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Effect of fire and cooling mode on the properties of slag mortars

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Abstract

When building materials are subjected to fire and exposed to slow or rapid cooling, some changes may occur in their characteristics; such as phase transformation, weight loss, aggregate-cement bond, etc., which directly affect its chemical resistivity and mechanical properties. In the present investigation, two types of slag — air-cooled (ACS) and water-quenched slag (WQS) — were used as a fine aggregate in slag mortars. The properties of these slag mortars were compared with the sand one. Three different water-to-cement (w/c) ratios were used: 0.4, 0.5, and 0.6. All mortar samples were prepared and cured in tap water for 90 days, then kept in laboratory atmosphere for about 4 months. The tests were carried out by subjecting the samples to a temperature of 600°C for 2 h, then cooling by three different methods: water, air, and furnace cooling. The results indicated that all mortars exposed to fire irrespective of cooling method suffered a significant depression in compressive strength. However, a remarkable reduction in compressive strength was observed for mortars cooled in air rather than those cooled in furnace or in water. ACS mortars exhibited a relatively higher thermal stability than others. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Blast furnace slag aggregate; Fire; Expansion; Chemical resistivity

1. Introduction

Concrete, although not a refractory material, is noncombustible and has good fire-resistance properties. The term *fire-resistant* should not be devoted to concrete alone in general definition, but to its main constituent materials, i.e., aggregate, cement, and water. The contributions of these components to the fire-resistance of a structure were discussed earlier [1]. On heating, a neat Portland cement paste first expands owing to its normal thermal expansion. This expansion, however, is exposed to a contraction due to the shrinkage of the material as water is driven off from it. The contraction due to drying eventually becomes much larger than its normal thermal expansion and the material then begins to shrink. The temperature at which the maximum shrinkage is reached varies with the size of the specimen and the conditions of heating. It may be as high as 300°C for air-dried specimens under conditions of fairly rapid heating. At higher temperatures, the neat cement steadily shrinks, the contraction from the original dimensions amounting ultimately about 0.5% or more. During this process, severe cracking occurs [2].

Hydrated Portland cement contains a considerable proportion of free calcium hydroxide, which loses its water above 400–500°C, leaving calcium oxide (quick lime). If CaO becomes wetted after cooling as exposed to moist air, it rehydrates to $Ca(OH)_2$ accompanied by an expansion in volume that may disrupt a concrete, which has withstood a fire without disintegration [3]. In mortar and concrete, the aggregate undergoes progressive expansion on heating while the set cement shrinks beyond the point of maximum expansion. The two opposing actions progressively weaken and crack the mortar or concrete. The various types of aggregates used in concrete differ considerably in their behavior on heating. Quartz, the principal mineral constituent of the acid igneous rocks, expands steadily up to 573°C. At this temperature, it undergoes a sudden expansion of 0.85%, caused by the transformation of low α-quartz to high β-quartz [4], which has a disruptive action in concrete. Sandstone shrinks on heating, and therefore, it counteracts to some extent the expansion of the quartz grains. The loss in strength of sandstone mortars on exposure to fire is, however, often high, and therefore, sandstone does not form a good fire-resistant aggregate [5].

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Table 1
Main properties of OPC used

Property	Result	BS limits
Initial setting time (h:min)	1:45	≥ 45
Final setting time (h:min)	4:25	≤ 10
Fineness (% retained on sieve 90 μm)	8	≤ 10
Compressive strength (MPa)		
After 3 days	22.5	≥ 16
After 7 days	29.7	≥ 24

The best fire-resistant aggregate, which is characterized by a very fine crystalline texture or a noncrystalline basic material such as limestone, expands steadily until a temperature of about 900°C is reached, then begins to contract owing to decomposition of $CaCO_3$ with liberation of CO_2. It has often been considered, on account of this decomposition, that the concrete with limestone has no fire-resistance beyond this temperature [6]. Broken bricks also form good fire-resistant aggregate, because it is free from pure quartz. Lightweight aggregates such as pumice, foamed slag, and expanded clay products have in themselves high resistance to fire, and concrete made from them has low heat conductivity. Therefore, their heat capacity is less than that of heavier concretes. Long series of tests on the fire-resistance of structures have been carried out in Britain and USA. All concretes, which are considered the most fire-resistant, attained a serious reduction in strength at a temperature above 600°C and fail if exposed for a considerable time to a temperature exceeding 900°C [7].

Blast furnace slag is a nonmetallic material consisting essentially of silicates and alumino-silicates of calcium [8]. It is considerably used in the production of cementing materials, as aggregate in concrete and in the production of lightweight aggregate. When the slag is allowed to cool slowly in air, it solidifies into a gray crystalline material known as slowly air-cooled (ACS) or crystallized slag. This slag is used in a road-stone and as a concrete aggregate. When the slag is cooled very rapidly by water, it solidifies and granulates as a glass-forming water-quenched, foamed or granulated slag (WQS). The chemical composition of slag can vary over a wide range depending on the nature of the ore, the composition of the limestone flux, coke consumption, and the type of iron being made [9]. Our previous work [10], showed the effectiveness of ACS and WQS as a fine aggregate on the mortar strength and durability.

Therefore, it comes out that although there are many research findings concerning the effect of fire on concrete made from different types of aggregates, there is a need to pay attention to the role of slag as a fine aggregate in improving the fire-resistance of concrete. The present work aims to study the effect of slag, as a fine aggregate, and water-to-cement (w/c) ratio on the properties of mortars exposed to fire at 600°C for 2 h soaking time with heating rate of 10–20°C/min followed by cooling using three different methods: water, air, and furnace cooling. Furnace cooling is leaving the samples in the semi-open muffle furnace till they cool down to room temperature.

Table 2
The chemical oxide composition of OPC and slag (wt.%)

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	IL
OPC	20.73	5.29	3.07	63.25	2.6	2.55	1.21
Slag	32.96	12.17	1.83	41.64	2.16	4.25	1.02

Table 3
Grading of sand and slags used

	% Passing		
Sieve size (mm)	Sand	ACS	WQS
5.00	100	100	100
2.50	92.0	93.5	91.5
1.25	70.5	80.7	63.8
0.60	50.0	61.2	27.3
0.30	20.0	38.0	7.30
0.15	10.0	15.0	2.60

2. Materials and experimentation

The cement used in mortar mixes was ordinary Portland cement (OPC) from Suez Cement and its properties are shown in Table 1. Two types of blast furnace slags: ACS and WQS were used. The Iron and Steel Company at Helwan, Egypt, provided these slags. The chemical oxide compositions of OPC and slag used are given in Table 2. The sand used was the locally available and it was clean and free from impurities. The grading of sand and slags used are given in Table 3.

The three types of mortar mixes (sand, ACS, and WQS mortars) were prepared using three different w/c ratios: 0.4, 0.5, and 0.6. Cement content of 400 kg/m^3 and sand- or slags-to-cement ratio of 3 were maintained throughout. For each mortar mix, four sets of specimen were prepared for

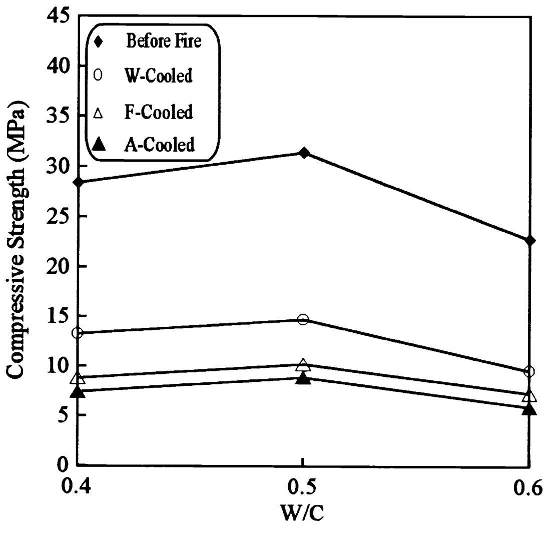

Fig. 1. Compressive strength vs. w/c ratios for sand mixes at age 7 months.

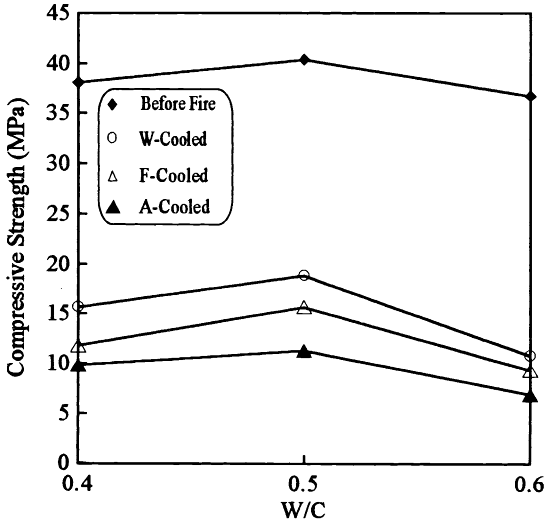

Fig. 2. Compressive strength vs. w/c ratios for ACS mixes at age 7 months.

each w/c ratio. Each set consists of three 7.5 × 15 cm cylinders. The mortar constituent materials were batched separately by weight. Mixing was performed in a small rotating-drum mixer. First, cement and sand or slag were dry mixed until a homogeneous color was observed and then water was gradually added while mixing continued for about 5 min. All specimens were cast in steel molds, then demolded after 24 h and cured in fresh water for 3 months.

After curing, the specimens were kept in the normal atmospheric conditions for 4 months to attain the steady state composition. Then, the specimens were exposed to fire at 600°C for 2 h soaking time in semi-open muffle furnace with heating rate 10–20°C/min. After fire, the specimens were cooled by water quenching (rapid cooling), air cooling, or left to cool with the furnace switched off, till the samples reach the room temperature. The compressive strength of the three tested mortars was measured before and after their exposure to fire and cooling process. The compressive test was carried out in a hydraulic universal testing machine of 1000 kN capacity. The X-ray diffraction (XRD) analysis (using Ni filter, Cu–Kα radiation technique) was performed on representative samples to detect the phase composition and chemical transformation. The XRD patterns obtained were converted to a series of lattice spacing, d (in Å), and the relative intensities were visually estimated.

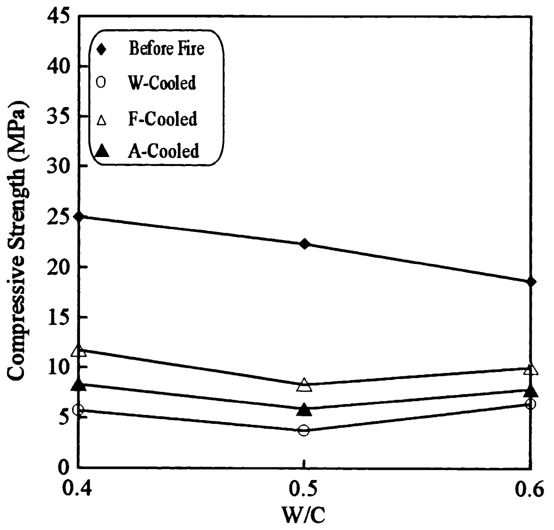

Fig. 3. Compressive strength vs. w/c ratios for WQS mixes at age 7 months.

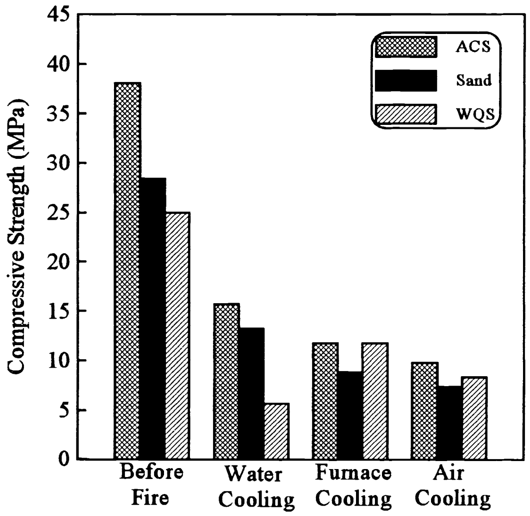

Fig. 4. Compressive strength vs. cooling method for mortars with 0.4 w/c at age 7 months.

3. Results and discussion

The variation of the compressive strength of the three different mortars mixes with w/c ratio is shown in Figs. 1–3. It is indicated that all mortar mixes exposed to fire irrespec-

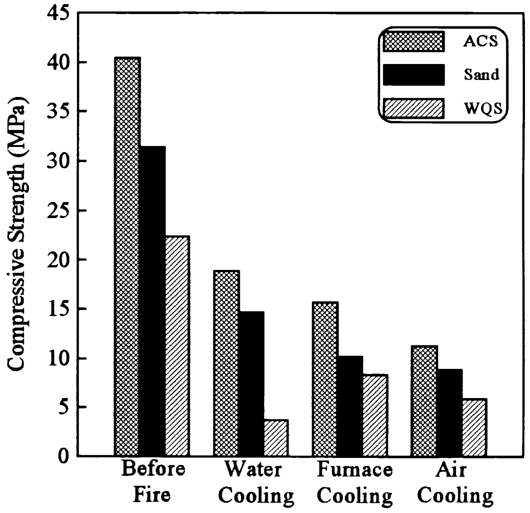

Fig. 5. Compressive strength vs. cooling method for mortars with 0.5 w/c at age 7 months.

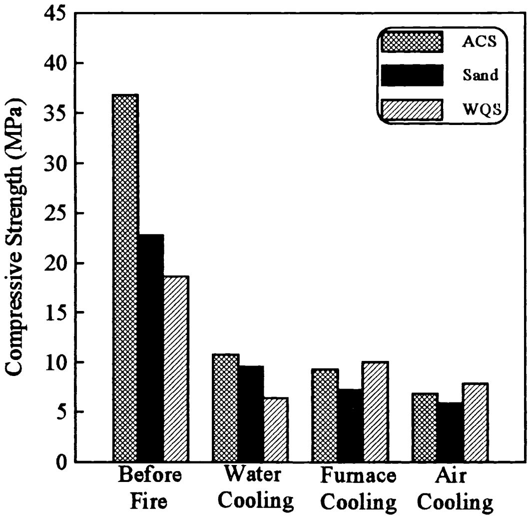

Fig. 6. Compressive strength vs. cooling method for mortars with 0.6 w/c at age 7 months.

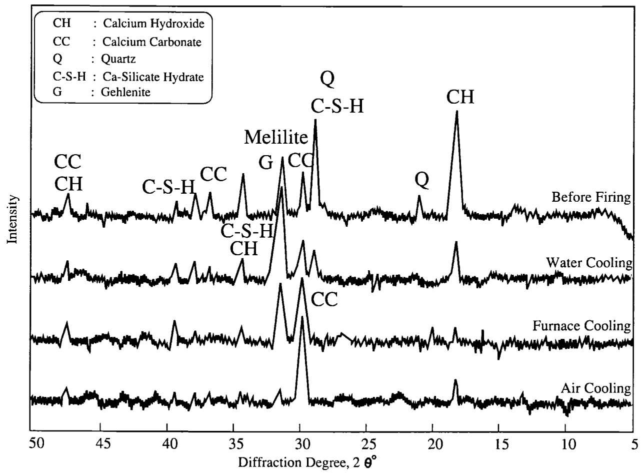

Fig. 8. XRD pattern of ACS mortars with 0.5 w/c ratio at age 7 months.

tive of cooling method suffered a significant depression in compressive strength. This is due to thermal decomposition of some binding products such as Ca sulphate aluminate hydrate and calcium silicate hydrates. The decrease in compressive strength of sand mortars cooled in water reached about 53%, 53%, and 58% for w/c ratios of 0.4, 0.5, and 0.6, respectively. Strength losses in sand mortars are caused by the transformation of low α-quartz to high β-quartz at 573°C accompanied by a sudden expansion and change in volume [4]. In general, there was loss in the weight, which mainly associated with liberation of water from the decomposition of $Ca(OH)_2$ and the other formed cement hydrates (C-S-H). In the case of ACS mortars cooled in water, the reduction in compressive strength reached about 59%, 53%, and 71% for the w/c ratios of 0.4, 0.5, and 0.6, respectively. However, in the case of WQS, it was about 77%, 83%, and 66%, respectively. The relatively less reduction in compressive strength of ACS mortars as compared with WQS mortars may be due to the fine crystalline composition of ACS, which is characterized by high thermal stability of mortars containing it. The chemical composition of ACS consisted of melilite, as the main constituent, gehlenite (C_2AS), and akermanite (C_2MS_2) [5]. But the significant reduction in compressive strength of WQS mortars may be due to the thermal stresses in the matrix during cooling.

The w/c ratio showed a remarkable effect on the mortars compressive strength after their exposure to fire and cooling process, as shown in Figs. 1–3. Mortar mixes with sand or ACS showed an increase in compressive strength with increasing the w/c ratio from 0.4 to 0.5, while significant decrease was observed when w/c ratio reached 0.6. On the contrary, WQS mortars showed a decrease in compressive strength at 0.5 w/c ratio and returned to increase again at 0.6 w/c ratio. This phenomenon can be related to relative hydraulicity of WQS in comparison with less or nonhydraulic sand and ACS that creates internal steam at high

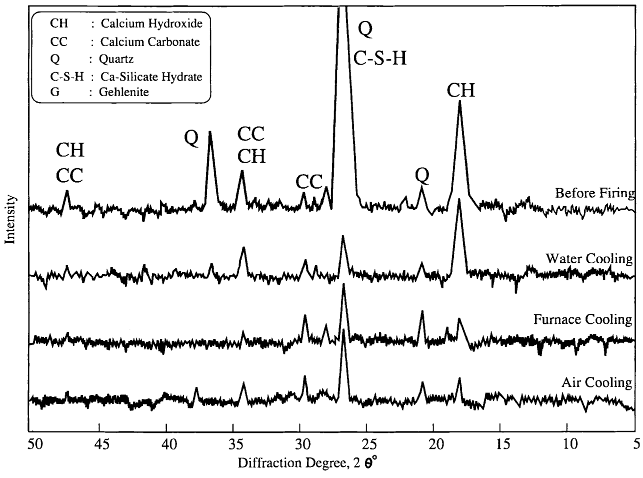

Fig. 7. XRD pattern of sand mortars with 0.5 w/c ratio at age 7 months.

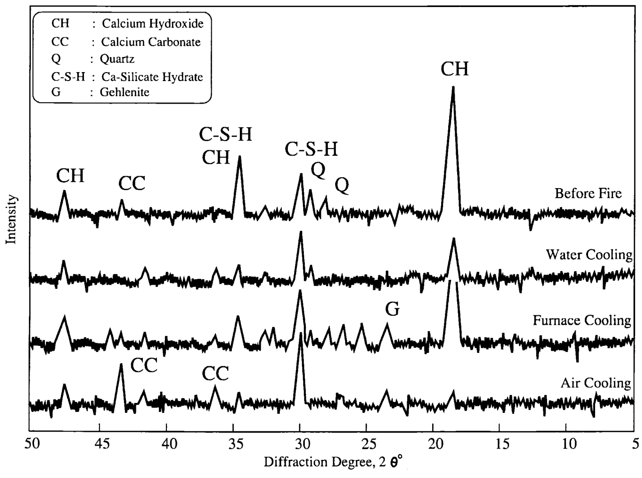

Fig. 9. XRD pattern of WQS mortars with 0.5 w/c ratio at age 7 months.

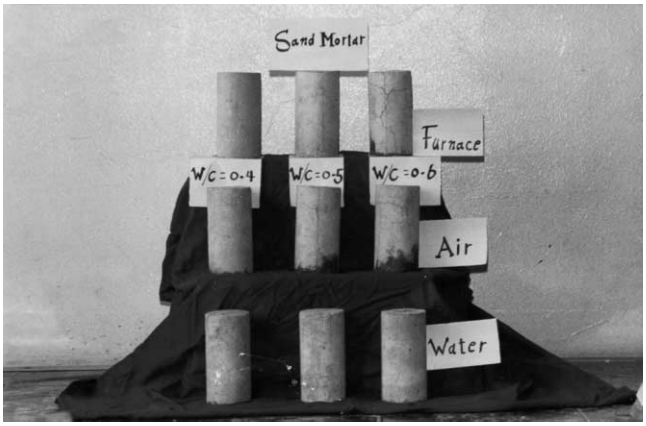

Fig. 10. Crack pattern of sand mortars.

temperatures (internal autoclaving) [11]. This accelerates the hydration of glass phases slag forming a relatively more fine crystalline material. In the nonhydraulic aggregates, the higher porosity that arose from high water content facilitates the interaction and penetration of carbon dioxide into inner layers of mortar.

Figs. 4–6 show the variation of compressive strength of the three different mortars with the three studied cooling methods. It is clear that the compressive strength of mortars exposed to fire is noticeably affected by the cooling method. A remarkable reduction in compressive strength was observed for mortars cooled in air rather than that cooled in furnace or in water as shown in Figs. 4 and 5 for 0.4 and 0.5 w/c ratios. This can be explained by the long time exposure to atmospheric conditions that result in the transformation of liberated active CaO due to fire to $CaCO_3$, which is accompanied by volume change and cracking. When mortars were rapidly cooled by water quenching or allowed to cool gently with furnace cooling, a relative higher compressive strength was observed.

The XRD patterns of the mortars made of sand, ACS, and WQS are shown in Figs. 7–9 for the three cooling methods. Sand mortars showed peaks of calcium carbonate ($CaCO_3$), calcium hydroxide [$Ca(OH)_2$], calcium silicate hydrate (C-S-H), and quartz phases. The intensity of C-S-H peak was decreasing as a result of thermal decomposition. The intensity of peaks characteristic for calcium carbonate phase increased in mortars cooled in air and in furnace than those cooled in water. This phenomenon can be used to explain the formation of cracks and the remarkable reduction in compressive strength of mortars cooled in air and in furnace as compared with those cooled in water. The patterns also showed an increase in $Ca(OH)_2$ peak intensity in the case of water cooling than other cooling methods, due to transformation of CaO to $Ca(OH)_2$ after wetting. This is true for sand and ACS mortars but not for WQS mortars (see Fig. 9 and furnace cooling).

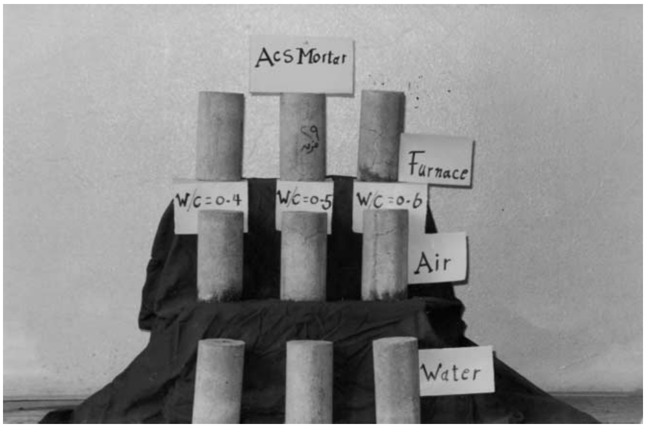

Fig. 11. Crack pattern of ACS mortars.

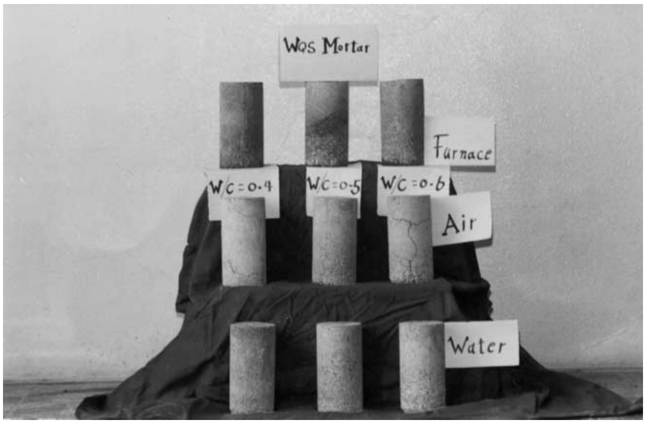

Fig. 12. Crack pattern of WQS mortars.

Mortars with ACS showed peaks of some crystalline constituents such as gehlenite and melilite, and therefore, ACS mortars exhibited a relatively high thermal stability. In the case of WQS mortars, small amount of hydrated phases was observed after fire. Furnace cooling of WQS mortars created more fine crystalline textures as a result of the heat treatment, but air cooling favored the formation of $CaCO_3$ due to the carbonation of active surface CaO into $CaCO_3$.

The general cracking pattern for all the tested mortar specimens is shown in Figs. 10–12. In air and furnace cooling, the formation of surface cracks took place at all w/c ratios, but their number and widths were increased with increasing the w/c ratio. This is mainly associated with physicochemical processes connected with the liberation of water at high temperatures, and therefore, mortars with 0.6 w/c ratio gave a relatively higher porosity than 0.4 and 0.5 w/c ratios. In the case of water cooling, the mortar specimens showed a higher stability without any observed surface cracks due to the insufficient time to transform CaO into $CaCO_3$. But after quenching, $Ca(OH)_2$ is formed and filled the surface porosity. The abovementioned cracking patterns explain the effect of the three different cooling methods on the mortars compressive strength, and confirm the use of water to extinguish fires.

4. Conclusions

This paper investigated the effect of three different cooling methods: water, air, and furnace cooling on the properties of mortars exposed to fire at 600°C for 2 h. In these mortars, three types of fine aggregates (sand, ACS, and WQS) and three different w/c ratios (0.4, 0.5, and 0.6) were used. The results showed that:

1. All mortar mixes exposed to fire irrespective of cooling method suffered a significant depression in compressive strength. The reduction in compressive strength of sand and ACS mortars reached an average of 55% and 60%, respectively, while it reached an average of 75% in WQS mortars.

2. The w/c ratios showed a remarkable effect on the mortars compressive strength after their exposure to fire and cooling process. The compressive strength of sand and ACS mortars was increased with increasing w/c from 0.4 to 0.5, while it decreased at 0.6 w/c ratio. In contrast, the compressive strength of WQS mortars was increased at 0.6 w/c ratio due to the relative hydraulicity of WQS.

3. The compressive strength of mortars is noticeably affected by the cooling method. A remarkable reduction in compressive strength was observed for mortars cooled in air rather than those cooled in furnace or in water.

4. Monitoring of cracks and XRD patterns illustrated the improved performance of mortars cooled in water as compared to those cooled in air or in furnace.

References

[1] British Standard Institution, Fire Tests on Building Materials and Structures: Part 23. Methods for Determination of the Contribution of Components to the fire resistance of a Structure (B.S. 476), British Standard Institution, London, 1987.

[2] F.M. Lea, The Chemistry of Cement and Concrete, fourth ed., Arnold Publishing Group, London, 1998.

[3] V.C. Ramachandran, Application of Differential Thermal Analysis in Cement Chemistry, Chemical Publishing, New York, 1969.

[4] H.F.W. Taylo, Cement Chemistry, Academic Press, New York, 1990.

[5] S.N. Ghosh, Advances in Cement Technology; Critical Reviews and Case Studies on Manufacturing, Quality Control, Optimization and Use, Pergamon Press, London, 1983.

[6] British Standard Institution, Specification for Lightweight Aggregate for Masonry Units and Structural Concrete (B.S. 3793), British Standard Institution, London, 1990.

[7] F. Škvàra, V. Ševèik, Influence of high temperature on GF Portland cement materials, Cem. Concr. Res. 29 (1999) 713–717.

[8] Siliceous by-products for use in concrete, Final Report of the 73 BSC RILEM, Committee, 1988.

[9] M. Regourd, Slags and slag cements, Concrete Technology and Design Cement Replacement Materials, vol. 31, Guild Ford, Surrey Univ. Press, France, 1986, pp. 73–99.

[10] M.E.A. Metwally, S.A. Ahmed, M.M. Balaha, Properties of crushed blast-furnace slag mortars, 6th Int. Conf. Build. Constr., Cairo, Egypt, 21–26 June, vol. I, (1999) 497–505.

[11] M.S. Morsy, A.F. Galal, S.A. Abo-El-Enein, Effect of temperature on phase composition and microstructure of artificial pozzolanic-cement pastes containing burnt kaolinite clay, Cem. Concr. Res. 28 (1998) 1157–1163.