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Research Paper

Physico- mechanical and micostructural effects of water/cement ratio on the mortars upon heating

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ABSTRACT

This experimental work studies the effect of the W/C ratio on the mechanical properties of the mortars subjected to the high temperatures. Fours mortars were made according to EN196-1standard. A typical mortar mix with W/C = 0.5 and three others mortars with W/C= 0.3; W/C= 0.4 and W/C= 0.6. They were exposed to a slow heating in a muffle furnace until the target temperature (100, 200,300,400,500,600,700 and 800°C) with a step of 2°C.min⁻¹ and cooling to room temperature took place in the closed and disconnected oven (approximately -0.3°C/min). After thermal stress, mortars with high W/C ratio (0.5 and 0.6) show a decrease in compressive strength much greater than those with low W/C ratio (0.3 and 0.4). The flexural strength of different mortars has a similar shape to those of the compressive strength. The weight loss and water absorption results are consistent with the mechanical behavior. The SEM tests were carried out on samples of unheated control cement and cements heated to 400°, 500° and 800°C. The SEM observations explain the mechanical strength. At upper temperature (800°C), the material is too much damaged and measurements become difficult.

1 Introduction

The temperature increase has many consequences, in phenomenological term, on the concrete material. Among those negative consequences, we mention drying shrinkage, thermal expansion, creep and mineralogical transformations. During exposure to high temperatures, a cementitious material undergoes a more or less important damage. The heating induces different modifications of concrete properties and, in particular, a microstructural change accompanied by loss in mechanical

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strength and also in concrete weight [1]. These changes are respectively responsible for cracking, spalling and splitting. They highlight the high sensitivity of concrete exposed to high temperatures; mainly due to residual water exists in different forms. Under heat effect, this water evaporates and escapes from the paste and causes dehydration of different hydrates.

Nevertheless, mechanical properties decreased when concretes are subjected to high temperatures. Studies by Gluekler [2], Ali et al. [3], Fu et al. [4], and Peng and Huang [5] reported that various phenomena led to the deterioration of the concretes properties at high temperatures. According to Heikal [6], these damages were engendered by physical, chemical and mechanical phenomena, which may happen at different temperature stages.

Main processing in the cementitious matrix with the temperature increase is described by Schneider et al. and Noumowe [7, 8]. Between 30 and 105°C, departure of the free water and bound water loss [9]. Noumowe [8] considers that free water is completely removed at 120°C. Castello et al. [10] notes that the decomposition of ettringite and its complete removal from the cement paste intervenes before 100°C. Between 130 and 170°C, the thermal decomposition of gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ [8, 9]. The first signs of destruction of CSH are noticed before 100°C and continue up to 300°C [10, 11]. From 450 to 550°C, the decomposition of Portlandite, it is done by releasing water [8, 9, 12].

The cement content and the water/cement ratio are the most influential factor in the durability of the concretes. It is in this sense that we tried in this experimental work to contribute to the comprehension of the effect of water/cement ratio on the mechanical strength, weight loss and absorption coefficient of mortar subjected to high temperature (25-800°C).

2 Experimental Procedure

In this experimental work, $4 \times 4 \times 16 \text{ cm}^3$ mortar specimens are studied. Four mortars were made in according to EN 196-1. Typical mortar mix (standardized sand, $W/C = 0.5$ and $C/S = 1/3$) and three other mortars with respectively $W/C = 0.3$; $W/C = 0.4$ and $W/C = 0.6$. The W/C ratios of 0.3 and 0.4 require the use of a plasticizer to enhance the fluidity. The adjuvant used is MEDAFLUID SFR122, it is a super plasticizer (SP) based polymers combined, provided by the company GRANITEX (Algiers- Algeria). The samples are removed from the molds after 24 hours and stored in water at 20°C for 30 days, then they are kept in air-conditioned room (25°C, 50% RH) until the mass is almost constant. Then they are exposed to a slow heating in an oven up to the target temperature (100, 200, 300, 400, 500, 600, 700 and 800°C) with a step of 2°C. min^{-1} . The stabilization phase takes one hour and cooling phase with closed oven to room temperature (about 3°C. min^{-1}). This choice is guided by the fire conditions and emergency services intervention (ISO834). They were then tested in three-point bending and compressive strength. The weight loss and water absorption were studied. The Portland cement used is a CEM II / B 42.5 NA 442 (Lafarge-Algeria), its chemical and mineralogical compositions (Bogue) are reported in Tables 1 and 2.

Table 1 – Chemical composition of cement (% by mass)

Elements	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃
(%)	23,07	4,94	4,88	58,95	/	0,35	0,13	1,92

Table 2 – Mineralogical composition of cement (% by mass)

Elements	C3S	C2S	C3A	C4AF
(%)	23,07	4,94	4,88	58,95

3 Physical characterization of mortar

Figure 1 shows the evolution of masses of mortar specimens. The masses mortars heated (MT: weight at temperature T) are normalized to the mass of the control mortar (M25: 30 days to water cure and measured at 25°C).

In the first zone that ranges from room temperature to 400°C, there is weight decreases up to 10%. In the second zone from 400°C to 600°C, weight maintenance is observed. In the third zone from 600°C to 800°C, a continuous drop weight about 20% is observed. Also, the weight is reduced by about 3% each time the W/C decreases.

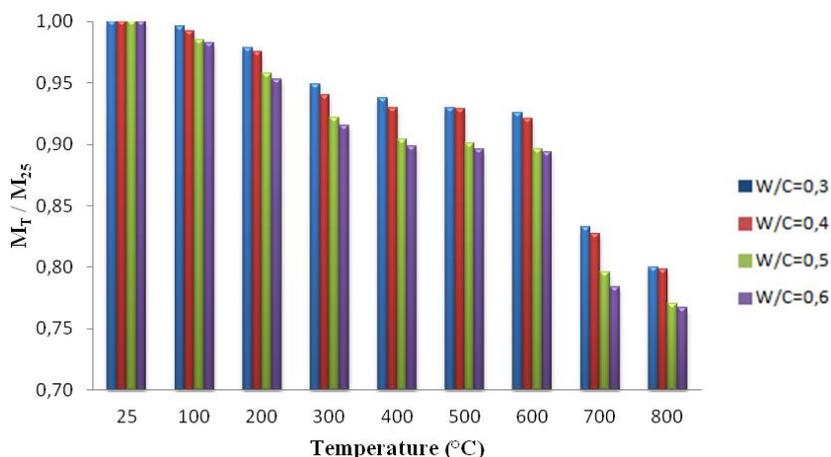


Fig. 1 – Evolution of weight as function of W/C and temperature

The test of water absorption by capillarity was made according to the recommendations AFPC-AFREM [13]. It is determined by immersion of a mortar specimen in water and measuring the mass increase as function of time root. Figure 2 shows the development of water absorption coefficient of the mortar specimens as function of the heating temperature and the water/cement ratio.

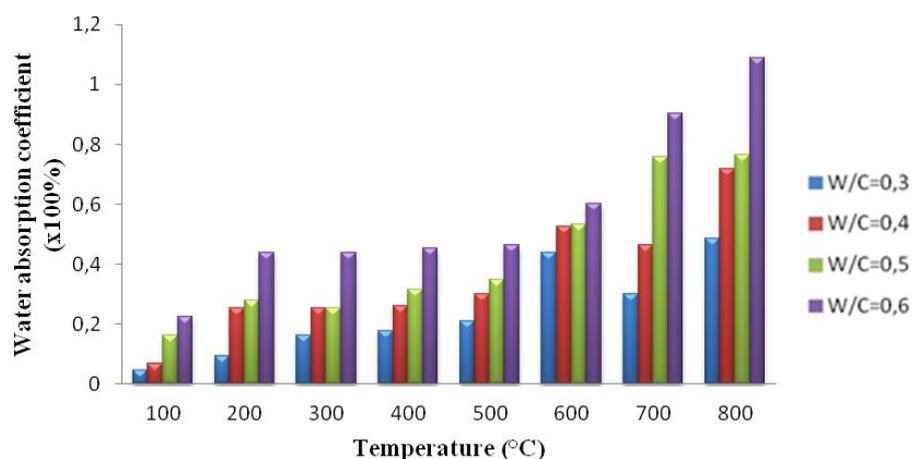


Fig. 2 – Water absorption coefficient

An opposite behavior with the weight loss is observed. In fact, irrespective of the heating temperature, the increasing of capillary absorption coefficient is proportional with the *W/C* ratio. In the first zone ranging from room temperature to 200°C and for a *W/C* ratio equal to 0.6; the capillary absorption increments with 20% from 25°C to 100°C and also between 100°C and 200°C to maintain at the same level in the second zone which ranges from 200°C to 500°C. In the third zone which ranges from 500°C to 800°C, there is a significant and continuous increase of 60% in water absorption ranging from 600°C and over 100% at 800°C for a *W/C* ratio of 0.6.

4 Mechanical Characterization

The mechanical properties of mortars before and after thermal stress were determined through compressive strength and 3-point bending tests. These tests evaluate the impact of thermal stress on the residual behavior of mortars. Mechanical tests were carried out with a loading rate of 0.5 mm/min for the 3-point bending and 0.25 mm/min for the compression.

Figures 3 and 4 shows the results of residual and relating compressive strength (normalized to the initial value measured at 25°C of unheated control mortar: $R_c(T)/R_c(25)$) depending on the *W/C* ratio and temperature.

The first obvious observation that can be noted is that before or after thermal stress, mortars high water-cement ratio (0.5 and 0.6) has a decrease in resistance much greater than low mortars water-cement ratio (0.3 and 0.4).

For a same water/cement ratio, in the first range from room temperature to 200°C, the compressive strength decreases in the order of 40 to 50%. In the second domain (300 to 400°C), we see a slight increase for mortar with water-cement ratio equal to 0.3 to 0.4, in fact, residual strength reaches 80%, but a strength maintaining for mortar with water-cement (0.5 and 0.6) can be noted. Finally, beyond 400°C the compressive strengths decrease significantly regardless the water-cement ratio: in the order of 23 MPa for a W/C = 0.3; 18 MPa for a W/C ratio = 0.4; 5 MPa for a W/C = 0.5 to 4 MPa for a W/C = 0.6. Furthermore, it is important to note that the mortars have significant microcracking (Fig. 5).

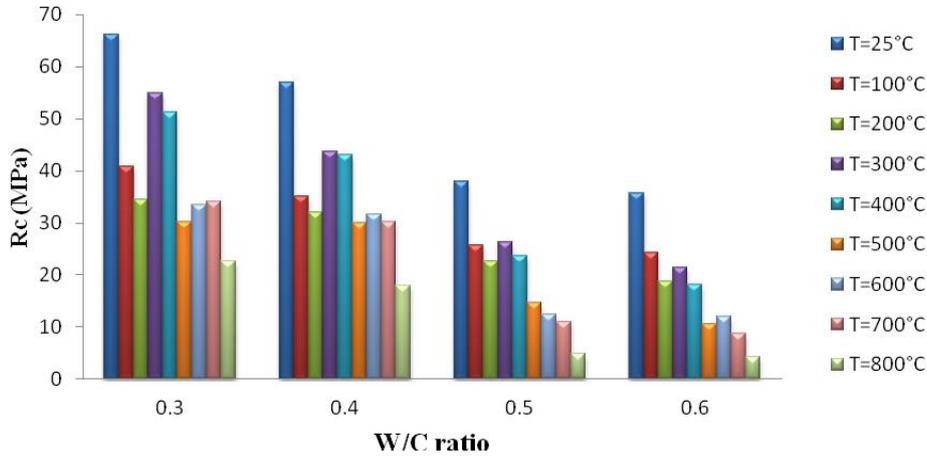


Fig. 3 – Residual compressive strength

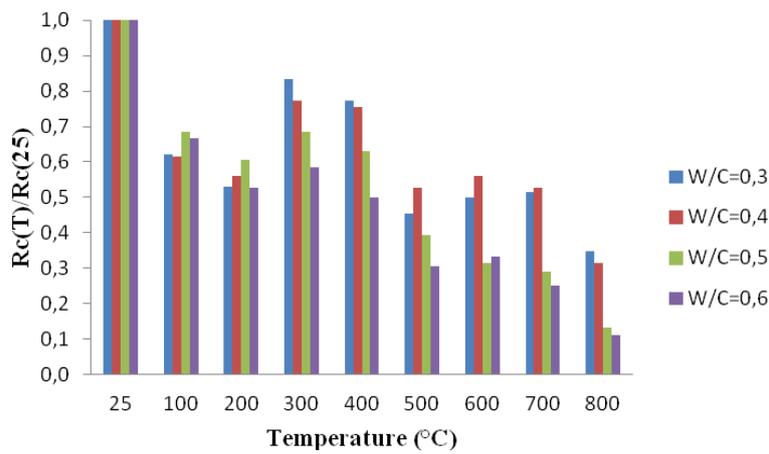


Fig. 4 – Relating residual compressive strength



Fig. 5 – Mortars Microcracking beyond 400°C (W/C=0,5 and 0,6)

Figures 6 and 7 present the results of the residual and relating flexural strength (normalized to the initial value measured at 25°C of unheated mortar: $Rf(T)/Rf(25)$) as function of the W/C ratio and temperature.

Such as for the compressive strength, three zones can be distinguished. In the first area from room temperature to 100°C, the flexural strength decreases approximately by 20% for the water-cement ratios equal to 0.3 and 0.4 and 10% for $W/C= 0.5$ and 30% for a W/C of 0.6. In the second zone (200 to 300°C), we observe a slight increase in flexural strength, especially for water-cement ratios equal to 0.5 and 0.6. In fact, flexural strength exceeds that of control mortar with 30% and 10% respectively. Finally, beyond 300°C, the flexural drop significantly regardless the water-cement ratio to cancel completely 800°C for water-cement ratios of 0.5 and 0.6.

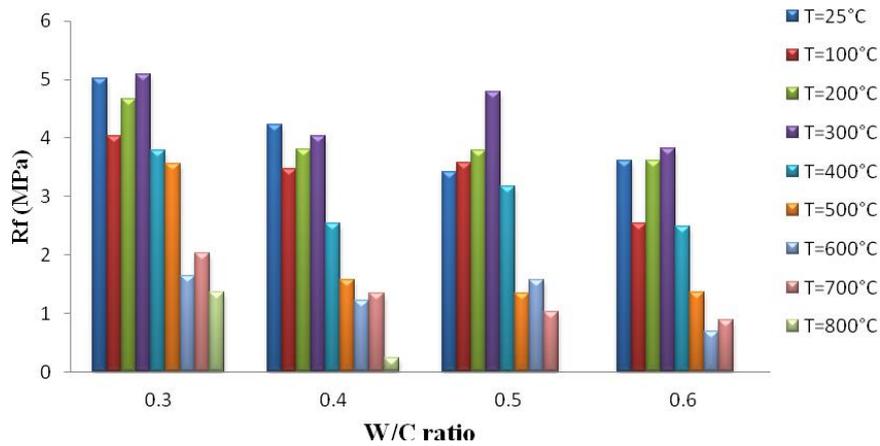


Fig. 6 – Residual flexural strength

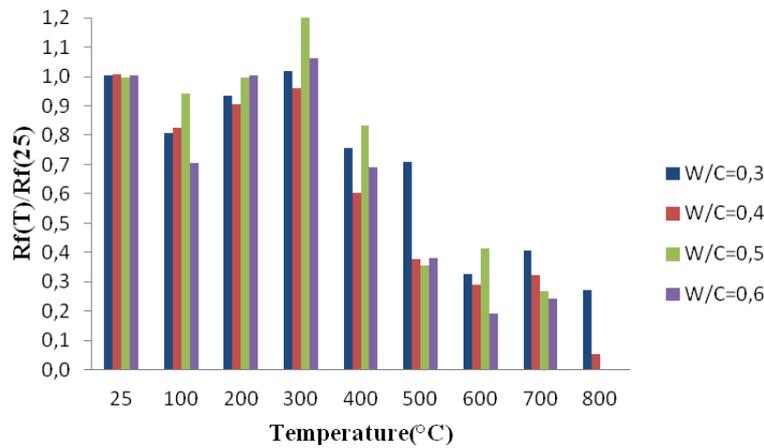


Fig. 7 – Relating residual flexural strength

5 SEM observations

The SEM tests were carried out on samples of unheated control cement and cements heated to 400°, 500° and 800°C. All the cement had the same water-cement ratio of 0.5. 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 observation 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 control 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. 8- photo a). After exposure to high temperature (400°C), the structure of the cement is porous (Fig. 8- photo b). This is due to the departure of the free water and part of the bound water of CSH and the majority of the structural water of Ettringite and Portlandite. At 500°C, the cement structure is more porous due to the total disappearance of portlandite and the progressive dehydration of C-S-H (Fig. 8- photo c). At upper temperature (800°C), the observations

are similar to the results of previous studies which showed that the material is too much damaged and measurements become difficult (Ezziane et al. 2011; Komonen et al. 2003) [14, 15] due to the total dehydration of C-S-H (Fig. 8- photo d).

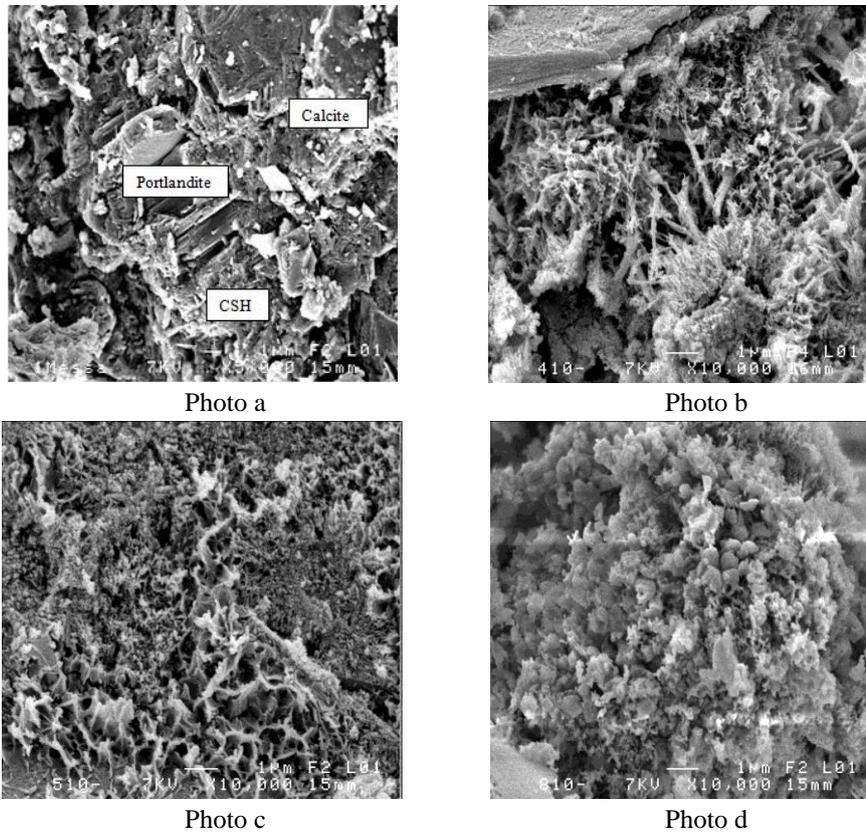


Fig. 8 – SEM observations of cements with W/C = 0.5

6 Discussion

Before and after the thermal stresses, mortars with high water-cement ratio (0.5 and 0.6) show a decrease in compressive strength much greater than mortars with low water-cement ratio (0.3 and 0.4). The decrease in residual compressive strength observed in all tested mortars is due according to Dias et al. [16] to the increase of the chemical energy of water, which allows, by increasing the number of layers adsorbed on the surface of solids, increase pressures disjunction between the different CSH layers. It also indicates that the strength gain observed after heating at 300°C (in this case for the water/cement ratios of 0.3 and 0.4) comes from the additional hydration of the cementitious matrix due to the migration of free water in the pores. This hydration allows for increases of the van der Waals' forces between C-S-H layers, which is recognized by microhardness testing by Xu et al. [17]; Khandaker et al. [18]. Beyond 400°C, all the mortars show a significant decrease of residual strengths. Microcracks in large quantities observed on the mortars are probably derived from the recrystallization phenomenon of Portlandite $\text{Ca}(\text{OH})_2$ during the rehydration of the cement paste [16]. After a thermal sollicitation over 400°C, mortars high water/cement ratio (0.5 and 0.6) have a much greater strength decreasing as mortars with low water/cement ratio (0.3 to 0.4). This is probably related to the difference in microstructure between the two mortars [19].

The residual flexural strength of different mortars has a similar shape to that of the residual compressive strength. However, in the second zone (200 to 300°C), we observe a slight increase in strength, especially for water/cement ratios of 0.5 and 0.6. Indeed, strength exceeds that of the control mortar in the order of 30% and 10% respectively. In this temperature range, the mortars with low W/C (0.3 and 0.4) have a lower flexural strength compared to mortar with high W/C ratio (0.5 and 0.6). This could be explained by a weakening of the largest C-S-H gel in the transition zone paste-aggregates (in our case, sand) for mortar with low W/C [18]. Indeed, the decomposition of C-S-H gel can be carried out in the temperature range between 100 and 300°C as indicated by Jonatka et al. [20]. Above 300°C, the flexural drop significantly regardless of the water/cement ratio to cancel completely at 800°C for water/cement ratios of 0.5 and 0.6. This flexural strength decrease with

increasing temperature joined the results of studies by Noumowe [8]; Kanema Tshimanga [19]; Ezziane et al. [21]; Li et al. [22] and Chang et al. [23] obtained by the tensile test carried out on concrete.

7 Conclusions

In this experimental work, we have tried to contribute the understanding physical, mechanical and microstructural effects of water/cement ratio on the mortars upon heating.

In the light of the results obtained, we can conclude that:

- After thermal stress, mortars with high *W/C ratio* (0.5 and 0.6) show a decrease in compressive strength much greater than those with low *W/C ratio* (0.3 and 0.4). The flexural strength of different mortars has a similar shape to those of the compressive strength.
- The mass loss and water absorption results are consistent with the mechanical behaviour.
- A strong correlation between the evolution of mass loss and thermal gradients in mortar. They highlight in particular the existence of two behavioural domains located before and after 300°C.
- At upper temperature (800°C), the material is too much damaged and measurements become difficult.

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