
EFFECT OF CALCINATION TEMPERATURE OF KAOLINITE CLAY ON THE PROPERTIES OF PORTLAND CEMENT PASTES CONTAINING METAKAOLIN

M.A.TAHER, A.Y.EL-SAYED , O.A.FARGHALY AND M.R.SHATAT

Chemistry Department, Faculty of Science, Al-Azhar University, Assiut, Egypt

Abstract

Metakaolin (MK) is a highly reactive pozzolan produced by calcination of kaolinite clay at high temperatures. It has a high specific surface, which makes it very suitable as a cementing material in concrete. The utilization of area calcined clay, in the form of MK, as a pozzolanic material for mortar and concrete has received a considerable attention in recent years. This interest is part of the widely spread attention directed towards the utilization of wastes and industrial by-products in order to minimize Portland cement (PC) consumption, the manufacture of which being environmentally damaging. Another reason is that mortar and concrete, which contain pozzolanic materials, exhibit considerable enhancement in durability properties. In this investigation, the physico-chemical properties of artificial pozzolanic cement pastes containing MK produced by calcination of kaolinite clay at different temperatures (700, 800, 900, and 1000°C) were studied. PC was partially substituted for by 0, 5, 10, 15 and 20 % of MK by weight at different calcination temperatures. The characteristics of prepared mortars were investigated after curing in water for different periods (3, 7, 28 and 90 days) by determination of compressive strength and total porosity. The hydration kinetics was evaluated by determination of free lime contents. IR spectroscopic analysis was used to investigate the change in structure of mortars after curing. The change in morphology and microstructure of some hardened pastes were investigated by scanning electron microscopy (SEM).

Keywords: Metakaolin, Pozzolan, Kaolin, Blended cement.

Abbreviations: C = CaO; S = SiO₂ ; H = H₂O ; A = Al₂O₃ ; F = Fe₂O₃ ; CH = Ca(OH)₂

Corresponding Author: M.A.Taher

E-mail Address: mahmoudtaher@excite.com

Introduction

Metakaolin (MK) has been introduced recently as a highly active and effective pozzolans for the partial replacement of cement in concrete. It is an ultra fine material produced by the dehydroxylation of a kaolin precursor upon heating in the temperature range of 700–800°C [1]. The calcinations conditions are the main factors, which can modify the reactivity of MK. Reactive MK can be obtained between 700-900°C. On calcining clays up to 1000°C, some physico-chemical

processes occur leading to change in the nature of these materials. Clays loose its adsorbed and chemically-combined water at the temperature range between 100-200°C and 500-700°C, respectively. The dehydration of clays gives an amorphous mixture of Al_2O_3 and SiO_2 (MK). At a temperature lower than 700°C, residual kaolin occurs, whereas above 900°C the crystallization of metakaolin into $\gamma\text{-Al}_2\text{O}_3$ containing silica in its spinal structure starts, which diminishes greatly the reactivity of metakaolin [2]. MK is a silica-based product that, on reaction with $\text{Ca}(\text{OH})_2$, produces CSH gel at ambient temperature. MK also contains alumina that reacts with CH to produce additional alumina-containing phases, including C_4AH_{13} , C_2ASH_8 , and C_3AH_6 [3,4]. The physical and mechanical properties of PC containing MK or combination of MK and slag and the compatibility between such materials and superplasticizers were investigated [5]. After MK was incorporated into PC, the compressive strength of the blended cement was enhanced. An investigation was carried out on a range of concrete mixes with three different proportions of MK to replace ordinary Portland cement [6]. Both the aggregate-binder ratio and the water-binder ratio were varied to yield concrete with a wide range of strength, workability and permeability. The results were used to establish the effect of mix constituents on strength, workability and permeability. It has been found that metakaolin used in different dosages to replace Portland cement changes properties of both the fresh and hardened concrete. It increases the compressive strength and reduces the slump for a given aggregate/binder ratio and water/binder ratio. The effect of using metakaolin as a cement replacement material on air permeability was found to be very marginal .Up to a maximum of 10% replacement level, the sorptivity of concrete is improved for high levels of cement content. The properties and the hydration procedure of cements containing metakaolin were monitored for periods up to 180 days [7]. Four metakaolins, derived from poor Greek kaolins, as well as a commercial metakaolin of high purity were used. Cement mortars and pastes, with 0%, 10% and 20% replacement of cement with the above metakaolins, were examined. Strength development, water demand and setting time were determined in all samples. In addition, XRD and TGA were applied in order to study the hydration products and the hydration rate in cement–metakaolin pastes. It was concluded that metakaolin has a very positive effect on the cement strength after 2 days and specifically at 28 and 180 days. Finally, it was concluded that a 10% MK content seems to be, generally, more favorable than 20%. The stress-strain relationships

(tension and compression) and bend strength measurements for concrete incorporating 0%, 5%, 10% and 15% MK were studied [8]. The test results showed that the tensile strength and peak strain increase with increasing MK content whereas the tensile modulus shows only small changes. Poon et al [9] studied the hydration progress in MK-blended high-performance cement pastes with age from the measurements of compressive strength, porosity, pore size distribution, the degree of pozzolanic reaction, and the CH contents of MK-blended cement pastes at water/binder ratio (w/b) of 0.3. Comparisons are also made with pastes containing silica fume (SF), fly ash (FA) and control Portland cement (PC). It was found that at early ages, the rates of pozzolanic reaction and CH consumption in the MK-blended cement pastes are higher than the SF- or FA-blended cement pastes. The higher pozzolanic activity of MK results in a higher rate of strength development and pore structure refinement for the cement pastes at early ages. Although the rate of pozzolanic reaction of MK becomes slower after 28 days of curing, the pozzolanic reaction in the blended cement pastes with a w/b of 0.3 still continues at the age of 90 days. At this age, about half of the MK still are unreacted. The effect of MK addition on the corrosion resistance of cement mortar was studied [10]. Several mixture proportions were used to produce mortar specimens, where metakaolin replaced either sand or cement. Mortar specimens were then exposed to the corrosive environment of either partial or total immersion in 3.5% w/w NaCl solution. It was concluded that MK improves the compressive strength and the 10% w/w addition shows the optimum contribution to the strength development.

Experimental

Materials:

The materials used in this investigation are Portland cement (Assiut Company, Assiut, Egypt), kaolinite clay (Kalabsha, Aswan, Egypt). The chemical oxide composition of Portland cement and Kalabsha kaolinite clay used in this study is shown in Table (1).

Table (1): Chemical oxide composition of Portland cement and Kalabsha kaolinite clay , (% weight).

Oxide contents	Portland cement	Kalabsha kaolinite clay
SiO ₂	20.60	44.18
Al ₂ O ₃	5.50	36.75
Fe ₂ O ₃	3.75	1.36
CaO	63.55	0.26
SO ₃	2.13	----
MgO	0.78	0.16
Na ₂ O	0.44	0.18
K ₂ O	0.47	0.25
TiO ₂	0.54	2.94
L.O.I	2.19	13.55
Total	99.95	99.63

Methods:*Preparation of calcined clay (metakaolin):*

Kalabsha Kaolinite clay (KK) was dried at 110°C for 48 hours, then crushed and passed completely through 1mm B.S. sieve. The crushed clay was burnt at 700, 800, 900 and 1000°C for a soaking period of 2 hours and then quenched in air. The burnt clay (MK), after air quenching, was subjected to ball mill grinding for 20 minutes and passed through a 75 µm B.S. sieve; the processes of grinding and sieving were repeated until the sample is passed completely through 90 µm.

Preparation of PC- MK specimens:

PC was mixed with four types of MK produced by burning Kalabsha kaolinite clay at 700, 800, 900 and 1000°C. Five dry mixtures were prepared from various proportions of PC and MK with the mass ratios of 100:0(B), 95:5(CI), 90:10(CII), 85:15(CIII), 80:20(CIV) and 75:25(CV), respectively, using ethanol to obtained complete homogeneity of the mixture; then dried overnight at 105°C. Each dry mixture was mixed for three minutes at different workability water/solid ratios by weight. The resulting mass was molded in 1-inch cubic moulds. The moulds were vibrated for one minute to remove any air bubbles and voids. Immediately after moulding, the specimens were cured in humidity cabinet at 100% relative humidity at room temperature for 24 hours in order to attain the setting of the specimens. The specimens then were demolded and cured under tap water for various hydration periods: 3, 7, 28 and 90 days. After the predetermined curing time (3, 7, 28 and 90

days) , groups of three specimens were used to determine the residual compressive strength according to ASTM specification (ASTM, 1992C). Total porosity was determined after each time of hydration as described elsewhere [11]. The hydration of cement pastes were stopped by employing alcohol–acetone method [12]. The samples were dried at 105°C for one hour and then collected in polyethylene bags; sealed and stored in desiccators for analysis. The degree of hydration was followed by determination of free lime [13,14]. The hydration products were analyzed by FTIR spectrophotometric technique using a Perkin-Elmer System 2000 FTIR spectrometer. The morphology and microstructure of some dry samples were investigated using scanning electron microscope (JEOL JSM-840SEM).

Results and discussion

Compressive strength:

The compressive strength of PC-MK specimens at different firing temperatures of KK 700, 800, 900 and 1000 °C are shown in Figs. 1, 2, 3 and 4, respectively. It is clear that the compressive strength values increased continuously with increasing age of hydration. Comparing the strength values of all specimens of mixes CI, CII, CIII, CIV and CV with blank PC specimen (B), it is clear that MK improves the strength of cement mortars at firing temperatures 800, 900 and 1000°C of KK at all ages of hydration. When MK is utilized in cement, the reaction mechanism can be divided to physical and chemical aspects [15,16]. The physical effect is that the ultra-fine particles fill the voids in cement, which makes the microstructure of cement paste denser. The chemical effect is the reaction of MK with the cement hydrates. The reaction of MK with cement hydrates is fast because MK has a loose microstructure after thermal activation at a temperature of 800°C (the chemically bound water in kaolin was driven out at higher temperatures, and water molecules enter MK more easily when met with water again). Mix CI (95%PC : 5% MK) which is richer in PC contents possess higher strength values at all stages of hydration and at all firing temperatures of KK(700, 800, 900, 1000°C) when compared with mixes CII, CIII , CIV and CV. Evidently, the larger PC content in the solid PC-MK mixture gives rise to a larger amount of the cement hydration products upon hydration ; in addition ; a larger quantity of free Ca(OH)_2 will be released as a result of PC hydration, which in turns increases the hydraulic reactivity of MK which absorbs Ca(OH)_2 to produce secondary CSH gel inside the cement paste [15,16]. The secondary formed CSH gel improves the microstructure of cement paste matrix and produce higher compressive strength values of cement

pastes. The compressive strength values decreased gradually by increasing the MK contents in PC-MK pastes at all firing temperatures of KK. This is attributed to the decreasing in released Ca(OH)_2 due to PC hydration, less formed CSH and increase the possibility of replacement of Al for Si in the CSH to form hydrogarnet. The latter phase as well as the consumption of CSH in this reaction decreases the strength of the cured specimens. In addition, the increase in the MK content in PC-MK mixes resulted in an increase in the optimum water of consistency (initial porosity) of the PC-MK pastes; this leads to higher porosity values for the pastes made with MK contents higher than 5% which is associated with a consequent decrease in their strength values.

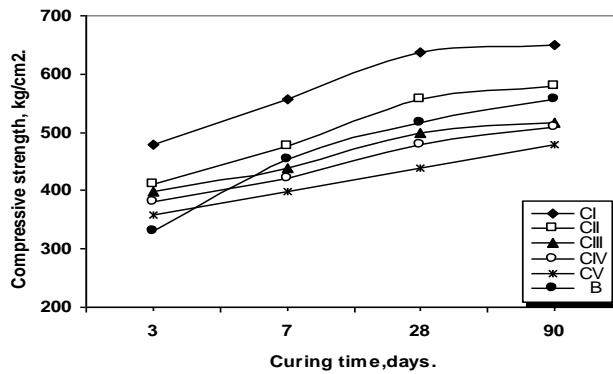


Fig.(1): Compressive strength of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 700°C) as a function of curing time.

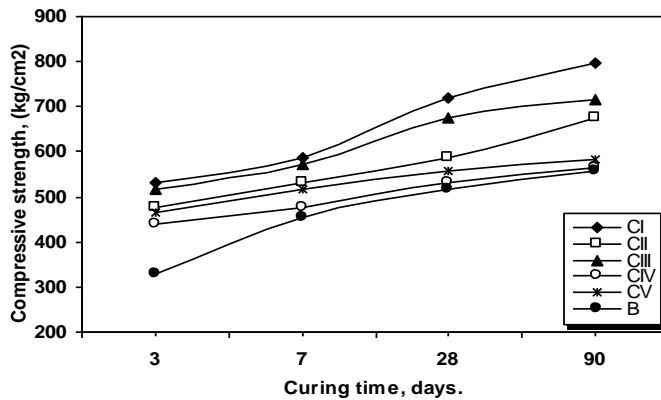


Fig.(2): Compressive strength of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 800°C) as a function of curing time.

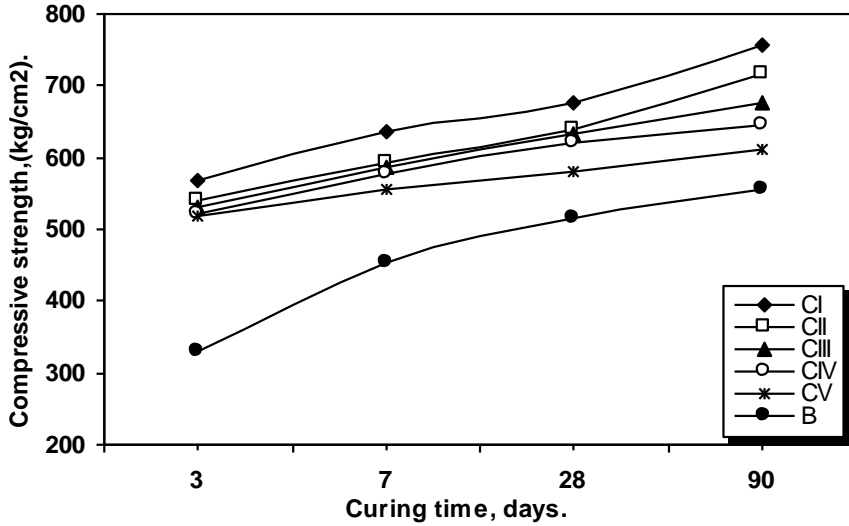


Fig.(3): Compressive strength of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 900°C) as a function of curing time.

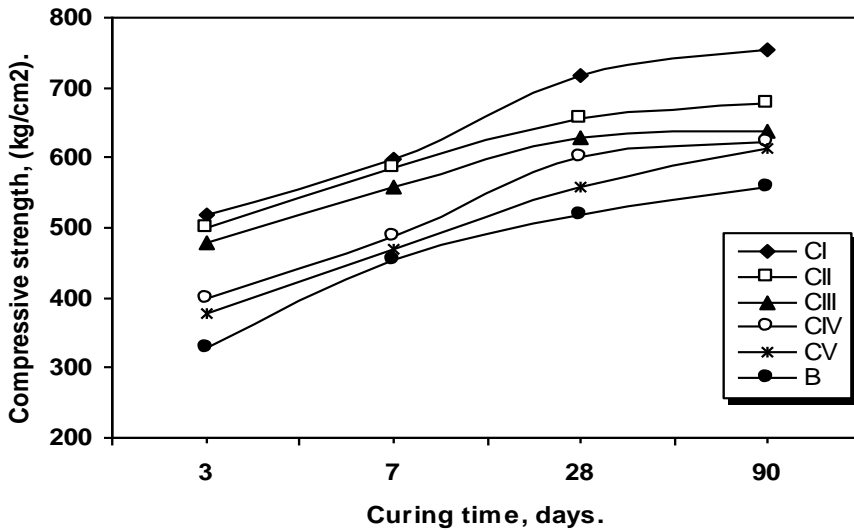


Fig.(4): Compressive strength of hardened specimens (mixes CI-CV) mad from PC and MK (burnt KK at 1000°C) as a function of curing time.

Total Porosity:

The total porosity values of the hardened PC-MK pastes produced by firing KK at different firing temperatures (700, 800, 900 and 1000°C) as a function of curing time are plotted against curing time as shown in Figs.(5, 6, 7 and 8). The results show that for all pastes, the porosity decreased as the curing age increased. The pastes containing MK had lower porosity values than those of control paste (B: 100% PC) at all hydration ages. This indicates that the MK used in this study is effective in the refinement of the pore structure of the blended cement system due to the accumulation of hydration products in the open pores of cement pastes. The cement pastes containing 25% MK (mix CV) showed higher porosity than all mixes (CI, CII, CIII and CIV); this is mainly due to the higher water of consistency (higher initial porosity) of the PC-MK paste having higher MK contents. These present results are however similar to those obtained by Khatib and Wild [17] and Frias and Cabrera [18], who reported higher porosity values were obtained for high MK content pastes in blended cement.

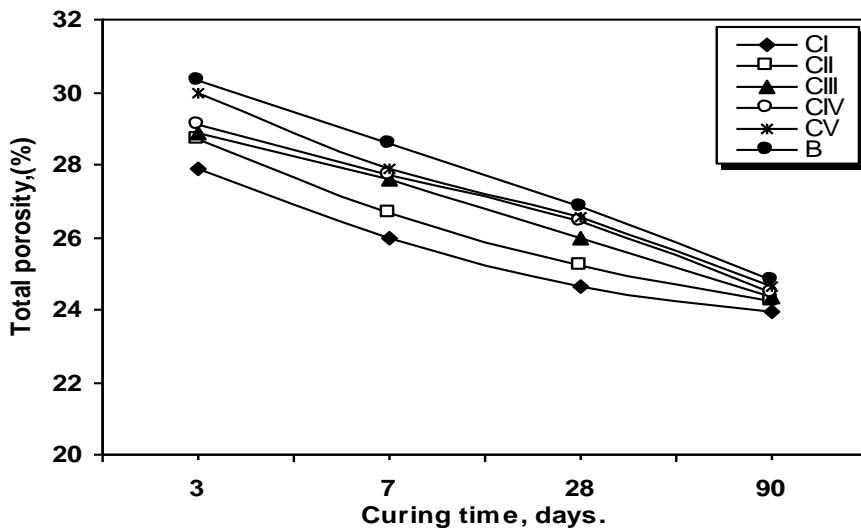


Fig.(5): Total porosity of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 700°C) as a function of curing time.

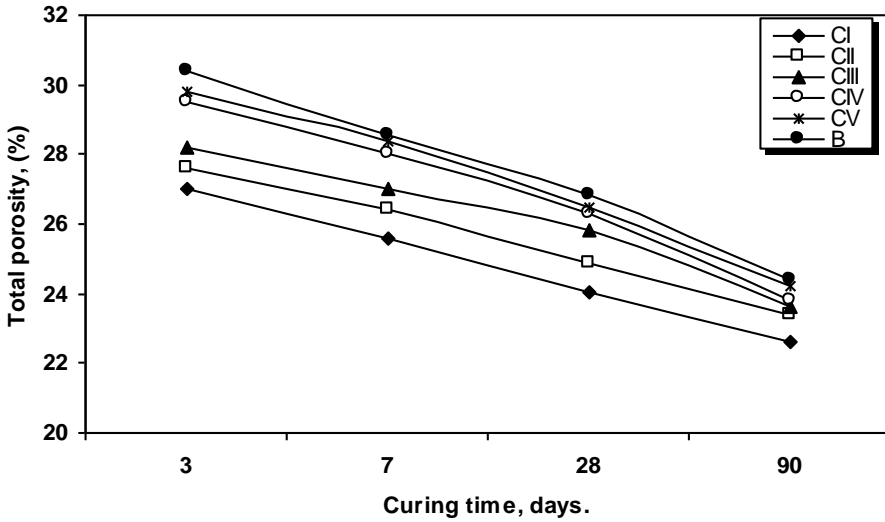


Fig.(6): Total porosity of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 800°C) as a function of curing time.

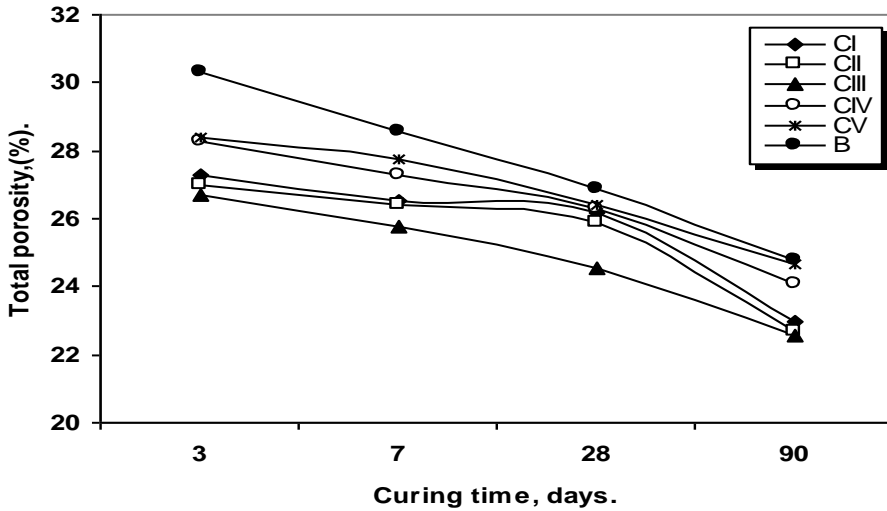


Fig.(7):Total porosity of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 900°C) as a function of curing time.

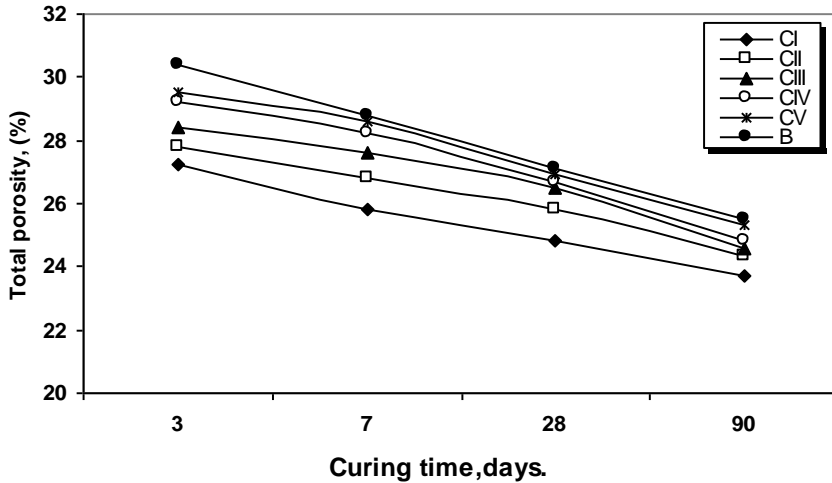


Fig.(8): Total porosity of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 1000°C) as a function of curing time.

Free lime contents:

The free lime contents, CaO%, of the hardened pastes made from the dry PC-MK mixes produced by firing KK at different temperatures (700, 800, 900 and 1000°C) as a function of curing time up to 90 days of water curing are graphically plotted as a function of curing time in Figs.(9, 10, 11 and 12). The determination of free lime gives an idea about the behavior of the solid state reaction between the phases of PC and water as well as between the liberated lime and MK constituents. The detection of free lime for PC pastes is mainly due to the hydration of C_3S and $\beta-C_2S$. Generally, the free lime contents decreases with increasing curing time for all PC-MK pastes and for all firing temperatures of KK. This is mainly attributed to the reaction between the liberated $Ca(OH)_2$ and the active Al_2O_3 and SiO_2 of MK. These minerals are formed as a result of dissociation of shale/clay due to calcinations at different temperatures (700, 800, 900 and 1000°C). It is clear that the free lime contents for pastes containing fired KK at 900 and 1000°C were smaller than pastes containing fired KK at 700 and 800°C which attributed to the activation of KK by increasing firing temperature. The addition of more MK consumes all the amount of liberated lime in the reaction with Al_2O_3 or SiO_2 with water to form hydrated phases of calcium silicate or calcium aluminosilicate. Also, increasing the amount of MK means increasing SiO_2 and Al_2O_3 contents. These oxides may have

chance to react with liberated Ca(OH)_2 to form hydrogarnet which has weaker hydraulic properties. Accordingly, the low hydraulic properties obtained for mixes CIII, CIV and CV in this investigation may be attributed to the formation of hydrogarnet; while the increase in the initial porosity of pastes made of these mixes (higher water of consistency) leads to lower strength values.

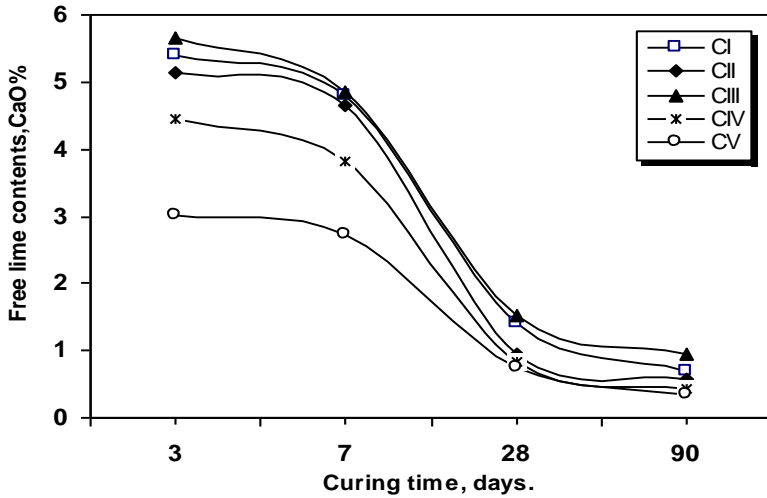


Fig.(9): Free lime contents of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 700°C) as a function of curing time.

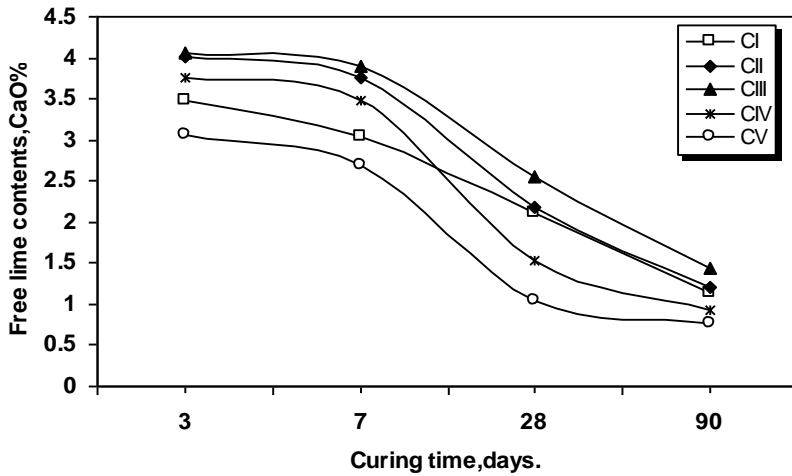


Fig.(10): Free lime contents of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 800°C) as a function of curing time.

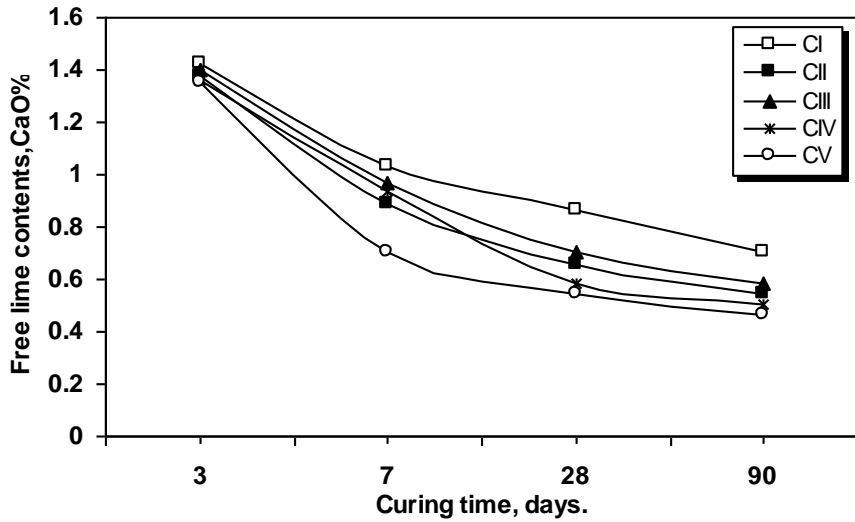


Fig.(11): Free lime contents of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 900°C) as a function of curing time.

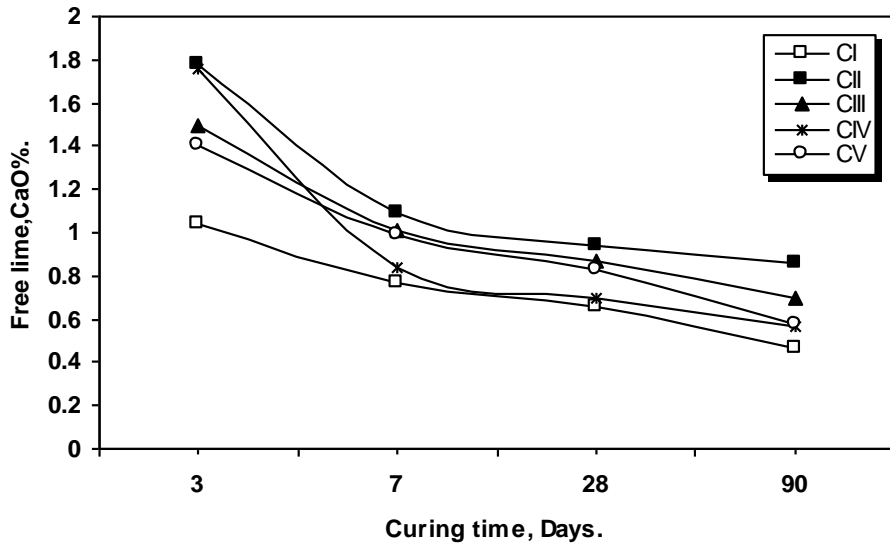


Fig.(12): Free lime contents of hardened specimens (mixes CI-CV) made from PC and MK (burnt KK at 1000°C) as a function of curing time.

FTIR spectroscopic study:

The infrared spectra of mix CI containing fired KK clay at 800°C after 3, 7, 28 and 90 days of curing are seen in Fig.(13). It is clear that the intensities of broad band at 3448 cm^{-1} and weak peak at 1656 cm^{-1} , which are both related to combined water [19], increased with curing time. The bands at 1458 and 876 cm^{-1} are attributed to presence of calcite, possibly formed due to aeration of the pastes [20]. Also, it is clear that the peak at 1014 cm^{-1} is attributed to formation or transformation of CSH [21], increased with curing time. IR spectra corresponding to hydrated PC-MK pastes of mixes CI-CV containing KK clay fired at 800°C and 900°C after 90 days curing are illustrated in Figs.(14&15), respectively. It is clear that the intensities of broad bands at 3456 and 3424 cm^{-1} (800&900°C, respectively) due to combined water varied with the variation of MK content in the pastes. Mix CI possesses lowest intensity while mix CV has the highest intensity. This is attributed to the highest pozzolana content of mix CV which need more water molecules for hydration process. There are two bands at 1460 and 1438 cm^{-1} for 800°C and 900°C, respectively, which are both related to carbonation of samples by absorbing CO_2 from the air forming CaCO_3 during the hydration process [21]. Generally, Mix CI has the lowest band intensity at different firing temperatures (800°C and 900°C), so it has lowest CaCO_3 content which causes an increasing in the rate of hydration process due to lowest carbonation. The bands at 1034 and 1028 cm^{-1} (800&900°C, respectively) are related to CSH (tobermorite) formation [20]. The intensity of these bands increased with increasing MK contents due to the production of more CSH products with different phases. There is a weak band appear at 540 cm^{-1} (800°C only) due to Al-O, hydrogarnet or Ca-O of C-A-H [21]. The intensity of this band increased with increasing MK content due to increasing of Al contents. Finally, it is clear from IR spectroscopic analysis that mix CI has the highest hydraulic properties of PC-MK pastes.

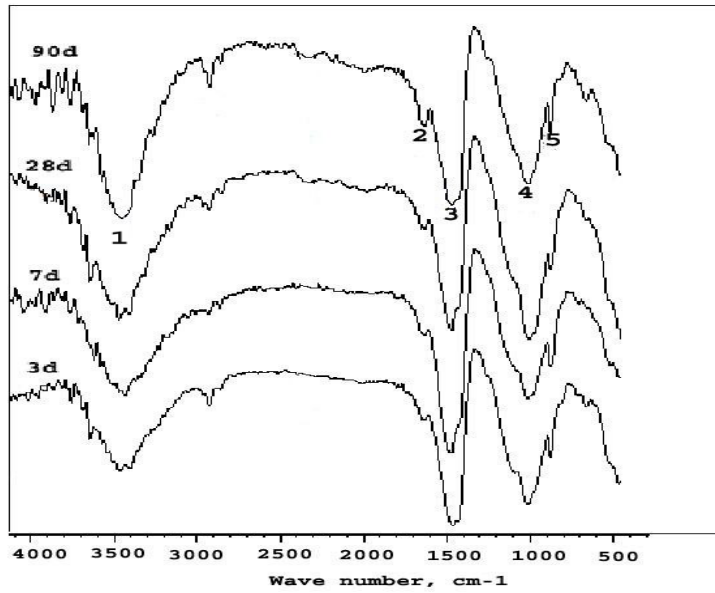


Fig.(13): IR spectra of hydrated mix CI at 800°C calcining temperature of KK clay after 3 , 7, 28 and 90 days curing.

(1= 3448 cm⁻¹, 2 =1656 cm⁻¹, 3 =1458 cm⁻¹, 4 =1014 cm⁻¹, 5 = 876 cm⁻¹)

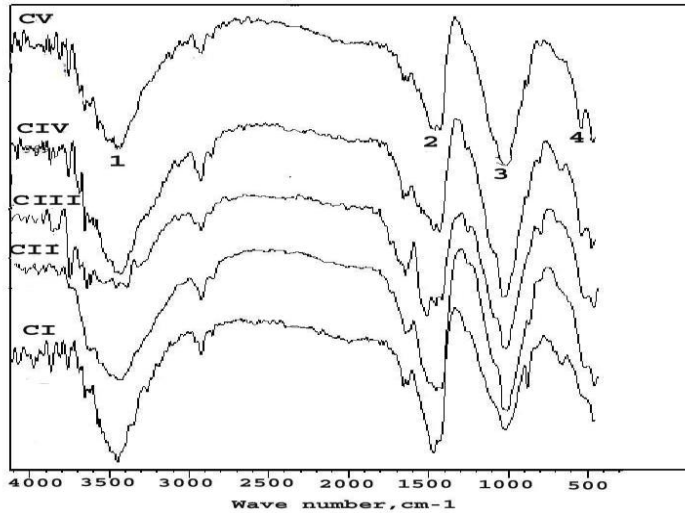


Fig.(14): IR spectra of hydrated mixes (CI-CV) containing calcined KK clay at 800°C after 90 days curing.

(1= 3456 cm⁻¹, 2 =1460 cm⁻¹, 3 =1034 cm⁻¹, 4 = 540 cm⁻¹)

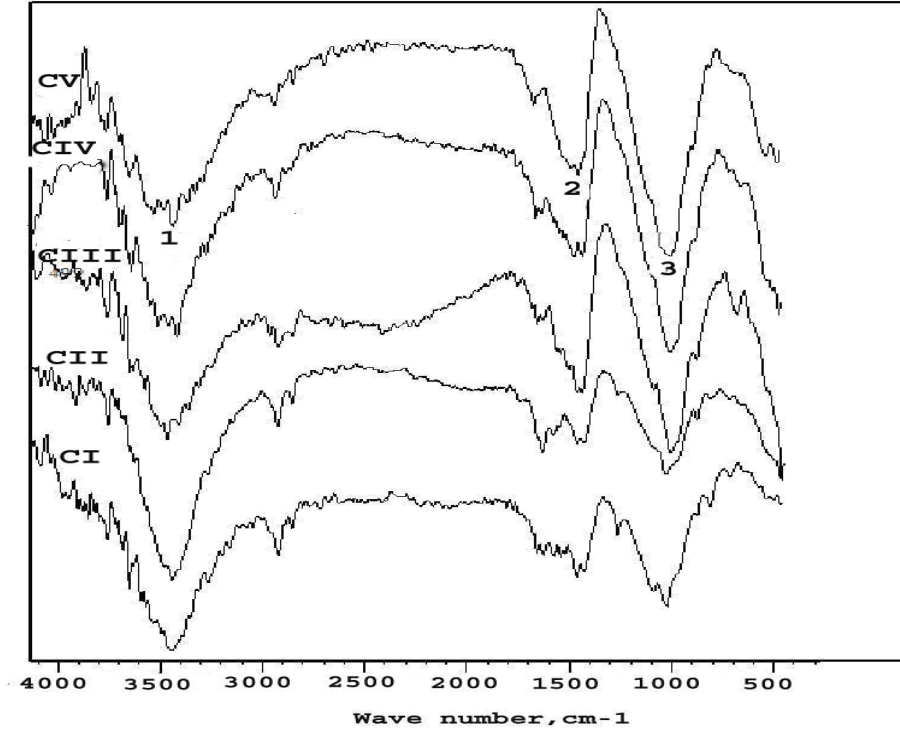


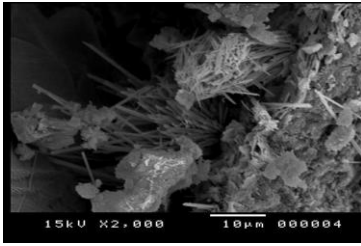
Fig.(15): IR spectra of hydrated mixes (CI-CV) containing calcined KK clay at 900°C after 90 days curing.

(1 = 3424 cm^{-1} , 2= 1438 cm^{-1} , 3 = 1028 cm^{-1})

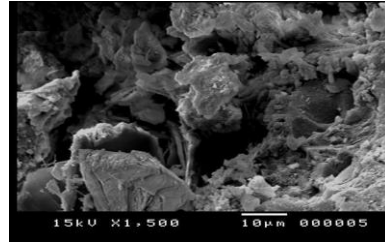
Morphology and microstructure:

Eight samples were investigated using scanning electron microscopy (SEM) as representatives for the hardened PC-MK pastes in this study. Four of these samples were made from mix CI (95% PC: 5% MK) at the firing temperatures of KK clay 700, 800, 900 and 1000°C after 90 days curing; their SEM micrographs are shown in Figs. 16, 18, 20 and 22, respectively. The other 4 samples were made from mix CV (75% PC: 25% MK) at the firing temperature of KK clay 700, 800, 900 and 1000 °C after 90 days curing; their SEM micrographs are shown in Figs.17, 19, 21 and 23, respectively. The SEM micrographs obtained for the specimens of mix CI at firing

temperature 700°C after 90 days curing [Fig.16 (a&b)], displayed a mixture of various hydration products mainly as nearly amorphous and short fibers of CSH. The micrographs of mix CV at same firing temperature and curing time [(Fig.17 (a&b))] indicated formation of fibrous and rod-like crystals of hydration products beside flocculent and porous structure. In this paste the wider pores are available for crystallization of the formed hydrates. So, this paste has a lower strength due to the higher porosity. The microstructure obtained after 90 days curing of mix CI containing burnt KK at 800°C, showed a dense structure of nearly amorphous CSH as representing the main hydration products [Fig.18 (a&b)]; accordingly mix CI possessed high hydraulic properties. The micrographs of mix CV at same firing temperature of KK and curing time [Fig.19 (a&b)], show thin platelet crystals flat or crumbled and their edges were hexagonal or irregular. Crystals of this kind possessed low hydraulic properties. The SEM micrographs obtained after 90 days curing of mix CI containing calcined KK 900°C [(Fig.20 a&b)], showed dense structure of platelet-like hydrates of CSH and fully crystalline hydrates having close-textured structure with interlocking arrangements .The micrographs also, show longer rod-like particles of CSH. The micrographs of mix CV at same firing temperature and curing time displayed close-textured, dense and massive structure composed of interlocking fibers and crumpled foils as well as particles with irregular appearance [Fig.21 (a&b)]. The microstructure obtained after 90 days curing of mix CI containing burnt KK at 1000°C, indicated the formation of amorphous hydrates and a massive structure of ill-crystallized CSH with dense structure [Fig.22 (a&b)]. The SEM micrographs of mix CV at same firing temperature of KK and same curing time, showed formation of nearly amorphous CSH as well as some cubic crystals of hydrogarnet-like calcium alumino-silicate hydrate [Fig.23 (a&b)]. From the morphology and microstructure studies in this investigation it is clear that specimens made from mix CI (95% PC: 5% MK) possess the highest hydraulic properties than specimens made from mix CV (75% PC: 25% MK) for all firing temperatures of KK and all curing times. These results are in a good agreement with the obtained physico-chemical measurements of these PC-MK pastes.

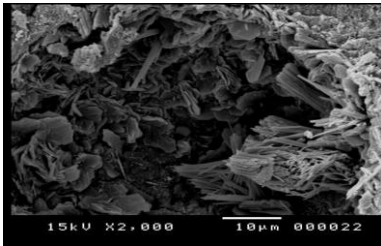


(a)

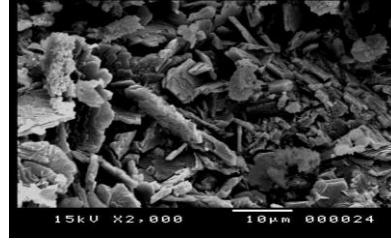


(b)

Fig.(16): SEM micrographs of mix CI contains calcined KK at 700°C after 90 days curing.

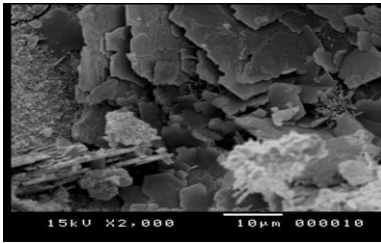


(a)

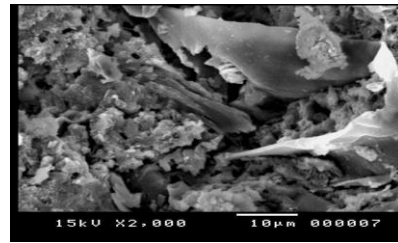


(b)

Fig.(17): SEM micrographs of mix CV contains fired KK at 700°C after 90 days curing

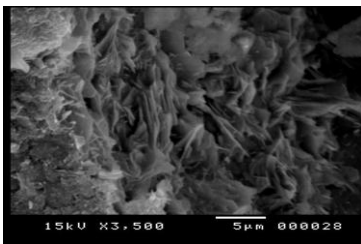


(a)

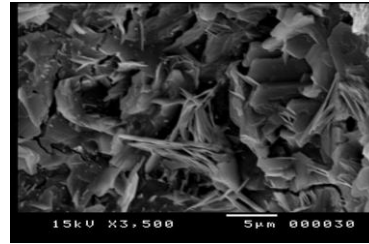


(b)

Fig.(18): SEM micrographs of mix CI contains calcined KK at 800°C after 90 days curing.



(a)



(b)

Fig.(19): SEM micrographs of mix CV contains calcined KK at 800°C after 90 days curing

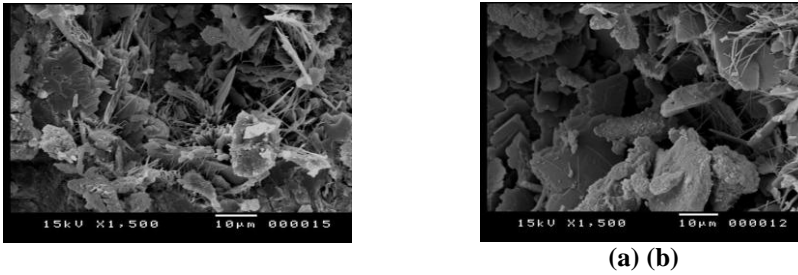


Fig.(20): SEM micrographs of mix CI contains calcined KK at 900°C after 90 days curing.

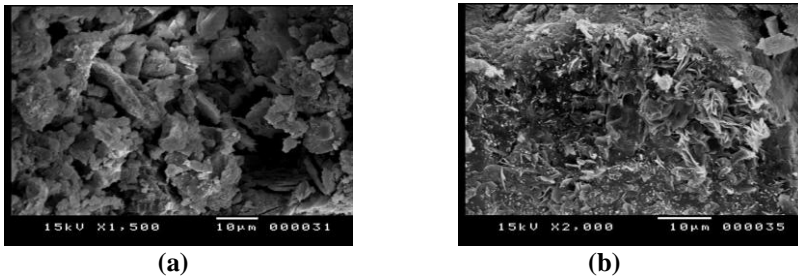


Fig.(21): SEM micrographs of mix CV contains calcined KK at 900°C after 90 days curing.

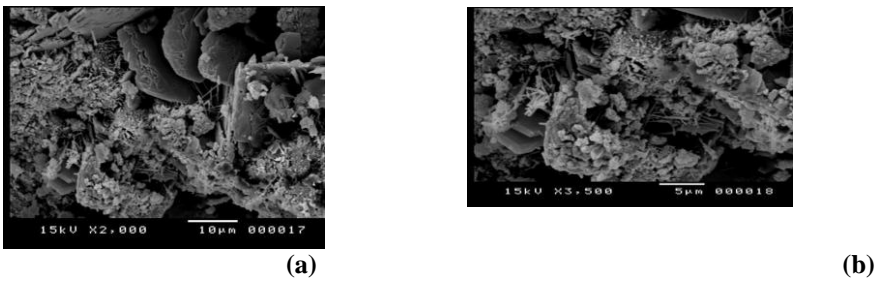


Fig.(22): SEM micrographs of mix CI contains calcined KK at 1000°C after 90 days curing

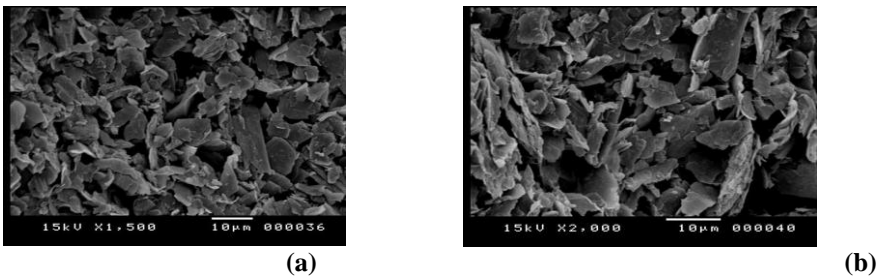


Fig.(23): SEM micrographs of mix CV contains calcined KK at 1000°C after 90 days curing

Conclusions

From this study we can conclude that:

- 1- The compressive strength of the various PC-MK pastes increases continuously with increasing age of hydration up to 90 days for all calcining temperatures of KK clay. Mix CI (95% PC: 5% MK) possess the highest compressive strength values at all calcining temperatures and all times of curing, the optimum value of compressive strength was at 800°C after 90 days of curing.
- 2- The porosity values decreased for all types of mixes at all curing times and all firing temperatures of KK clay. Generally, Mix CI possesses lower porosity values at all firing temperatures of clay and all curing times which is in harmony with compressive strength results.
- 3- The results of free lime contents indicate that the free lime was consumed gradually with increasing age of hydration for all types of the hardened PC-MK pastes.
- 4- The infrared spectroscopic analysis of some PC-MK pastes indicated the appearance of bands at different wave lengths due to formation of different phases. For all selected samples there are different bands appear due to combined water, carbonation, CSH and CAH formation with variation of intensities according to the mix composition, firing temperature and curing time. Generally, mix CI (95% PC: 5% MK) has improved hydraulic properties.
- 5- The morphology and microstructure of studied hardened PC-MK pastes of mix CI showed formation of nearly amorphous and short fibers of CSH, dense structure of nearly amorphous CSH, dense structure of platelet-like hydrates of CSH and fully crystalline hydrates having close-textured structure with interlocking arrangements and a massive structure of ill-crystallized CSH with dense structure.
- 6- Finally, this study indicated that partial substitution of ordinary Portland cement with 5% to 25% burnt Kalabsha kaolinite clay at 800°C will produce pozzolanic cement with higher hydraulic properties than ordinary Portland cement and the optimum substitution value is 5% burnt Kalabsha kaolinite clay at 800°C.

References

1. D.S. KLIMESCH AND A. RAY, *Adv. Cem. Based Mater.*, **7**, 109–118 (1998).
2. H. EL-DIDAMONY, K.A. KHALIL AND M.S. EL-ATTAR, *Cem. Concr. Res.*, **30**, 7-11 (2000).
3. H. CHANGLING, B. OSBAECK AND E. MAKOVICKY, *Cem. Concr. Res.*, **25(8)**, 1691–1702 (1995).
4. M.H. ZHANG AND V.M. MALHOTRA, *Cem. Concr. Res.*, **25(8)**, 1713–1725 (1995).
5. T. RAMLOCHAN, M.D.A.THOMAS AND R.D. HOOTON, *Cem. Concr. Res.*, **34(8)**, 1341-1356 (2004).
6. L. COURARD, A. DARIMONT, M. SCHOUTERDEN, F. FERAUCHE, X.Willem and R. Degeimbre, *Cem. Concr. Res.*, **33(9)**, 1473-1479 (2003).
7. E. BADOGIANNIS, G. KAKALI, G. DIMOPOULOU, E. CHANIOTAKIS AND S. TSIVILIS, *Cem. Concr. Composite*, **27(2)**, 197-203 (2005).
8. XI. QIAN AND Z. LI, *CEM.Concr.Res*, **31**, 1607-1611 (2001).
9. C.S. POON, L. AM, S.C. KOU, Y. WONG AND R.WONG, *Cem. Concr. Res.*, **31**, 1301-1306 (2001).
10. G. BATIS, P. PANTAZOPOULOU, S. TSIVILIS AND E. BADOGIANNIS , *Cem. Concr. Composite*, **27(1)**, 125-130 (2005).
11. M. ABD EL AZIZ , S. ABD EL ALEEM , M. HEIKAL AND H. EL-DIDAMONY , *Silicate Industriels.*, **68(1/2)**,73–84 (2004).
12. H. EL-DIDAMONY , S.A. ABO-EL-ENEIN AND M.Y. HAGGAG , *Cem. Concr. Res.*, **8**, 351 (1978).
13. M.P. JAVELLANA AND I. JAWED , *Cem. Concr. Res.*, **12**, 399–403 (1982).
14. Soroka ,” Portland cement past and concrete”. New York: Macmillan; 1970.
15. Z. DING, D. ZHANG AND Y. XIE, *J. Build. Mater.* , **1**, 201-205 (1998)
16. Z. DING, D. ZHANG AND R.YU, *China. Mater. Sci. Technol.*, **1**, 14-17 (1999).
17. J.M. KHATIB AND S.WILD, *Cem. Concr. Res.* , **26(10)**, 1545-553 (1996).
18. M. FRIAS AND J. CABRERA, *Cem. Concr. Res.* **32(1)**, 133-138 (2002).
19. N.J. SAIKA, P. SENGUPA, P.K. GOGOI AND P.C. BORTHAKUR, *Cem. Concr. Res.*,**32**, 1717-1724 (2002).
20. N.J. SAIKA, P. SENGUPTA, P.K. GOGOI AND P.C. BORTHAKUR, *Cem. Concr. Res.*, **32**, 207-302 (2001).
21. F. PUERTAS, H. SANTOS, M. PALACIOS AND S. MARTINZ-RAMIREZ, *Advances in Cement Research*, **17(2)**, 77-89 (2005).

تأثير حرارة حرق طفلة الكاولين على خصائص العجائن المحتوية على الأسمنت البورتلاندى والميتاكاولين

محمود أحمد ظاهر، عبد العزيز السيد يوسف ، عثمان عبد المعطى فرغلى ، محمد رضا شتات
قسم الكيمياء- كلية العلوم- جامعة الأزهر فرع أسيوط

ملخص البحث

تعتبر طفلة الكاولين المحروقة (الميتاكاولين) من أهم المواد البوزولانية ذات النشاطية العالية بسبب القيمة العالية لمساحة سطحها النوعى وكذلك إحتوائها على خليط من اكاسيد السيليكا والألومينا فى حالة غير بللورية بعد المعالجة الحرارية وعندما تضاف هذه الطفلة المحروقة الى الأسمنت البورتلاندى فى وجود الماء تزداد عملية التآدرت وتتكون مركبات هيدراتية لها خصائص هيدروليكية ملحوظة وإن الزيادة فى تكوين هذه المركبات الهيدراتية فى حالة عجائن الأسمنت البورتلاندى- الميتاكاولين يعود الى التفاعل بين الجبر المنطلق من تآدرت الأسمنت مع الميتاكاولين. لكل ماسبق ذكره فإن طفلة الميتاكاولين الناتجة من حرق الكاولين لاقت إهتماما كبيرا فى إمكانية استخدامها فى صناعة الأسمنت لما لها من مردود إقتصادى وبيئى.

فى هذا البحث تمت دراسة مدى تأثير استخدام طفلة الميتاكاولين الناتجة من حرق طفلة الكاولين (من منطقة كلابشة بأسوان) عند درجات حرارة 700، 800، 900، 1000 درجة مئوية على الخواص الفيزيوكيميائية لعجائن الأسمنت البوزولانى المصنعة بإحلال جزء من الأسمنت البورتلاندى بالميتاكاولين بالنسب الوزنية 0% ، 5% ، 10% ، 15% ، 20% ، 25% من الميتاكاولين وذلك لكل درجة حرارة حرق للكاولين . وقد تم تحضير العجائن بالخلط بالماء والصب فى قوالب ثم المعالجة بالغمر فى الماء للعجائن المتصلدة . وقد تم دراسة مدى التغير فى الخواص الفيزيوكيميائية للعجائن بقياس نسبة الجبر الحر وقوة الأنضغاط والمسامية لعجائن الأسمنت البورتلاندى - ميتاكاولين بعد غمرها فى الماء فى الأزمنة 3و7و28و90 يوم . وكذلك تمت دراسة التغير فى الأطوار لبعض العينات باستخدام القياسات الطيفية للأشعة تحت الحمراء (IR spectroscopy) . و تمت دراسة التغير فى التركيب الدقيق لبعض العينات باستخدام الميكروسكوب الماسح الألكترونى (Scanning electron microscop) .

وقد استنتج من هذه الدراسة أنه يمكن إنتاج أسمنت بوزولانى له خصائص هيدروليكية أعلى من الأسمنت البورتلاندى العادى وذلك بإحلال كاولين كلابشة محروق عند درجات حرارة 800 أو 900 أو 1000 درجة مئوية محل الأسمنت البورتلاندى بنسب تتراوح بين 5% الى 25% وإن نسبة الإحلال الذى تحقق أعلى خصائص هيدروليكية هى 5% من كاولين كلابشة المحروق عند درجة حرارة 800 درجة مئوية .