COUPLED EFFECT OF ELEVATED TEMPERATURE AND COOLING CONDITIONS ON THE PROPERTIES OF GROUND CLAY BRICK MORTARS

ABSTRACT

When a concrete structure is exposed to fire and cooling, some deterioration in its chemical resistivity and mechanical properties takes place. This deterioration can reach a level at which the structure may have to be thoroughly renovated or completely replaced. In this investigation, four types of cement mortars, ground clay bricks (GCB)/sand namely 0/3, 1/2, 2/1 and 3/0, were used. Three different cement contents were used: 350, 400 and 450 kg/m³. All the mortars were prepared and cured in tap water for 3 months and then kept in laboratory atmospheric conditions up to 6 months. The specimens were subjected to elevated temperatures up to 700°C for 3h and then cooled by three different conditions: water, furnace, and air cooling. The results show that all the mortars subjected to fire, irrespective of cooling mode, suffered a significant reduction in compressive strength. However, the mortars cooled in air exhibited a relatively higher reduction in compressive strength rather than those water or furnace cooled. The mortars containing GCB/sand (3/0) and GCB/sand (1/2) exhibited a relatively higher thermal stability than the others.

KEY WORDS

- Ground clay bricks,
- fire resistance,
- XRD,
- microstructure,
- compressive strength, and cooling mode.

1 INTRODUCTION

The behaviour of concrete structures at elevated temperatures is of significant importance in predicting the safety of structures in response to certain accidents or particular service conditions. Elevated temperatures affect concrete’s microstructure, strength properties, mass, and porosity as well as permeability. Factors such as the type of aggregate and cement, porosity, moisture content, thermal properties, sizes of structural members and construction type, determine the level of the fire resistivity of the material. An increase in the size
of the structural members increases their fire resistance. The type of cooling (in air or water) affects the residual compressive and flexural strength, the effect being more pronounced as the temperature increases. Residual mechanical properties where natural cooling was usually employed that have been reported in most previous literature might be overestimated.

An evaluation of the fire resistance of concrete needs more experimental data obtained under various cooling regimes such as water spraying or quenching, where they cause different stresses in reinforced concrete members at high temperatures, and the structural member can lose its load-bearing capacity.

Concrete structures can be exposed to an elevated temperature, e.g., concrete foundations for launching rocket-carrying spaceships, concrete structures in nuclear power stations or those accidentally exposed to fire as has been the case of many tunnels registered in many countries [1]. The fire-induced collapse of the World Trade Center in New York, USA, with the resulting heavy casualties, has highlighted the importance of the performance of construction materials at elevated temperatures. Engineering properties such as strength and stiffness and the thermal properties (thermal conductivity and thermal expansion) of these materials have to be understood by designers when selecting alternative materials [2].

Two major problems in concrete occur when it is heated: one is a deterioration in the mechanical properties of concrete such as physico-chemical changes in the cement paste and aggregates, and thermal incompatibility between the aggregates and cement paste according to the temperature level, heating rate, load applied, and moisture loss. The thermo-physical properties of concrete decrease with an increase in temperature except for a specific heat; specifically, conductivity and diffusivity are 50% lower at 900°C as compared with the same values at room temperature [3]. The other problem is the spalling of concrete [4]. Elevated temperatures also reduce the tendency of irreversible creep and shrinkage. Scanning electron microscopy (SEM) studies have shown that at elevated temperatures, the outer product of calcium silicate hydrate (CSH) gel is denser and does not fill the capillary pore space as effectively; thus, the microstructure is more heterogeneous [5].

At elevated temperatures, 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. The contraction due to drying eventually becomes much larger than its normal thermal expansion, and then the material begins to shrink. The temperature at which the maximum shrinkage is reached varies with the size of the specimen and the heating conditions. It may be as high as 300°C for air-dried specimens under conditions of fairly rapid heating. At higher temperatures, cement paste steadily shrinks, the contraction from the original dimensions ultimately amounting to about 0.5 % or more. During this process, severe cracking occurs [6].

The most important effects of an elevated temperature on concrete are: the dehydration of the cement paste, an increase in porosity, modifications in the moisture content, thermal expansion, alteration of pore pressure, loss of strength, thermal cracking due to incompatibility, thermal creep and thermal spalling due to excessive pore pressure. Water distribution and transport, whether in a gaseous or liquid form, play important roles in the local damage to concrete structures [7-10]. During heating, the endothermal nature of vaporization creates locally high thermal gradients and high vapour pressure, which can lead to tensile stresses exceeding the concrete’s strength [11]. The escape of chemically bound water in CSH leads to the failure of concrete at temperatures over 450°C. Hydrated Portland cement pastes contain a large amount of Ca(OH)₂, which dehydrates to CaO between 450 and 600°C. Upon cooling and exposure to moist air or wetting, CaO rehydrates to Ca(OH)₂ with a volume expansion of 9.7% in addition to the formation of cracks [12]. Pozzolanic materials can react with the liberated lime from ordinary Portland cement to form additional amounts of calcium silicate hydrates. CSH phases are deposited within the pore system, which can accommodate larger amounts of hydration products, leading to relatively high compressive strength values as compared with ordinary Portland cement (OPC) paste. Pozzolanic cements possess good fire resistance and dimensional stability when exposed to high temperatures and then high humidity or wetting [13].

The type of aggregate strongly influences the behaviour of concrete at elevated temperatures. The aggregates’ thermal expansion is partly opposed to the drying of the cement paste. The aggregates undergo progressive expansion upon being heated, 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 upon being heated. Quartz, the principal mineral constituent of 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, which has a disruptive action in concrete [14]. The best fire-resistant aggregates are characterized by a very fine crystalline texture or a non-crystalline basic material such as limestone, which expands steadily until a temperature of about 900°C is reached; it then begins to contract owing to the decomposition of CaCO₃ with the liberation of CO₂. This phenomenon makes it possible to think that limestone aggregate, whose thermal coefficient of expansion is lower than that of siliceous aggregate, is more favourable to the behavior of concrete at elevated temperatures. Recent studies have shown the weak influence of the kinetics and durations of heat treatment on the residual properties of concrete [9,10,15]. It has often been considered on account of this decomposition that concrete with limestone has no fire resistance beyond this temperature [16].

Sandstone shrinks upon been heated; therefore, it counteracts to some extent the expansion of quartz grains. The loss in strength of sandstone mortars upon exposure to fire is high; therefore, sandstone does not form a good fire resistant aggregate [17]. In contrast to sandstone, ground clay bricks (GCB) form a good fire-resistant aggregate. GCB is a solid waste material, which is mainly constituted of silica quartz, aluminosilicate, anhydrite, and hematite. Therefore, it acts as a pozzolanic material [18]. Expanded clay products in themselves have a high fire resistance; therefore, their heat capacity
is less than that of heavier concretes. All concretes which are considered the most fire resistant attain a series of reductions in strength at a temperature above 600°C and fail if exposed for a considerable time to a temperature exceeding 900°C [19].

Concrete structures may accidentally be exposed to fire. To extinguish it, water is generally sprayed on the structure that is on fire. This causes a rapid cooling of the heated concrete, and its structural members can lose their load-bearing capacity. Therefore, it turns 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 GCB as an aggregate in improving the fire resistance of concrete. This work is aimed at investigating the coupled effect of elevated temperatures and cooling conditions on the properties of GCB mortars.

2 EXPERIMENTAL TECHNIQUES

The materials used in this investigation were ordinary Portland cement (OPC) from Bene-Suef Cement Company, Egypt, and ground clay bricks (GCB) from Misr Brick Company, Helwan. The properties of the OPC used are shown in Table 1. The chemical oxide compositions of the OPC, sand and GCB used are listed in Table 2. The sand used was the locally available sand (6-October City sand); it was clean and free from impurities. The grading of the sand and GCB used are shown in Table 3.

Mortar mixes made of GCB sand, 0:3, 3:0, 1:2 and 2:1, were prepared using different cement contents of 350, 400 and 450 kg/m³. The mix composition of the mortars investigated is shown in Table 4. The fine aggregates and cement were mixed with the water contents, which were sufficient to obtain a flow of 110±5 with 25 drops of the flow table (ASTM 1992).

Each of the mortar-constituent materials were batched separately by weight and mixed in a rotating mixer to ensure complete homogeneity; then they were mechanically and gradually mixed with water for about 5 minutes. All the mortar specimens were cast in steel molds (50×50×50 mm cubes), demolded after 24 hours and cured in fresh tap water at 23 ± 1°C for 90 days; they were then kept in laboratory atmospheric conditions for up to 6 months to attain a steady state composition.

The specimens were exposed to fire at 700°C for 3 hours’ soaking time in a muffle furnace with a heating rate of 10°C/min; they were then cooled by three different cooling modes (water quenching, air cooling, or switched-off furnace cooling till the specimens reached room temperature). The compressive strength of the tested mortars was measured according to the ASTM specifications before and after their exposure to the fire and cooling process (ASTM 1992) [20]. A compressive test was carried out in a hydraulic univer-
sal testing machine (ADR) of a 2000 KN capacity, and the furnace used was a Muffle furnace with a heating rate of 10°C/min as shown in Figure 1. The X-ray diffraction analysis (XRD) was performed on representative samples to detect the phase composition and chemical transformation. Also, a digital camera was used to detect the cracking patterns of the selected tested mortars as shown in Figure 2. The microstructure of the selected samples was examined using a JEOL-JXA-840 (high-resolution imaging at up to 100,000 ×) scanning electron microscopy.

3 RESULTS AND DISCUSSION

The variations in the compressive strength of the mortars investigated for 6 months with different cement contents, namely 350, 400 and 450 kg/m³, are graphically represented in Figures 3-6. The mortar specimens were exposed to fire at 700°C for a 3h soaking time. The mortars suffered a significant depression in compressive strength irrespective of the cement constituents and cooling mode. This may be due to two factors: the first is the thermal decomposition of some cementitious binding hydrated products such as calcium silicate hydrate (CSH), calcium aluminate hydrate (CAH), and calcium aluminosilicate hydrate (CASH), which led to the formation and enlargement of micro-cracks. The second factor is due to the increase in the degree of crystallinity of the formed hydrates, leading to a sort of opening of the pore system. Therefore, the values of the compressive strength decreased with the firing temperature.

![Fig. 1 Compressive strength testing machine (ADR 2000 KN) and Muffle furnace with heating rate 5°C/min.](image1)

![Fig. 2 Cracking patterns of the investigated mortars before and after firing (W: water cooling, A: air cooling, F: furnace cooling).](image2)

![Fig. 3 The variations of the compressive strength values of sand mortars (GCB:sand; 0:3) cured at 6 months.](image3)

![Fig. 4 The variations of the compressive strength of GCB: sand; (3:0) with cement content at 6 months.](image4)
is mainly replaced by a crystalline phase with a structure similar to layer space at 750-800°C; the CSH gel completely disappears, and the additional products as a result of the pozzolanic reaction to form a denser strength is due to the filling up of the pores with additional hydrates. The increase in compressive strength is due to the formation of CSH. The cement specimens subjected to firing indicated the structural modification of the CSH gel. The cement content showed a remarkable effect on the values of the compressive strength for all the mortars investigated. Before firing, the values of compressive strength increase with the increase in cement content from 350-450 kg/m³, due to the production of additional amounts of CSH, CAH, and CASH. But after firing, the compressive strength of cement mortars increases with the increase in the cement content up to 400 kg/m³ and then decreases at 450 kg/m³. The increase in cement content above 400 kg/m³ leads to a reduced permeability relative to 350 or 400 kg/m³. Mortar containing a cement content higher than 400 kg/m³ may be more susceptible to explosive spalling under fire conditions due to the buildup of the pore pressure in the cement mortar.

In fact, it is true that those dehydrated phases induced by high temperatures are subjected to a meta-stable state. They may quickly polymerize each other and form new products again, especially CaO. This process does not need to overcome the protective layer effect and nucleation process [27]. Generally, CSH gel, as a primary hydrated mineral in hydrated cement paste, mainly consists of the dimer and chain of silicate anion, which are presented by 29Si MAS-NMR [28-29]. After high temperature exposures at 450 and 750°C in turn, the spectrum of Q³ and Q⁴ gradually disappears and is replaced by the new dimer and monomer of Q⁵ and Q⁶ [30]. Thus, the silicate polymerization of CSH gel is gradually depolymerized to form a low degree of polymerization structure such as short linear chains, dimer and monomer [31-32]. When meeting cooling water, they may react with it quickly and transform into CSH gel again. In addition, the dehydrated phase, CaO, will quickly react with water and consume more water. At a higher temperature > 500 ºC the unhydrated phases display the formation of inner and outer hydrated products to form dense masses of hydration products having micro and narrow pores, without the formation of micro-cracks [33]. The higher compressive strength of the mortar containing GCB/sand in a ratio of 3/0 and 1/2 than all the other mortars investigated.
especially before the firing process, is due to the pozzolanic reaction of the GCB with the liberated lime to produce additional amounts of CSH, CAH and CASH hydrates, which are deposited within the pore system and lead to the formation of a dense and compact internal structure. From the literature, it is known that GCB increases the amount of water of a consistency for pastes when used as a cement replacement [13,19]. GCB consumes a greater amount of water than sand. As expected, the addition of GCB not only affects porosity or density, but also the packing factor, and therefore, the strength of the system. The interface condition is mainly responsible for the mechanical strength. The increase in strength of the GCB:sand (1:2) is due to the interfacial transition zone in the matrix with the sand fraction or is due to the nucleating effect of the GCB [13,34]. The increase in the amount of GCB (GCB:sand; 2:1 and/or 3:0) and the development of strength depend primarily on the formation of hydrated calcium silicate as the main hydration product, which is precipitated into the water-filled spaces to form a more compact body. This is mainly attributed to the crystallization of the initially formed hydrates having strong binding forces and/or their transformation into other hydration products having weaker binding forces [35].

But after thermal treatment, the decrease in the compressive strength of the mortar containing GCB:sand in a ratio (2:1) was lower than the others. This is due to the two opposing actions of GCB. The action is due to the pozzolanic effect of GCB, which leads to an increase in cementitious binding hydrates. The second action is also due to the increase in the total porosity of the GCB/sand mortar by increasing the GCB content, because the higher porosity which arises from a higher water content facilitates the interaction and penetration of carbon dioxide into the inner layers of the mortar. Therefore, the susceptibility of a cement mortar to explosive spalling under fire conditions may be decreased by increasing its total porosity due to the decrease in the sudden expansion, volume change and cracking. 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 [14]. The relatively lower reduction in the compressive strength of GCB/sand mortars as compared with sand mortars under fire conditions may be due to the composition of GCB, which is free from quartz. Therefore, GCB forms a good fire-resistant aggregate [33].

The variations in the compressive strength of the mortars investigated with the cooling mode conditions are graphically represented in Figures 7-9. It is clear that the compressive strength of the mortars exposed to fire is noticeably affected by the cooling conditions. A remarkable reduction in compressive strength was observed for the mortars cooled in air rather than cooled in a furnace or in water. This may be due to the long-term exposure to atmospheric conditions that results in the transformation of the liberated active calcium oxide (CaO) to calcium carbonate (CaCO₃). This is called the carbonation process, and it is the reversible process of the thermal decomposition of calcium carbonate. In water cooling, the dehydrated or decomposed phases containing specimens may react with water quickly and transform into CSH gel again. At a higher temperature > 500 °C the unhydrated phases...
displayed the formation of inner and outer hydrated products to form dense masses of hydration products having micro and narrow pores, without the formation of micro-cracks [33].

The XRD patterns of mortars made of sand and GCB as a fine aggregate are graphically illustrated in Figs. 10-12. The XRD patterns of the sand mortars are shown in Figure 10. Figure 10 shows the presence of the peaks of hydrated products such as calcium carbonate, calcium silicate hydrate and calcium hydroxide as well as the quartz phases. The intensity of the CSH peak was decreased with the firing as a result of the thermal decomposition. The intensity of the peaks characteristic of the calcium hydroxide (CH) phase increased in the mortars cooled in water versus those mortars cooled in air and/or in a furnace, due to the transformation of the calcium oxide to calcium hydroxide in the presence of water. The patterns also showed an increase in calcium carbonate (CC) peaks, especially in mortars cooled in air and in a furnace versus those mortars cooled in water. This phenomenon can be used to rationalize the formation of cracks and the reduction in the compressive strength of mortars cooled in air and in a furnace as compared to those cooled in water. Figures 11 and 12 show the XRD patterns of the mortars containing sand and GCB. Evidently, the pozzolanic reaction of GCB (active pozzolana) with lime leads to the formation of a calcium silicate hydrate (CSH) phase. Therefore, the peak area of calcium hydroxide (CH) decreases with increasing additions of GCB. The intensity of the CSH peak increased with the GCB content, especially in mortars cooled in water versus those mortars cooled in air or in a furnace.

The SEM images of cement mortars containing GCB:sand; 1:2 thermally treated at 700°C and then subjected to water, furnace and air cooling are illustrated in Figure 13, where the morphological differences in the microstructure may be observed. Figure 13A shows the presence of the surface morphology of the CSH gel, which forms around cement mortar particles. Meanwhile, it may be observed that the surface morphology of the hydration microstructure is due to the fast formation of the hydration products. The rehydrated structure is rebuilt quickly during the water cooling. The micrograph displays products having micro and narrow pores, without the formation of microcracks (Figure 13B).

Figure 13C shows a micrograph of the thermally treated cement mortars (GCB: Sand; 1:2) at 700°C, which were then subjected to air cooling. The formation of the massive changes in the morphology affected the cement mortar with the formation of microcracks and voids.

Figure 14 shows the SEM of cement mortars thermally treated at 700°C and then subjected to air cooling. From the micrograph (Figure 14A (GCB: Sand, 2:1)) it is seen that the cement mortar becomes denser due to the sintering or coarsening of the pore size distribution. The crystallization of some dehydrates such as wollastonite and gehlenite confirmed the formation of additional CSH hydrates from the pozzolanic reaction [33]. These hydrates decomposed to form monocalcium silicate (wollastonite), especially in the presence of GCB. The formation of the massive changes in the morphology affected the cement mortar with the formation of small microcracks and voids. Therefore, the observed loss in the strength of the cement
mortality subjected to air cooling takes place at 700 °C as shown in Figs. 3-5. The microcracks ranged between 12.96–15.92 µm [Figure 14B (GCB:Sand, 0:3)].

**CONCLUSION**

In this paper a series of experiments was performed to investigate the effect of three different cooling conditions, i.e., water, air, and furnace cooling, on the mechanical properties of GCB and sand mortars exposed to fire at 700 °C for 3h. In the mortars investigated, three different cement contents (350, 400, 450 kg/m³) were used. Based on the experimental results presented in this paper, the following conclusions may be drawn:
1. The cement contents showed a considerable effect on the compressive strength of the mortars after their exposure to the fire and cooling conditions.
2. The compressive strength of the GCB and sand mortars increased by increasing the cement content from 350–400 kg/m² and then decreased by increasing the cement content up to 450 kg/m².
3. The compressive strength of all the mortars investigated is remarkably affected by the cooling condition.
4. A considerable decrease in compressive strength was observed for the mortars cooled in air versus those cooled in a furnace or in water.
5. SEM of cement mortars thermally treated at 700 °C and then subjected to air cooling for the GCB:Sand, 2:1 mortars. It becomes denser due to the sintering or coarsening of the pore size distribution. The crystallization of some dehydrates (wollastonite and gehlenite) confirmed the formation of additional CSH hydrates from the pozzolanic reaction with the formation of small microcracks and voids. On the other hand, the loss in the strength of the cement mortar subjected to air cooling takes place at 700 °C, which is due to the microcracks ranging between 12.96–15.92 µm for the GCB:Sand, 0:3 mortars.

REFERENCES


