EFFECT OF THE QUENCHING IN WATER OF THE CEMENTITIOUS MATERIALS SUBJECTED TO FIRE

EFFET DE LA RÉ-HUMIDIFICATION SUR LES MATÉRIAUX CIMENTAIRES SOUMIS À UN INCENDIE

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RESUME

De nombreux travaux ont permis d'analyser le comportement mécanique d'un béton soumis aux hautes températures. Toutefois aucune étude n'a été faite sur l'effet de la ré-humidification des matériaux cimentaires exposés à un incendie réel. Cette étude en constitue une première. Afin de mieux simuler les effets d'un incendie réel, un test au feu de bois est mis au point. Les différents matériaux cimentaires (pâtes de ciment durci et mortier) sont soumis à cet échauffement pendant 1h. Les résultats d'essais effectués montrent :

- La perte moyenne de résistance est de l'ordre de 60 % après l'exposition au feu.
- La ré-humidification (refroidissement brusque dans l'eau) permet une récupération de masse de l'ordre de 10% et par conséquent un gain de résistance mécanique en compression d'environ 40%.
- L'analyse de la microstructure interne des pâtes de ciment étudiée par diffraction X et observations au MEB montrent qu'après ré-humidification, le ciment se réhydrate à nouveau (formation de la portlandite et CSH). Cela permet d'expliquer l'évolution des résistances.

MOTS CLES: Matériaux cimentaires, feu de bois, ré-humidification.

ABSTRACT

Many studies have permitted the analysis of the mechanical behavior of concrete subjected to high temperatures. However, no one has done any work on the effect of re-humidification of cementitious materials exposed to a real fire. This is the first study. To better simulate the effects of a real fire, a test with wood fire is developed. Different cementitious materials (hardened cement pastes, and mortar) are subject to this heating for 1hour. The results of tests indicate that:

- The average resistance loss is in the order of 60% after exhibition in fire.
- The re-humidification (sudden quenching in water) allowed a recovery of mass about 10% and consequently a gain of compressive strength about 40%.
- Internal micro-structure analysis of cement pastes studied by XRD and the observation by a scanning electron microscope (SEM) shows that after the re-humidification, the cement rehydrates again (formation of Portlandite and CSH). Those observations explain the increase mechanical strength.

KEYWORDS: Cementitious materials, wood fire, quenching in water.

1 INTRODUCTION

The main aim of this study is to test the influence of sudden immersion in the water of cementitious materials exposed to fire.

During exposure to high temperatures, a cementitious material is subjected to a greater or lesser damage. Heating induces different changes of its properties and, in particular, changes in microstructure accompanied by loss of mechanical strength (1) and (2).

2 EXPERIMENTAL PART

2.1 Realization of the test body

Mortar samples 4x4x16cm^3 and cement pastes with a standard consistency are being studied in this experimental work. The mortar be carried out in accordance with the standard EN 196-1 (standard sand, water/cement = 0.50, cement/sand = 1/3). The cement pastes be carried out in accordance with the standard EN 196-3 with water/cement ratio = 0.25. The samples were demoulded after 24h, and cured in water at 20°C for 30 days. Then, they are kept in the air-conditioned chamber (20°C, 50% RH) until their mass was stabilized. Then, they are exposed to wood fire for 1hour. This choice is guided by fire conditions and emergency intervention.

The CEM II/B 42.5 NA 442 (Lafarge-Algeria) cement is used. Its mineralogical composition is presented in Table 1.

Table1: Mineralogical composition of CEM II/B cement

C_3S	C_2S	C_3A	C_4AF
54,08	22,33	4,44	15

2.2 Heating condition

To simulate a real fire, we proceeded to fabricating a device which ensures a homogeneous distribution of fire on all samples of cement pastes and mortar (see Fig. 1). The device in the form of a cage is composed of high temperatures resistant steel rods. It consists of three superimposed rooms separated by grids to facilitate the dissemination of heat through the test body. The device is instrumented with thermocouples.

The body's tests are covered by wood. The fire is lit on a bed of gasoil (see Figs. 2 and 3). The wood is added in a regular way to maintain the fire for one hour. This choice is guided by fire conditions and emergency intervention. The test is conducted in the open-air in spring weather at a temperature of 23°C and a moderate wind from the northeast.

The flame and the surface of the body's test temperature is recorded every 10 min. At the end of the test, the flame has reached 700°C and 410°C for the surface of the test body. The fire is allowed to turn in air.

The fire resulted the bursting of test body (Fig. 4). However, four specimens and two pastes have kept their good form and are subject to two types of cooling before to be tested: cooling at ambient air and sudden quenching in water.

The bursting of test body is explained by rapid heating of its surfaces. Rapid heating causes thermal stresses, from the high thermal gradients that create compressive stresses and tension in the structure. Khoury (3) considers that these constraints have a key role to play in instabilities due to the geometry of the material. Kanema Tshimanga (4) shown that the phenomenon of bursting happens for surface temperature between 300 and 350°C (in our case 410°C).

Figure 1: The arrangement of the test body

Figure 2: Test body covered with wood

Figure 3: Test fire

Figure 4: Burst test bodies

2.3 Physical analysis

Figure 5 shows the evolution of the masses of mortar specimens for both cooling methods: cooling at ambient air

and sudden quenching in water. The masses of burned mortar were compared to the mass of the witness mortar (30 days of curing in water). The specimens are weighed until stabilization of the masses.

On the one hand, we observe:

- The burned mortar has undergoes strong mass loss (16%) resulting from the departure of the free water and bound water.
- The burned mortar and cooled in water has regained 10% its lost mass. This can be explained by re-saturation of specimens and probably a new rehydration of cement components.

On the other hand, the burned mortar and cooled at ambient air is characterized by very low weight gain (4%). It can be explained by a water uptake in the pores due to moisture from the air.

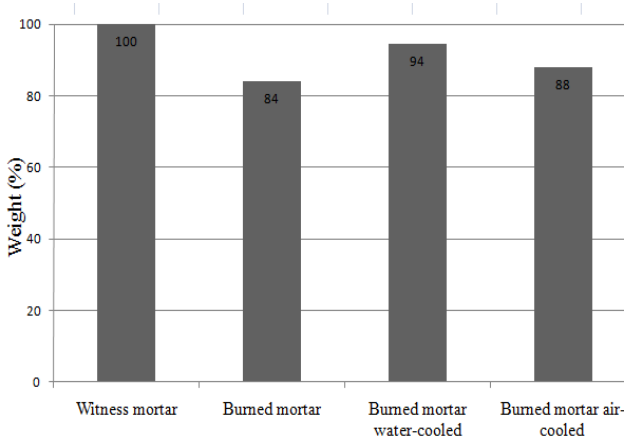

Figure 5: Evolution of mortar mass

2.4 Mechanical tests

Mechanical tests were carried out with a loading rate of 0.5mm/min for the 3-point bending and 0.25mm/min for the compression.

Figure 6 shows the average of residual and relative strengths (normalized to resistance of the witness mortar unheated) to the 3-point bending obtained for the different types of mortars. Whatever the mode of cooling of burned mortar, flexural strengths show a significant drop: 70% for the water-cooled mortar and 90% for the air-cooled mortar.

Figure 7 shows the average of residual and relative compressive strength obtained for the different types of mortars. The strength of air-cooled burned mortar dropped significantly (about 60%), while that the burned mortar cooled in water dropped by about 20%.

These resistances are consistent with the evolution of mortars mass. This confirms that the re-humidification in water allows a new hydration of cement components. However; this rehydration has no appreciable effect on the flexural strength.

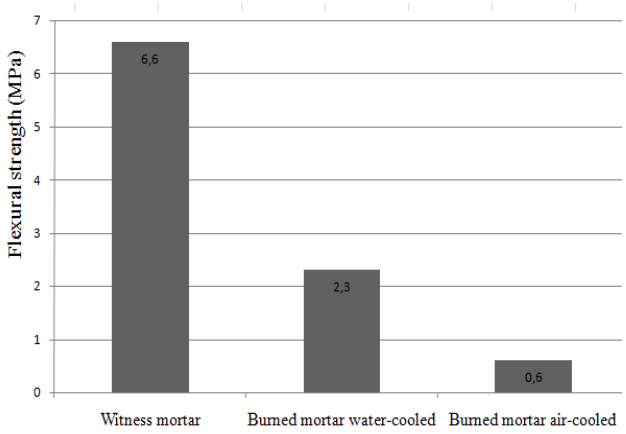

Figure6a : Flexural strength

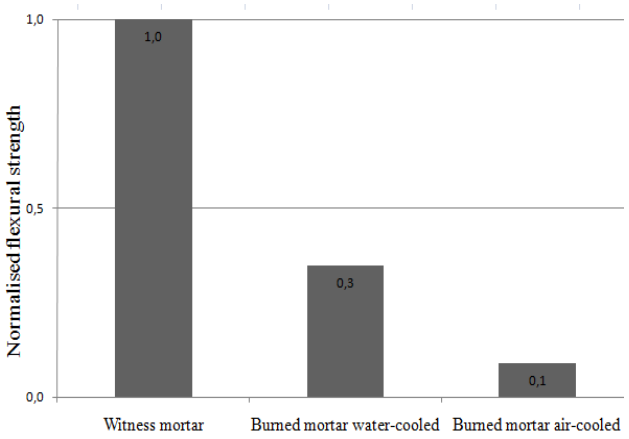

Figure 6b: Flexural strength: values normalized to the strength of witness mortar

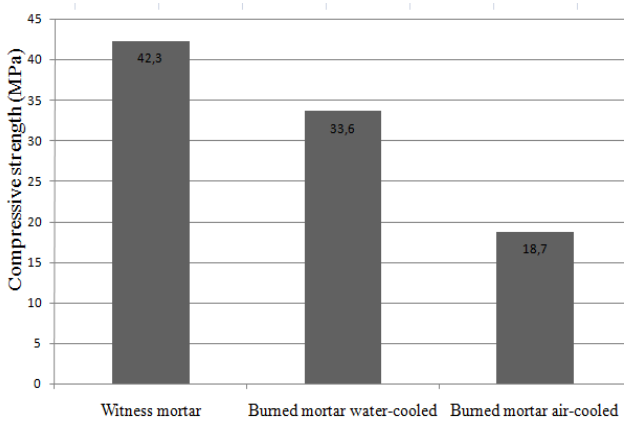

Figure 7a: Compressive strength

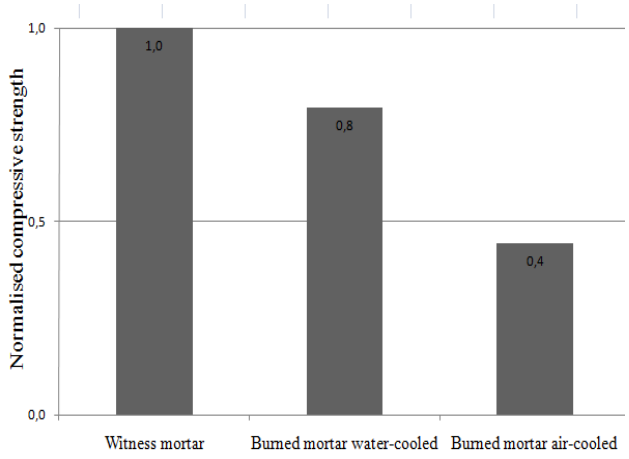

Figure 7b: Compressive strength: values normalized to the strength of witness mortar

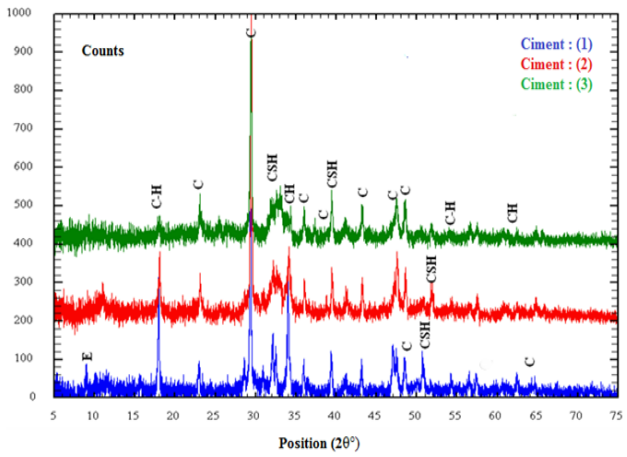

Figure 8: X-ray diffraction of cement pastes 1, 2 and 3

2.5 X-Ray diffraction

X-ray diffraction was performed on cement paste powder passing all in sieve of 50 microns. The XRD data were collected on a Philips PW 3710 X-ray diffractometer with Bragg–Brentano geometry using Ni-filtered Cu Kα radiation, operating at a voltage of 30 kV and a current of 20 mA. The step-scan covered the 2θ -range 5–65° with a step-length of 0.02°.

In the diagrams of figure 8, cements 1, 2 and 3 represent, respectively, the cement pastes: witness, burned water-cooled and burned air-cooled.

The mineralogical composition of the witness cement (cement 1) is constituted by the products of hydration, namely: Calcium-Silicate-Hydrate (C-S-H), Portlandite (CH) and Ettringite (E). The diagram also shows the diffraction of Calcite due to the presence of limestone in the cement.

A crude approach of the quantity of hydrate may be carried out by comparing the intensities of the diffraction peaks. Portlandite (CH) and Ettringite (E) have virtually disappeared for the burned cement (cement 3) and the quantity of CSH is considerably reduced.

Quenching in water from the burned cement paste has resulted in a new hydration of cement (cement 2), In fact, diagram thereto shows a remarkable increase in the amounts of CH and CSH.

2.6 SEM observations

Samples of cement paste are deposited on a brass support by means of a double face carbonaceous and covered with a conductive Or-Pd. SEM observations were conducted on a SEM FED (JEOL-JSM-6301F) using an accelerating voltage of 7kV and a working distance of 15mm.

The internal microstructure of witness cement observed by SEM shows the presence of hydration products such as Portlandite (hexagonal sheets) and CSH gel having a dense structure. Traces of calcite indicate that the cement used is composed of limestone (Fig. 9).

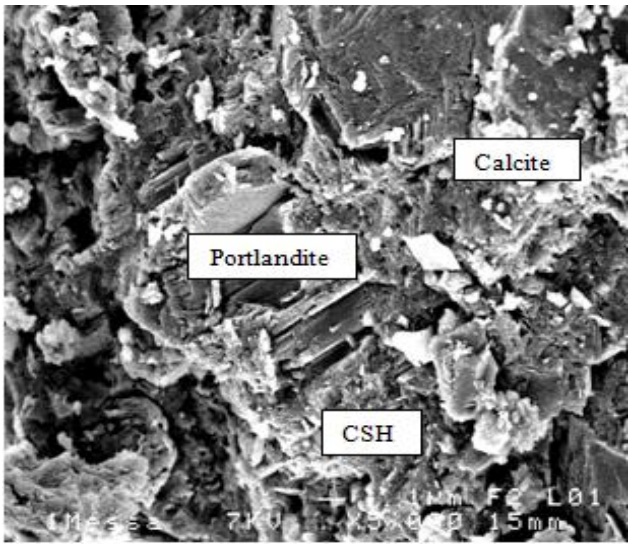

Figure 9: SEM observation of witness cement

After exposure to fire, the structure of the cement paste is porous (see Fig. 10). This is due to the departure of the free water and part of the bound water of CSH (sample temperature was about 410°C) and the majority of the structural water of Ettringite and Portlandite. Schneider (5) and Noumowé (6) describe the major changes in the cement

matrix with a temperature increase as follows:

- Departure of the free water and bound water loss, between 30 and 105°C (7).
- Noumowé (6) considers that free water is completely removed at 120°C.
- Castellote (8) notes that the decomposition of Ettringite and its complete removal from the cement paste intervenes before 100°C.
- From 130 to 170°C, held the double endothermic reaction of decomposition of gypsum $CaSO_4.2H_2O$ (6) and (7).
- The first signs of destruction of CSH are noticed before 100°C and continue up to 300°C (8) and (9).
- From 450 to 550°C, the decomposition of Portlandite. It is done by releasing water (6) and (7).

In fact, the internal microstructure of cement fire and air-cooled shows only the CSH gel which has also lost water and OH, having a dense structure as above with consequences more porosity at its periphery. Traces of calcite were observed indicates the presence of limestone in cement. The limestone was not decomposed because the temperature has not reached 650°C (6).

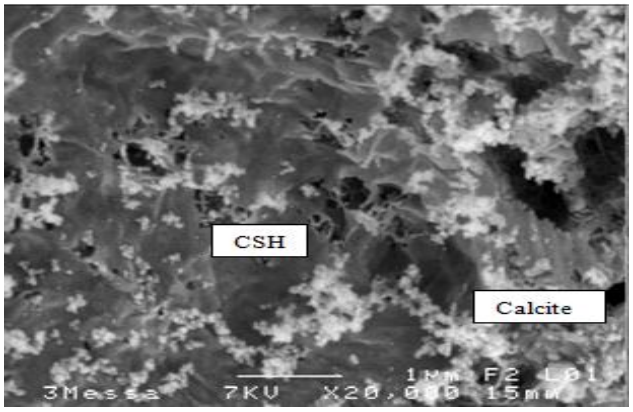

Figure 10: SEM observation of burned cement

Quenching in water of burned cement paste resulted in the formation of new hydrates of cement such as Portlandite and CSH with a denser structure compared to burned cement (see Fig. 11).

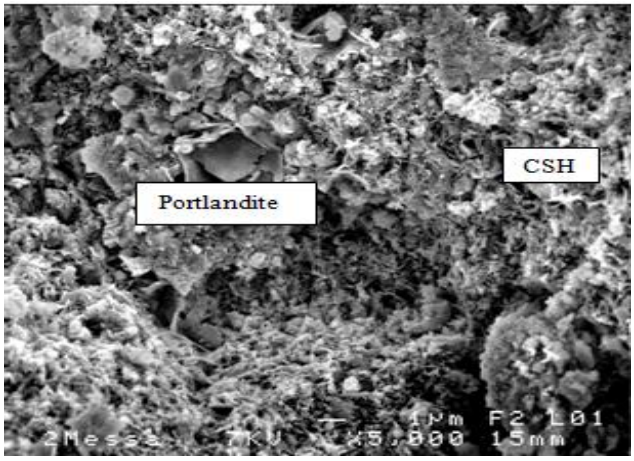

Figure 11: SEM observation of burned cement soaked in water

These SEM observations confirm the results of X-ray diffraction analysis and explain the evolution of compressive strength.

3 CONCLUSIONS

Firstly, it is interesting to note that this experimental study is an original work for two reasons: The first is the technique used to heat cementitious materials (simulating a real fire by wood fire) and the second is the study of the effect of cooling in water.

Among the interesting results of this study:

- The mechanical properties of mortar subjected to fire degrade following the dehydration of the cement matrix.
- The quenching in water allows to burned mortar to recover a portion of its compressive strength (approximately 40%) resulting of a new rehydration of the cementitious matrix.

However, many questions remain and must be answered in future work: what is the influence of soaking in water on the behavior of plain concrete, high performance concrete and innovative concrete ... etc. Subjected to high temperatures?

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