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Ceramics International 38 (2012) 201–209

CERAMICS INTERNATIONAL

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Fire resistance of fired clay bricks–fly ash composite cement pastes

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Received 17 April 2011; accepted 25 June 2011
Available online 6th July 2011

Abstract

This work aims to study the effect of substitution of fly ash for homra on the hydration properties of composite cement pastes. The composite cements are composed of constant proportion of OPC (80%) with variable amounts of fly ash and homra. The addition of fly ash accelerates the initial and final sitting time, whereas the free lime and combined water contents decrease with fly ash content. The fly ash acts as nucleation sites which may accelerate the rate of formation of hydration products which fill some of the pores of the cement pastes. The fire resistance of composite cement pastes was evaluated after firing at 250, 450, 600, 800 °C with rate of firing 5 °C/min with soaking time for 2 h. The physico-mechanical properties such as bulk density and compressive strength were determined at each firing temperature. Moreover, the phase composition, free lime and microstructure for some selected samples were investigated. It can be concluded that the pozzolanic cement with 20 wt% fly ash can be used as fire resisting cement.

Keywords: Ground clay brick; Fly ash; Kinetic hydration; Heat-treatment; Phase composition

1. Introduction

Pozzolanas are natural or artificial and can define as siliceous and aluminous materials, which possess little or no cementitious properties. But, in a finally divided form and in the presence of moisture, they react with lime to form calcium silicate and calcium aluminate silicate hydrates [1–3]. The most important are pulverized-fuel ash (fly ash; pfa), ground granulated blast furnace slag (Ggbfs), natural pozzolanas and microsilica.

The pozzolanic action or reactivity of ashes defined as an reaction of the metastable silicates (SiO_2) and aluminates (Al_2O_3) in fly ash with $Ca(OH)_2$ liberated during the hydration of Portland cement to form insoluble cementitious CSH and calcium aluminate hydrates [4].

Homra is a waste product of about 5–10% produced from the manufacture of clay bricks in Egypt. It shows slow rate and heat of hydration. The utilization of this by-product is widely used to reduce cost, energy and pollution. Homra acts as a good pozzolanic material and filler in filled pozzolanic cement. It shows resistance to aggressive media such as sulphates and chlorides of Portland and sulphate resisting blended cements [5,6].

Wild et al. [7] showed that ground clay bricks calcined at 600–1000 °C exhibit a significant pozzolanic activity equivalent to fly ash. It was generally accepted that artificial pozzolanas such as calcined clays require an optimum of calcination temperature at 800–850 °C to give maximum pozzolanic activity. The firing characteristics of clay are governed by their mineralogical and chemical composition. On calcining kaolin at high temperature, some physico-chemical processes occur leading to changes in these materials [8].

Fly ash is a finely divided waste by-product that precipitated electrostatically from the combustion of pulverized coal at the thermal power plants. It is the most common artificial pozzolana. The fly ash particles are spherical and have the same fineness as cement so that the silica is readily available for reaction [9].

Composite cements are composed of Portland cement and one or more inorganic materials that take part in the hydration reactions and thereby make a substantial contribution to the hydration products [9].

When building materials are subjected to fire and exposed to slow or rapid cooling, some changes may occur in their

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doi:10.1016/j.ceramint.2011.06.050

characteristics, such as phase transformation, weight loss, and aggregated-cement bond, which directly affect its chemical receptivity and mechanical properties [10].

Xu et al. [11] studied the influence of PFA on cracking of concrete and cement paste after exposure to high temperatures. Small cracks were observed after heating up to 250 °C. After exposure to 450 °C or higher temperatures, the cracking of concrete was more significant. The crack density increased almost linearly with temperature up to 650 °C. But significant cracking was observed between 650 and 800 °C. Pure OPC concrete exhibited several major cracks. With the addition of PFA, a fine network of cracks was observed. Higher PFA contents led to more evenly distributed cracks. Lower *w*/*b* ratios resulted in fewer cracks. However, the cracks were wider and longer in comparison with those made with higher *w*/*b* ratios. Concretes suffered losses in compressive strength only when severe cracking occurred after exposure to 450 °C or higher. High temperature exposures induced more damage to the tensile strength of concrete than to the compressive strength.

The aim of the present work is to study the hydration and fire resistance characteristics of homra–fly ash composite cement pastes fired up to 800 °C.

2. Experimental work

The materials used in this work were ordinary Portland cement (OPC) provided from Suez Cement Company and ground clay bricks (homra) from Misr Brick, Egypt. Fly ash was provided from South Africa. The chemical composition of starting materials is shown in Table 1. The fired clay sample constitutes mainly of SiO_2, Al_2O_3, Fe_2O_3, CaO, SO_3, MgO, and Na_2O, K_2O as traces. The mineralogical composition of the fire clay bricks is seen from the XRD pattern in Fig. 1. Homra (GCB) sample constitutes mainly of free silica quartz from added sand and from clays and amorphous aluminosilicate from the decomposition of clay minerals as well as albite and hematite.

The fly ash constitutes mainly of SiO_2, Al_2O_3, F_2O_3, CaO, K_2O, MgO and Na_2O in descending order, in addition to traces of SO_3 and TiO_2. About 0.5% of weight loss is also detected which is mainly due to the elimination of some hydrated and/unburned carbon in the fly ash. The mineralogical composition of fly ash is seen also from the XRD pattern in Fig. 2. The fly ash composes of mullite, quartz (SiO_2) and amorphous aluminosilicate phase. The crystalline and amorphous phases are formed as result of reactions between the major components, i.e. SiO_2 and Al_2O_3 as well as fluxing impurity oxides at high temperature. The hump in the XRD pattern in the range between 15 and 30 2θ is due to the presence of amorphous phase in the fly ash.

The phase composition of OPC is 52.0% alite, 19.20% belite, 8.8% C_3A and 11.1% C_4AF. The surface area determination was made using Blaine apparatus according to ASTM method [12] and the results are shown in Table 1.

The mix composition of the batches is shown in Table 2; the homra–fly ash composite cement is prepared by the substitution

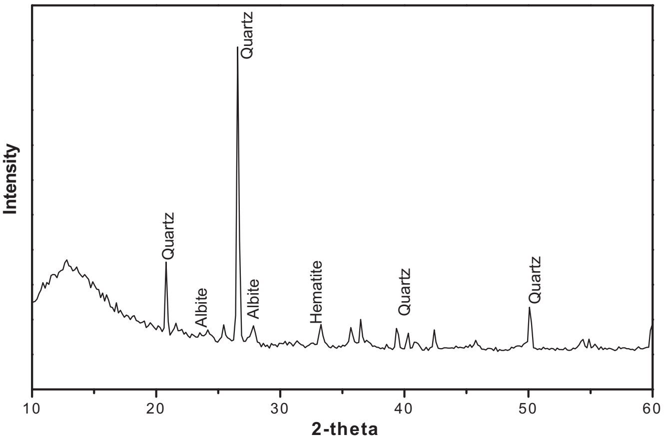


Fig. 1. XRD pattern of clay bricks (homra).

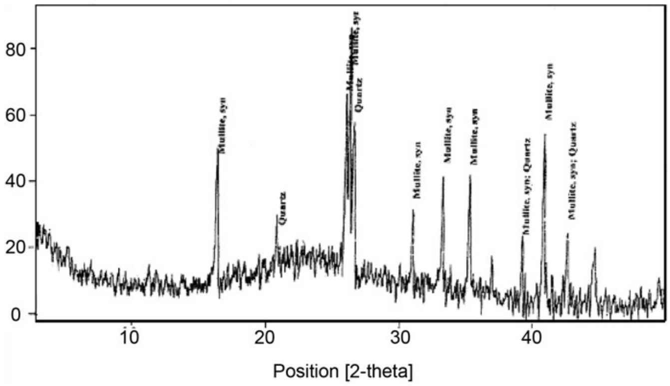


Fig. 2. XRD pattern of fly ash.

Table 1
Chemical analysis, wt% and Blaine surface area, cm^2/g of starting materials.

Oxides	Material		
	Homra	Fly ash	Ordinary Portland cement
SiO_2	69.68	51.36	20.35
Al_2O_3	11.69	36.31	3.75
Fe_2O_3	10.18	3.27	3.63
CaO	2.14	4.46	60.82
MgO	1.71	0.77	1.64
SO_3	2.12	0.33	3.68
K_2O	0.28	0.49	0.16
Na_2O	0.68	0.13	0.42
TiO_2	1.07	1.84	–
P_2O_5	–	0.58	–
I.L.	0.11	0.54	4.31
Specific surface area cm^2/g	3750	4500	3488

Table 2
Mix composition of the investigated mixes, wt%.

Sample no.	OPC	Homra	Fly ash
C.0	80	20	0
C.1	80	15	5
C.2	80	10	10
C.3	80	5	15
C.4	80	0	20

of 20 wt% homra with fly ash. The dry constituents were mixed in a ball mill with four balls for 1 h to attain complete homogeneity. The samples were kept in airtight containers until the time of paste preparation.

The water of normal consistency, the initial and final setting times were determined according to ASTM methods [13], using Vicat Apparatus. The physico-chemical characteristics of composite cement pastes such as combined water, free lime contents [14] as well as insoluble residue contents, bulk density and compressive strength [15] were also measured. Some selected hydrated samples were investigated using a differential thermal analyzer of the Type Schimaduz DTA-50 thermal analyzer (Co-Kyoto, Japan). A sample of 50 mg was housed in a small Pt–Rh crucible with a heating rate of 10 °C/min using α-Al_2O_3 as a reference sample. The interpretation of the thermal charts was based on the data given by [3].

Heat treated mixes were prepared using the standard water of consistency of cement paste. The samples were demoulded after 24 h, cured for 28 days under tap water, dried for 24 h at 105 °C, then subjected to thermal treatment for 2 h at 250, 450, 600 and 800 °C with rate of heating 5 °C/min, then cooled to room temperature in the furnace. Bulk density and compressive strength of treated cement pastes were determined after immersing the pastes overnight under kerosene [16]. The free lime content of heat treated cement pastes is determined with ethylene glycol [14].

The crystalline phases in the specimens were identified using XRD technique of BRUXER, Axs D8 ADVANCE A8, and GERMANY Diffractometer. The samples were finely ground to pass a 200-mesh sieve. The identification of all samples was conformed by computer-aided search of the PDF database obtained from the Joint Committee on Powder Diffraction Standards-International Center for Diffraction Data (JCPDA-ICDD), 2001. The microstructure of the fired samples was studied by using SEM. SEM examination was done on freshly fractured specimens, coated with a 300–400 Å layer of gold, using a JEOL Apparatus with high resolution scanning electron microscope [3].

3. Results and discussion

3.1. Hydration of ground clay bricks–fly ash composite cement pastes

The water of consistency, initial and final setting times of the composite cement pastes are graphically represented in Fig. 3. The water of consistency decreases with the amount of substituted fly ash due to the presence of some crystalline phases in fly ash, and the spherical shape of most fly ash particles permits greater workability than with conventional concrete mixes. The increased ratio of solids to water volume produces a paste with improved plasticity and more cohesiveness and also the presence of unburned carbon which acts as lubricant then decreases the water of consistency. It is clear that substitution of homra with fly ash somewhat retards the initial setting time up to 10% FA. This is due to the slight pozzolanic activity of fly ash at early ages and its coating on the cement grains as well as the formation of ettringite [14]. As homra content decreases up to complete substitution the initial and final setting times are accelerated due to the decrease of water of consistency of cement pastes and the filling effect of fly ash than homra.

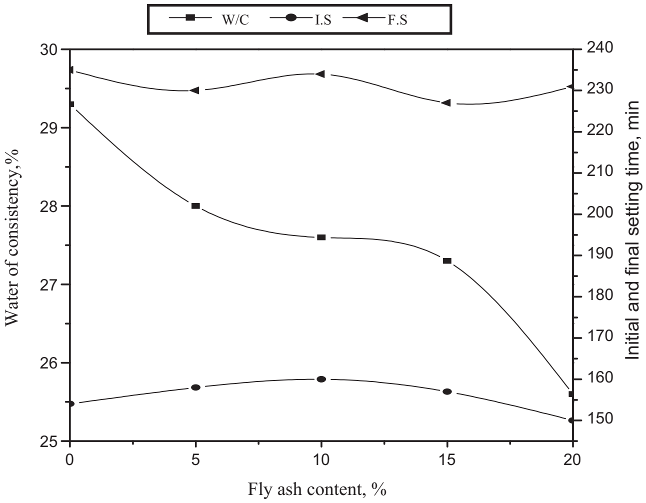


Fig. 3. Water of consistency, initial and final setting time of composite cement pastes made with fly ash and homra.

The combined water content of composite cement pastes is graphically represented up to 90 days in Fig. 4. The combined water content increases gradually with curing time for all cement pastes due to the progress of hydration and increasing of hydration products. It is clear that the combined water decreases with fly ash content. This is mainly due to the low water of consistency of cement pastes and fly ash is composed of some crystalline phases which decrease the hydration characteristics [18,19], as well as the coating of fly ash particles on the cement grains [17]. On the other side, cement paste with 10 wt% fly ash gives higher value of combined water than all composite cement pastes. This is mainly due to the presence of two pozzolanic materials which act as nucleating agent [20], that increase the rate of hydration of cement and

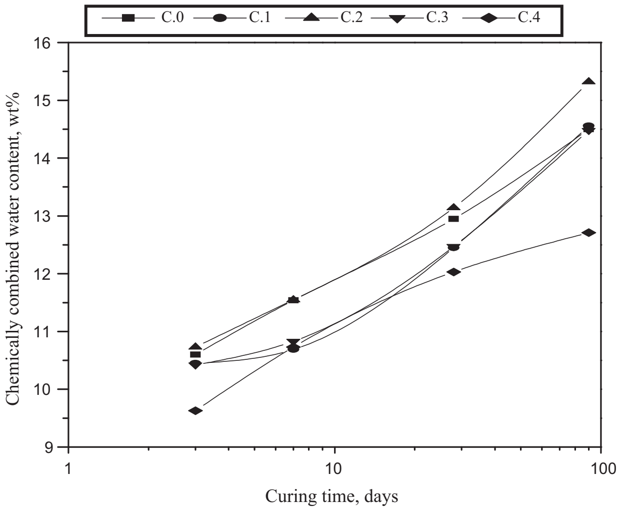


Fig. 4. Chemically combined water of composite cement pastes up to 90 days.

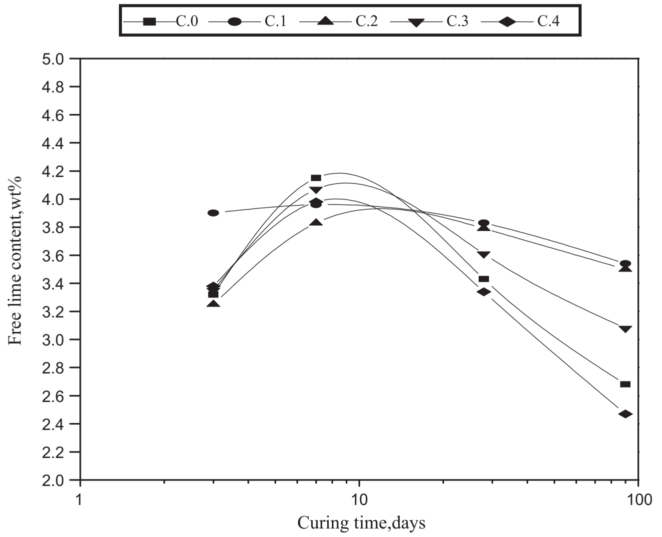


Fig. 5. Free lime content of composite cement pastes up to 90 days.

pozzolanic reaction of homra as well as fly ash to form more hydration products.

The free lime contents of cement pastes are represented up to 90 days in Fig. 5. The free lime content of cement pastes increases with curing time up to 7 days then decreases up to 90 days. This is due to that the rate of liberation of free lime during the hydration of cement pastes exceeds the rate of consumption up to 7 days. After 7 days the rate of consumption is higher than its liberation. On the other side, the increase of substitution of homra to 20 wt% fly ash decreases the free lime up to 90 days. This is due to that fly ash has higher pozzolanic activity than homra to react with liberated lime to form more hydrates than homra.

Fig. 6 illustrates the DTA thermograms of OPC with 20 wt% homra and composite cement pastes (C.3, C.4) cured up to 28 days. The DTA thermograms show the occurrence of endothermic peaks at 90, 110, 160, 360, 473 and 759 °C. The endotherms below 250 °C are mainly due to the dehydration of interlayer water of CSH (tobermorite-like phase) as well as calcium sulphoaluminate hydrate (ettringite or monosulphate) as well as C_4AH_{13}. The fourth endothermic peak at 360 °C is attributed to the decomposition of hydrogarnet, whereas the endotherm at 480 °C represents the dehydroxylation of $Ca(OH)_2$. The endotherm at 759 °C is related to the decomposition of $CaCO_3$. The addition of fly ash increases the peak areas of CSH, CAH and CASH due to the pozzolanic reaction between liberated lime and homra forming hydration products. The peak area of portlandite in the case of C.4 with 20% fly ash is smaller than that of C.3 with 15% fly ash and 5% homra. This means that fly ash is more hydraulic than homra. The peak area of portlandite in OPC is higher than that of pozzolanic cement pastes due to the consumption of free lime with pozzolana.

The insoluble residue contents of homra–fly ash composite cement pastes are graphically represented up to 90 days in Fig. 7. It is clear that the insoluble residue content decreases with curing time as well as fly ash content for all cement pastes, due to the high reactivity of fly ash. The insoluble residue of cement pastes is slowly decreased up to 28 days and then sharply up to 90 days. This is mainly due to the poor pozzolanic activity of fly ash and homra at early ages of hydration from the coating of fly ash particles on the cement grains as well as the formation of ettringite [17] and the high content of crystalline phases of the fly ash which act as filling material [18,19]. The mix C.4 (20% fly ash) shows higher rate of hydration especially at later ages of hydration (90 days).

The bulk density of homra–fly ash composite cement pastes is represented up to 90 days in Fig. 8. The bulk density increases with curing time as well as fly ash content. This is due to the relatively low water of consistency of the fly ash pastes, and its

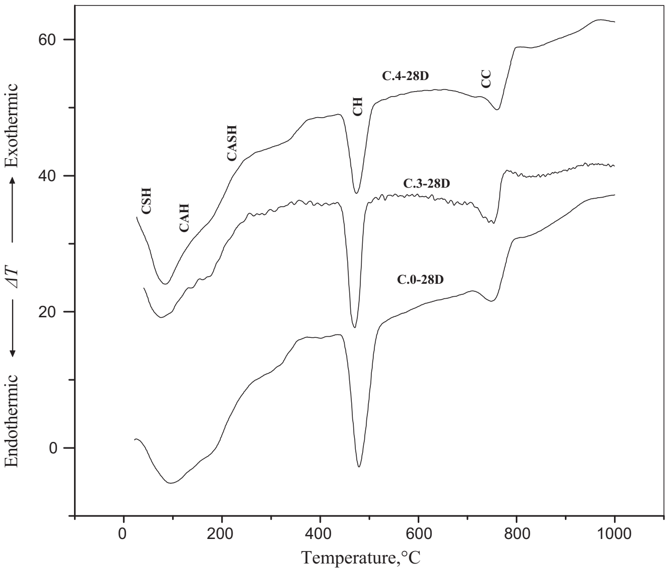


Fig. 6. DTA thermograms of OPC with 20 wt% homra and composite cement pastes C.3, C.4 cured up to 28 days.

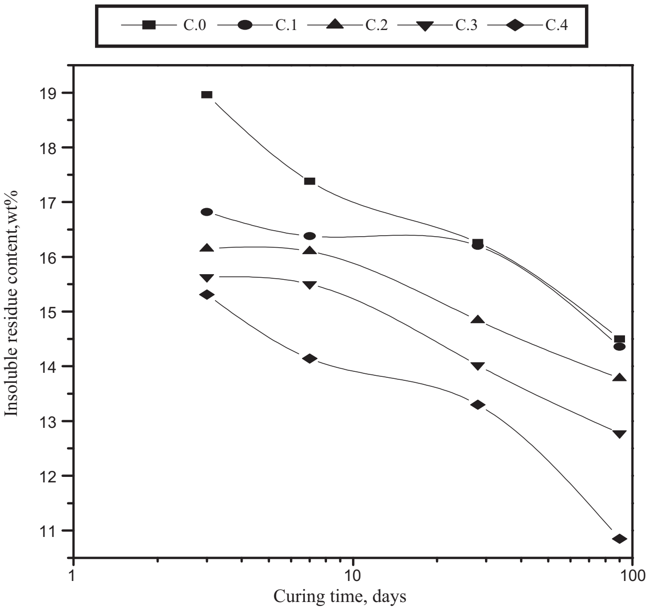


Fig. 7. Insoluble residue contents of composite cement pastes of C.0, C.1, C.2, C.3 and C.4 up to 90 days.

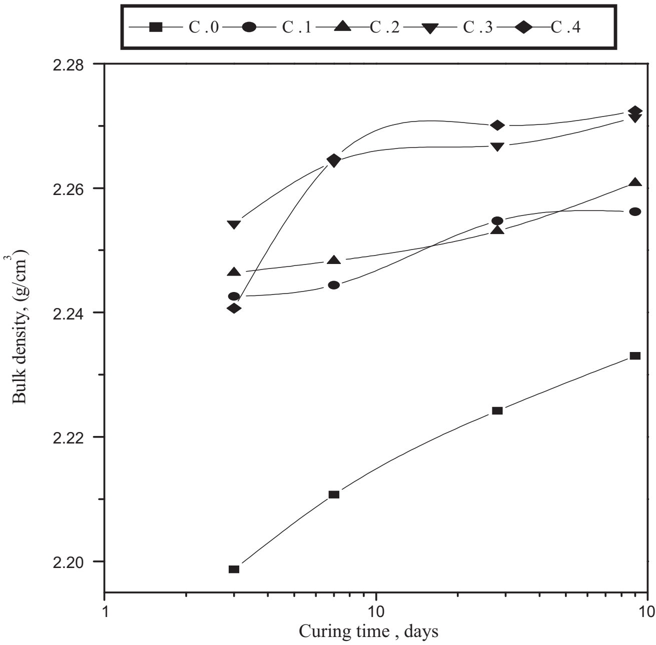


Fig. 8. Bulk density of composite cement paste up to 90 days.

higher pozzolanic activity than homra which lead to form more hydration products. Therefore, the bulk density increases and the total porosity decreases. The mix C.4 (with 20 wt% fly ash) shows higher bulk density than the other mixes. This is due to the relatively low water of consistency of the fly ash pastes, and acceleration the rate of hydration forming more hydration products.

The compressive strength of homra–fly ash composite cement pastes is represented up to 90 days in Fig. 9. The compressive strength of cement pastes increases up to 90 of hydration. This is attributed to the formation of hydration products especially CSH gel and its later accumulation within the available pore spaces giving more strength. The CSH phase is the most cementitious or binding property to the final product (by Van der Waal force of attraction) and hence contributes to the strength of cement paste. The compressive strength increases with fly ash content up to 15% and then decreases at 20% fly ash at early ages due to the slow rate of pozzolanic activity of fly ash from the effect of unburned carbon [21]. On the other side, it gives strength higher at later ages of hydration. This means that fly ash is more pozzolanic than homra due to the high content of combined silica as compared with homra.

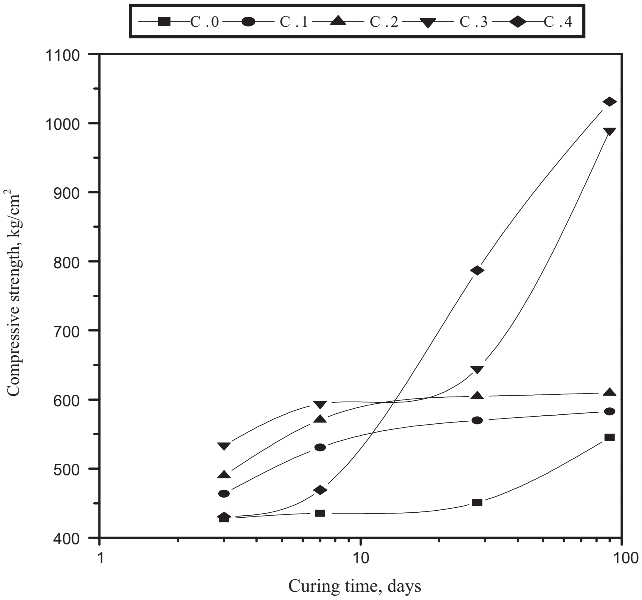


Fig. 9. Compressive strength of composite cement pastes of C.0, C.1, C.2, C.3 and C.4 up to 90 days.

3.2. *Effect of treated temperature on clay bricks–fly ash cement pastes*

The bulk density of composite cement pastes (C.0, C.1, C.2, C.3 and C.4) as a function of treatment temperature from 105 °C up to 800 °C is represented in Fig. 10. The bulk density increases with 5% fly ash and then decreases. This is mainly due to the presence of two pozzolanic materials which act as a nucleating agent [20] that increases the rate of hydration of cement and pozzolanic reaction of homra, fly ash and lime to form more cementitious materials that fill some of the pores. The bulk density of cement pastes decreases from 105 °C up to 250 °C, due to the removal of free, adsorbed and combined water leaving pores. From 250 °C up to 450 °C, the bulk density increases due to increase the degree of crystallinity of the formed hydrates leading to increase the bulk density. As the temperature increases up to 800 °C, the bulk density increases for all composite cement pastes due to that the homra and fly ash react with lime to form extra cementitious materials. Pastes C.1 (5% FA) and C.3 (20% FA) give the higher values of the bulk density at 800 °C. This is the optimum composite cement which leads to form more hydration products.

The compressive strength of homra–fly ash cement pastes is represented as function of temperature in Fig. 11. The compressive strength of cement pastes increases up to

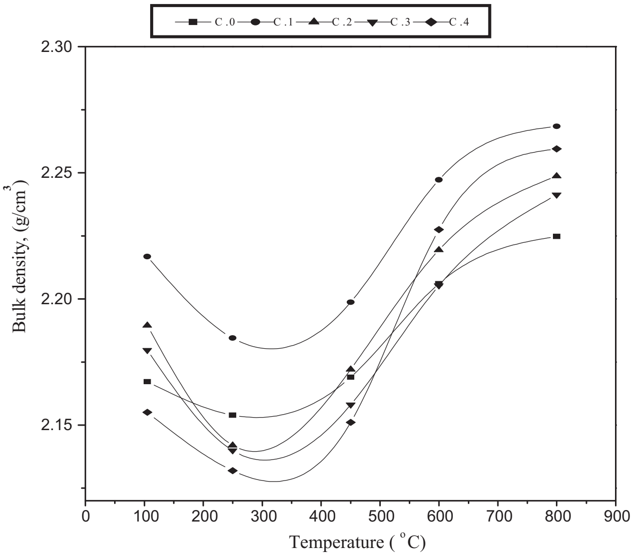


Fig. 10. Bulk density of composite cement pastes C.0, C.1, C.2, and C.3 and C.4 with treatment temperature up to 800 °C.

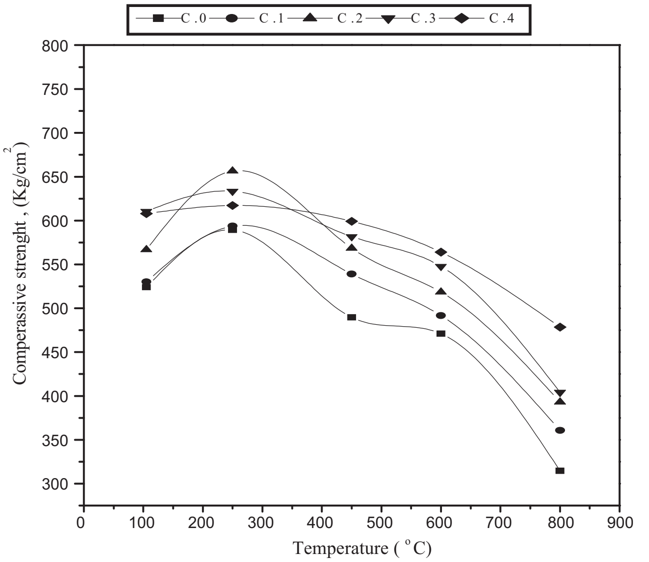


Fig. 11. Compressive strength of composite cement pastes C.0, C.1, C.2, C.3, and C.4 with treatment temperature up to 800 °C.

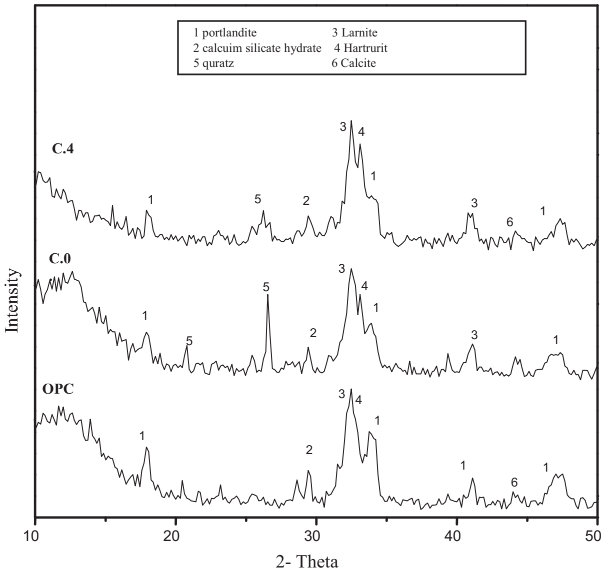


Fig. 13. XRD patterns of hardened pozzolanic cement pastes made from OPC with 20 wt% homra as well as 20 wt% fly fired at 800 °C.

250 °C, as a result of enhancing the hydration of unhydrated cement clinker [22], and pozzolanic reaction to form extra CSH with low Ca/Si ratio [23] which gives higher strength. The steam produced from elimination of capillary, physically adsorbed, interlayer and bound water in CH and CSH increases the pressure in the pastes and forms internal autoclaving reaction of homra as well as fly ash [24,25]. This leads to the formation of more hydration products which fill some of pores, and increase the bond strength. The compressive strength increases with fly ash content due to its higher pozzolanic activity than homra to react with liberated lime. The compressive strength decreases with treatment temperature up to 450 °C, due to the decomposition of cementitious materials, and decomposition of $Ca(OH)_2$, which losses its water above 450 °C, and increases the porosity. The relatively sharp reduction in the compressive strength at 600 °C is due to the thermal decomposition of some binding hydration products such as calcium silicate hydrates, and swelling of the water layer in the cement pastes [9,26]. The mix C.0 with 20% homra shows the lower compressive strength due to increase the water/cement ratio. On the other hand, the compressive strength of the mix C.4 (20%FA) is higher than all mixes due to that fly ash has higher pozzolanic activity than homra. The increase of the temperature up to 800 °C, the compressive strength decreases. It can be included that 20 wt% fly ash with OPC has a higher resistance to fire than all cement pastes.

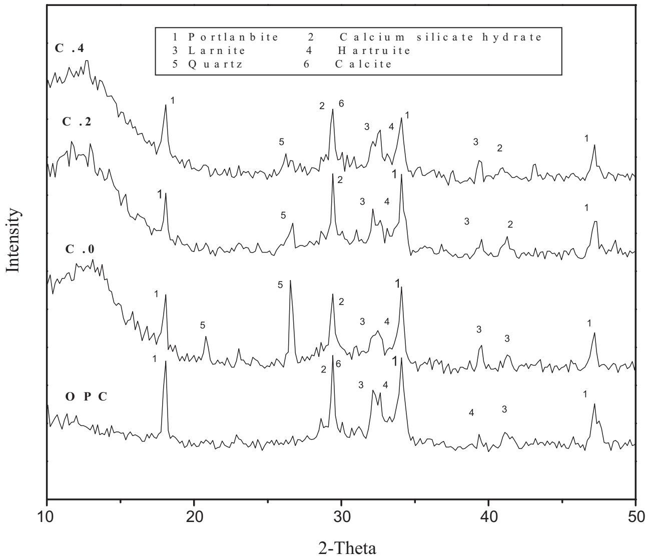


Fig. 12. XRD pattern of OPC and composite cement pastes made from 20 wt% homra and 10 wt% fly ash with 10 wt% homra as well as 20 wt% fly ash fired at 450 °C.

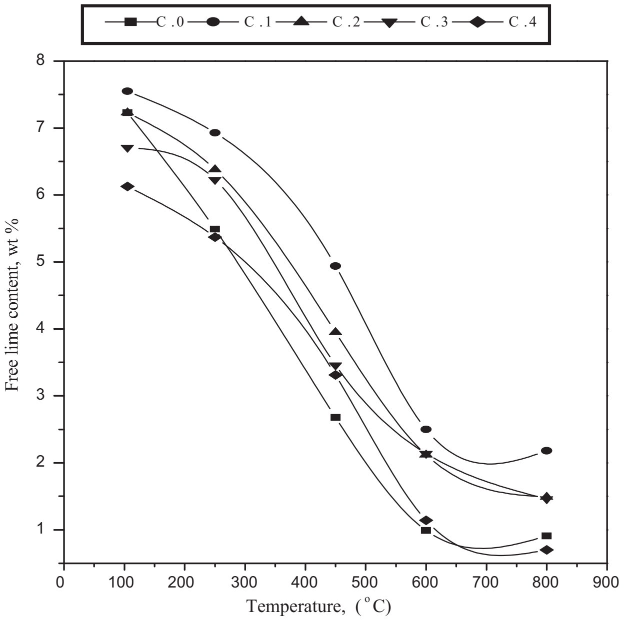
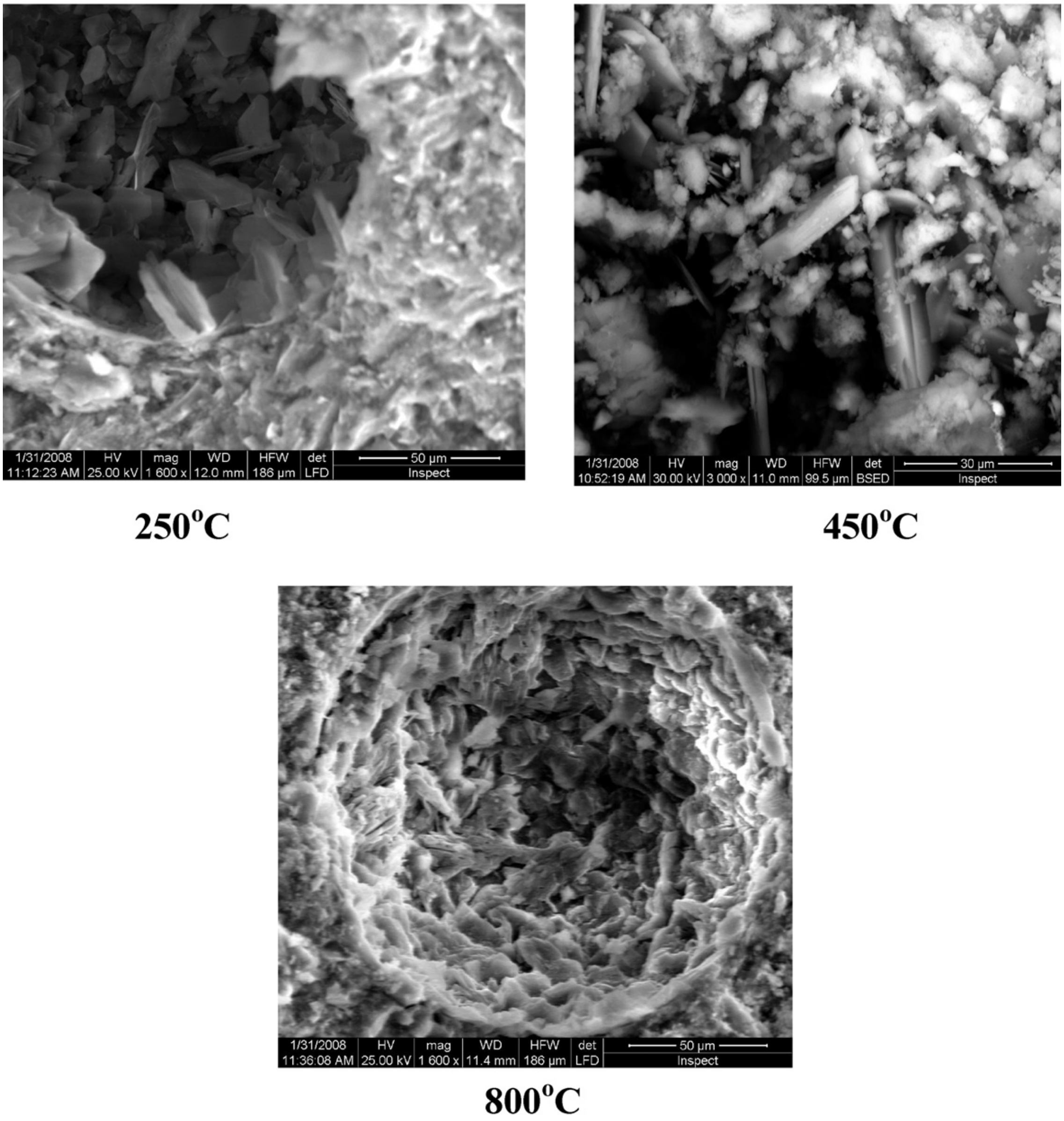


Fig. 14. Free lime content of composite cement pastes with treatment temperature up to 800 °C.

The XRD patterns of the hardened OPC and composite cement pastes C.0, C.2 and C.4 fired at 450 °C are shown in Fig. 12. The results indicate that the intensity of CH peak decreases as result of substitution of OPC with 20% homra as well as 10% and 20% fly ash. The intensity of quartz decreases with fly ash content and decreasing homra content. The cement paste with 20% homra shows silica quartz and decreases with fly ash due to the addition of quartz sand in clay brick making. The intensity of calcium silicate minerals as larnite (β-C_2S) and harturite (C_3S) increases due to the reaction of fly ash with lime forming additional CSH gel which fills some of open pores enhancing the fire resistance of hardened cement pastes.

The XRD patterns of OPC and composite cement C.0 and C.4 fired at 800 °C are shown in Fig. 13. It is clear that, the intensity of portlandite in fly ash shows the lower value than that of homra and OPC. The CSH gel is nearly completely disappeared due its transformation to larnite (β-C_2S) and harturite (C_3S) [27,28]. The decrease of CH and the presence of higher amounts of calcium silicate hydrate are evidence that fly ash has higher pozzolanic activity in comparison with homra. The fly ash consumes CH forming additional calcium silicates which fill some of open pores enhancing the fire resistance of hardened cement pastes. The presence of portlandite at 800 °C is mainly due to the rehydration of some decomposed portlandite as well as $CaCO_3$. This is in a good agreement with the free lime results of the fired samples. The rate of reaction of fly ash with liberated lime decreases with fly ash content due to the presence of unburned carbon which decreases its pozzolanic activity.

The free lime contents of cement pastes are represented as a function of thermally treated temperature up to 800 °C in Fig. 14. The free lime content decreases with temperature and increases with fly ash content up to 10 wt%, due to that 5–10 wt% fly ash

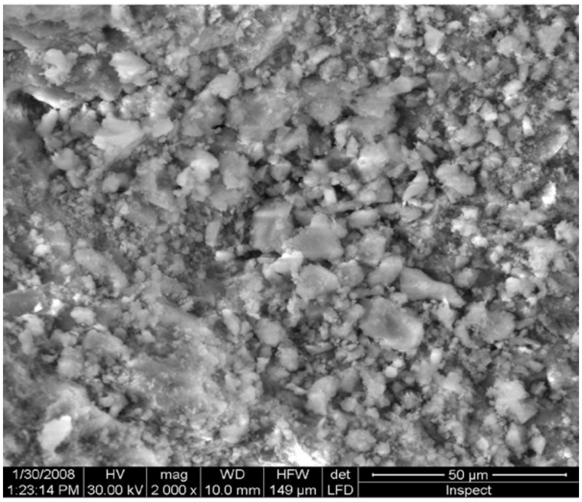


250°C

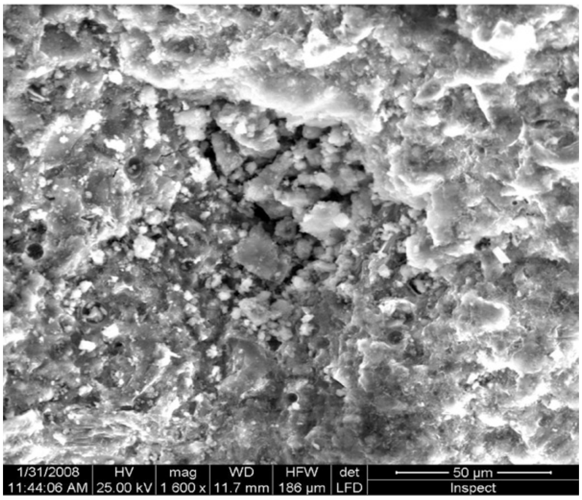
450°C

800°C

Fig. 15. The SEM of OPC thermally treated at 250, 450and 800 °C.



20 % Homra fired at 450°C



20% FA fired at 450°C

Fig. 16. The SEM of pozzolanic cement pastes made from OPC with 20 wt% homra as well as 20 wt% fly fired at 450 °C.

act as nucleating agent that increase the rate of hydration [20]. The rate of reaction of fly ash with liberated lime decreases with fly ash content due to the presence of unburned carbon. Also, the free lime of hydrated cement pastes decreases up to 250 °C as a result of enhancing the hydration of unhydrated cement clinker [22] and the pozzolanic reaction to form extra CSH with low Ca/Si ratio [23]. The steam produced from elimination of capillary, physically adsorbed, interlayer and some of bound water in CH as well as CSH increases the pressure in the pastes and forms internal autoclaving reaction between the liberated lime and fly ash [24]. As the temperature increases up to 800 °C, the free lime decreases due to the decomposition of $Ca(OH)_2$. The presence of portlandite at 800 °C is mainly due to the rehydration of lime (CaO) from the decomposition of portlandite as well as $CaCO_3$.

High-resolution SEM of hydration products of heat treated OPC pastes at 250, 450 and 800 °C is shown in the micrograph in Fig. 15. It shows the SEM of OPC pastes heat treated at 250 °C. CSH is well formed as rod like crystals of CSH. This is formed due to the heat treatment at this temperature which accelerates the formation of CSH as in the autoclaving condition. The $Ca(OH)_2$ appears as sheets stacked as parallel layers. As the heat treatment temperature increases the presence of $Ca(OH)_2$ phase as thin plates of hexagonal symmetry decreases. Also, lath-lick rods of crystals of CSH well formed and addition to anhydrous β-C_2S at 450 °C. The micrograph of OPC paste heated at 800 °C shows the formation of massive structure in the microstructure. It is clear that the crystallinity of CSH as well as $Ca(OH)_2$ decreased due to the decomposition of CSH and CH at higher temperature. There are some pores at 800 °C.

Fig. 16 shows the SEM micrograph of the pozzolanic cement pastes made from OPC with 20 wt% homra (C.0) as well as 20 wt% fly ash (C.4) heat treated at 450 °C. The sample fired at 450 °C illustrates the formation of some hexagonal $Ca(OH)_2$ in addition to lath-lick rods of CSH. The matrix in fly ash pozzolanic cement paste is denser than OPC only and with homra. This is due to the higher reaction of CH with the fly ash than homra. This leads to formation more crystalline products, which fill some pores.

4. Conclusions

When fly ash is substituted instead of homra the water of consistency decreases with the amount of fly ash, the initial and final setting times are accelerated. The combined water decreases with the fly ash content due to the low water of consistency of cement pastes. The free lime content of cement pastes increases with curing time up to 7 days then decreases up to 90 days. The substitution of homra to 20 wt% fly ash decreases the free lime up to 90 days due to that fly ash has higher pozzolanic activity than homra. The compressive strength increases with fly ash content up to 15% substitution and then decreases at 20% fly ash. 20 wt% fly ash gives lower strength at early ages and higher at later ages due to presence of some unburned carbon.

The effect of fire on the mechanical properties as well as phase composition of composite cement pastes was carried out by the determination of bulk density, free lime content and compressive strength of heat treated hardened cement pastes treated up to 800 °C. The mineralogical composition, X-ray diffraction and microstructure for some selected samples were also studied using SEM technique.

Acknowledgment

The authors would like to thank Dr. Randa M. Osman researcher in Department of Chemical Engineering, National Research Centre, Dokki, Cairo, Egypt, for her kind help and continuous interest throughout the research work.

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