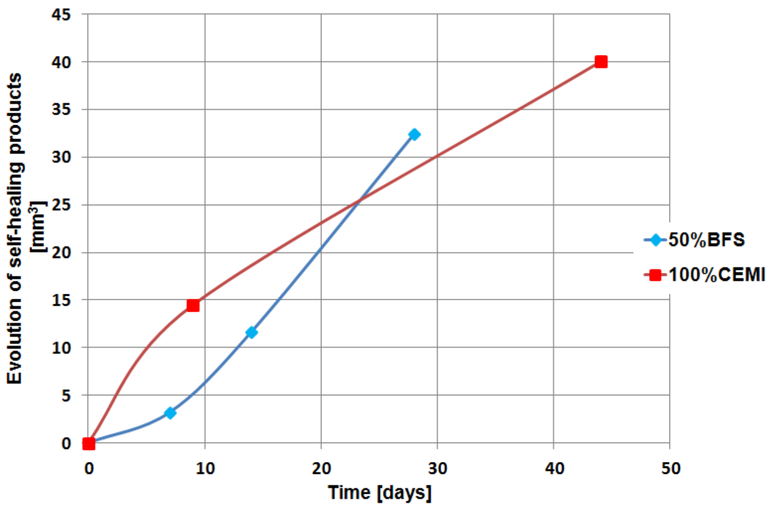

Figure 3: Evolution of self-healing products in the crack (after a splitting test)

In figure 3, the evolution of the self-healing products formed in the crack over several weeks confirms the decrease of crack size. We can also observe a different behavior between the two cementitious materials (CEMI and 50%BFS). At early age, the formation of the self-healing products is three times faster for the CEMI than for the 50%BFS. After 22 days, this behavior changes and the 50%BFS sample seems to have a greater self-healing capacity at long term. This is well correlated with the hydration degree presented in figure 4. At early age, the hydration of the CEMI specimen is faster and reaches a degree close to 0.6 at 7 days. The hydration degree of the 50%BFS specimen is less significant but it increases progressively. This is due to the latent hydraulic behavior of the slag. The 50%BFS sample needs the formation of portlandite to active its hydration. During the first few days, the hydration of the anhydrous cement in the CEMI sample is activated by water contact. The chemical reaction is quick and the crack can self-heal. In the same period, the cement and thus portlandite quantities are less important in the 50%BFS specimen. After this period however, slag hydration is activated and the self-healing capacity is progressively increasing.

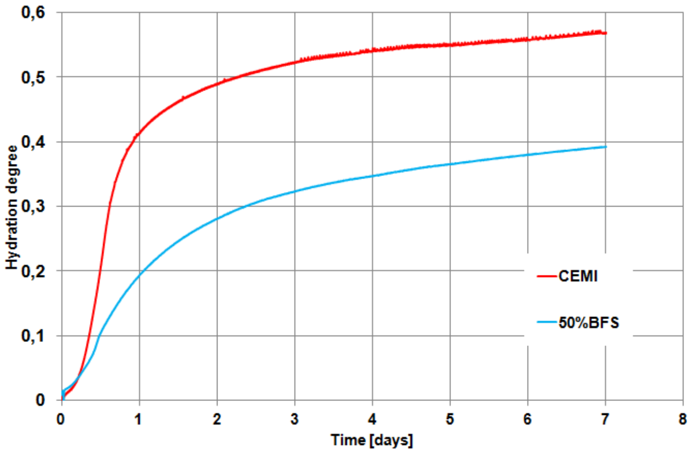

Figure 4: Evolution of the hydration degree by semi-adiabatic calorimetry

4. CONCLUSIONS

Thanks to the use of tomography it was possible to observe and quantify the self-healing capacity of two types of cementitious materials. If a formulation with 50% slag has the tendency to a slower self-healing in an early age than a formulation with 100% portland cement, its latent hydraulic behavior is critical to its self-healing in long term. Future work will be focus on validating this behavior at very long term.

REFERENCES

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