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## **PHYSICOCHEMICAL PROPERTIES OF METAKAOLIN-LIME PASTES AT DIFFERENT CALCINATION TEMPERATURES OF KAOLINITE CLAY**

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### **Abstract**

The study of metakaolin-lime hydration process is of great interest in the field of building materials technology. This study aimed to investigate experimentally the physicochemical properties of pastes containing metakaolin (MK) and lime as activator. MK was produced by calcining Kalabsha kaolinite clay (Aswan, Egypt) at different calcination temperatures 700, 800, 900 and 1000°C for 2 hr. Five mixes were prepared by partial substitution of MK by 5, 10, 15, 20 and 25% wt CaO (from CaCO<sub>3</sub>) by weight, then hydrated up to 90 days. The characteristics of prepared specimens were investigated by measuring compressive strength and total porosity. The hydration kinetics were evaluated by determination of free lime contents. The change in phases was investigated by IR technique. The morphology and microstructure of hardened pastes were investigated by scanning electron microscopy (SEM) tests. The results show that, partial substitution of burnt kaolinite clay at 900°C with 15 % CaO improves the hydraulic properties of MK- lime pastes.

**Keywords:** Metakaolin, Pozzolan, Kaolin, Lime.

**Abbreviations:** C:CaO ; S:SiO<sub>2</sub> ; H:H<sub>2</sub>O ; A:Al<sub>2</sub>O<sub>3</sub> ; F:Fe<sub>2</sub>O<sub>3</sub> ; CH: Ca(OH)<sub>2</sub>

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### **Introduction**

The hydraulic reactivity of some artificial pozzolana made from burnt clay using lime as an activator was previously studied [1]. These pozzolans were made by firing of montmorillonite, montmorillonite-allite mixed layer, and kaolinite clay at 600, 700 and 800°C. Pozzolana-lime pastes with water were hydrated at room temperature for various time intervals. The hydration of calcined clays with Ca(OH)<sub>2</sub> produces calcium silicate as well as calcium aluminosilicate hydrates as cementing materials. A study was carried out to determine the effect of curing temperature on the kinetics of reaction of a metakaolin (MK)/ lime mixture [2]. MK and analytical grade Ca(OH)<sub>2</sub> were mixed in a ratio of 1:1 by weight and with a water/binder ratio of 2.37 then cured at 20 and 60°C. In the first case, the curing time varied from 2 h up to 180 days and, in the second case, from 2 h up to 123 days. A mathematical model was applied to calculate the rate constant for the hydration reaction. The identity and the amount of the phases present were determined from thermal analysis (TG and DTA) data. The results showed that the rate constant for the samples cured at 60 °C was 68 times greater than the rate

constant at 20°C for the same curing period (up to 9 days). At 20 °C, the sequence of appearance of the hydrated phases was C–S–H, C<sub>2</sub>ASH<sub>8</sub> and C<sub>4</sub>AH<sub>13</sub>; while at 60°C, the sequence was C–S–H, C<sub>2</sub>ASH<sub>8</sub>, C<sub>4</sub>AH<sub>13</sub> and hydrogarnet (C<sub>3</sub>ASH<sub>6</sub>).

Ambroise, Joseph and Pera [3] used X-ray diffraction analysis to investigate the influence of temperature on thermal activation of two specimens of montmorillonite clay. The specimens were subjected to thermal activation for 5 hours at 650,750,780,800,820,850,900 and 950°C in fixed bed reactor. The XRD before and after heating showed that thermal activation did not produce a completely amorphous material. These activated specimens showed pozzolanic activity when mixed with lime and water. Measurement of compressive strength up to 90 days showed that the rate of strength development depends on the activation temperature. The maximum 28-days compressive strength was obtained when the material was activated at 800°C.

Murat [4] found that when mixed with Ca(OH)<sub>2</sub> and water, metakaolinite, obtained by fixed-bed calcination of kaolinite at 730°C and develops 28-days compressive strengths of 10-15 MPa. Hydration products are essentially 2CaO.Al<sub>2</sub>O<sub>3</sub>.SiO<sub>2</sub>.8H<sub>2</sub>O (gehlenite hydrate) and CSH. Four mixes were prepared of weight ratios 80:20, 70:30, 60:40 and 50:50 of China: pure Ca(OH)<sub>2</sub> [5]. Each mix was calcined for 2 hours at 800°C and then paste hydrated in 100% relative humidity at room temperature up to 28 days. The hydration products were studied by XRD as well as DTA and TG techniques. The chemically-combined water and Ca(OH)<sub>2</sub> contents were quantitatively determined from TG curves. The results illustrated the formation of gehlenite hydrate (C<sub>2</sub>ASH<sub>8</sub>) as the main hydration product; its amount increases with curing time. Cabrera and Frias [6] made a detailed study of the pozzolanic reaction between MK and lime, showing the main phases produced at early hydration times from 2hours to 9 days. It was reported that C<sub>2</sub>ASH<sub>8</sub> and C<sub>4</sub>AH<sub>13</sub> were stable under the condition of the study, and there was no evidence of a possible conversion reaction from the phase to hydrogarnet. Also, the study focuses on the influence of curing temperature on the mechanism of reaction in MK-lime systems which gives valuable information about the nature of the reaction products and their stability with hydration time. At early stages of the reaction, the C-S-H was the main phases for both temperatures. Subsequently, C<sub>2</sub>ASH<sub>8</sub> (stratignite) and C<sub>4</sub>AH<sub>13</sub> appear, and finally the C<sub>3</sub>ASH<sub>6</sub> (hydrogarnet) was the predominant phase in the samples cured at 60°C. No hydrogarnet formation was detected. At 20°C, there is clear evidence of the existence of C<sub>4</sub>AH<sub>13</sub> in the absence of Ca(OH)<sub>2</sub> [7].

The effect of calcination temperature on clay and limestone as well as hydration characteristics of calcined products were investigated [8]. Three mixes 50/50, 60/40, 70/30 wt.% clay-limestone were calcined at 700, 800, 900, and 1000°C for 2 h, then hydrated for up to 90 days. The degree of calcination was investigated from the free lime content and the ignition loss for each mixture. Also, the mineralogical composition of the fired mixes was investigated with the aid of X-ray diffractometry. The results revealed that the free lime of each mix increased up to 800°C then decreased gradually up to 1000°C. Mix 60/40 clay-limestone fired at 800°C shows the presence of  $\text{Ca}(\text{OH})_2$  with quartz. As the firing temperature increased gehlenite appeared and increased up to 1000°C with the disappearance of lime. Mix 50/50 gave the highest hydration kinetics as measured from the determination of free lime and combined water contents. As the limestone decreased, the rate of hydration decreased. The suitable firing temperature of the clay-limestone mixes was 800°C for 2 hours.

### Experimental:

#### Materials:

The materials used in this investigation were Kalabsha kaolinite clay (KK) and CaO prepared from  $\text{CaCO}_3$  BDH grade. The chemical oxide compositions of Kalabsha kaolinite clay used in this study are shown in Table (1).

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

Oxide contents	Kalabsha kaolinite clay
$\text{SiO}_2$	44.18
$\text{Al}_2\text{O}_3$	36.75
$\text{Fe}_2\text{O}_3$	1.36
CaO	0.26
$\text{SO}_3$	-----
MgO	0.16
$\text{Na}_2\text{O}$	0.18
$\text{K}_2\text{O}$	0.25
$\text{TiO}_2$	2.94
L.O.I	13.55
Total	99.63

#### Methods:

##### *Preparation of calcined clay (MK) and CaO:*

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  $\mu\text{m}$  B.S. sieve; the processes of grinding and sieving were repeated until the sample is completely passed through a 90  $\mu\text{m}$  sieve. CaO was prepared by firing  $\text{CaCO}_3$  (BDH grade) at 1000 °C in muffle furnace for 2 hrs and kept in desiccators until mixing with MK for the preparation of different specimens.

*Preparation of MK- CaO specimens:*

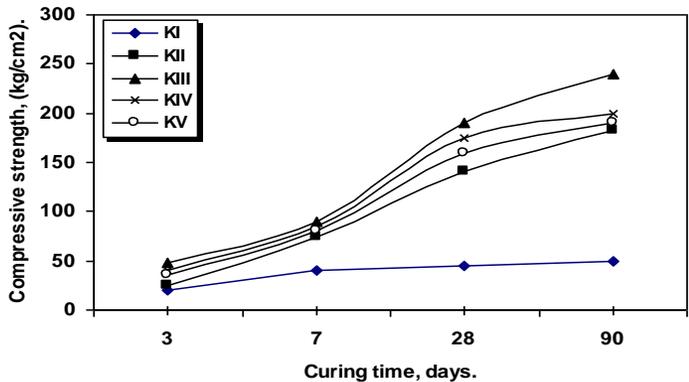
CaO was mixed with four types of MK produced by burning Kalabsha kaolinite clay at 700,800,900 and 1000°C. Five dry mixtures were prepared from various proportions of MK and CaO with the mass ratios 100:0(B), 95:5(KI), 90:10(KII), 85:15(KIII), 80:20(KIV) and 75:25(KV), respectively, using ethanol to obtain complete homogeneity 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 molding, 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, three specimens were used to determine the residual compressive strength according to ASTM specification (ASTM, 1992 C). Total porosity was determined after any time of hydration as described elsewhere [9]. The hydrations of pastes were stopped by employing alcohol–ether method [10]. 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 [11, 12]. 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 values of the various hardened pastes made from MK (produced by firing KK at different temperatures 700,800,900 and 1000°C) and CaO as a function of curing time are graphically represented in Figs. 1, 2, 3 and 4, respectively. The results indicate that, the compressive strength increases gradually from 3 to 90 days curing for all mix composition and all firing temperatures of clay.

This result might be attributed to the increase of hydration products which act as binding centers in the cured specimens with time of hydration. The reduction in compressive strength value in some mixes in the early stage of hydration (3-7days) is mainly due to the interaction between the initially formed calcium silicate hydrates and the remaining parts of pozzolanic grains leading to a decrease of the lime content of these hydrates. At later ages (28-90 days), the hardened specimens process high strength values due to the accumulation and later stabilization of the hydration reaction products. Moreover, the strength values are higher for specimens prepared from mixes KIII, KIV and KV with higher lime contents (15%, 25% and 25% , respectively) as compared with the strength values of specimens made from mixes KI and KII with low lime contents (5% and 10%, respectively) at all firing temperature of clay. This result may be attributed to the increase of the formation of stabilized hydrates in presence of high lime contents. Mix KIII(85% MK:15% CaO) has the highest compressive strength values at all curing ages and firing temperatures of clay and the optimum value for this mix was at 90°C after 90 day curing. Also, mix KI (95% MK: 5% CaO) has the lowest compressive strength values at all curing ages and firing temperatures. Although mix KV has the highest lime contents, but it possessed compressive strength lesser than mixes KIII and mix KIV at all curing ages and firing temperatures. This may be attributed to the presence of high lime contents which causes formation and later accumulation of calcium aluminosilicate hydrates (hydrogarnet) having weak hydraulic properties. Accordingly, mix KIII at firing temperature 900°C of KK was suitable for the production of building materials containing lime and fired KK.



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

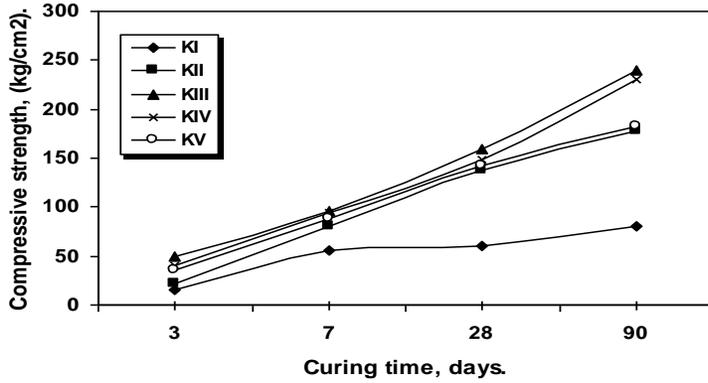


Fig.(2): Compressive strength of hardened specimens ( mixes KI-KV ) made from MK (burnt KK at 800°C) and CaO as a function of curing time

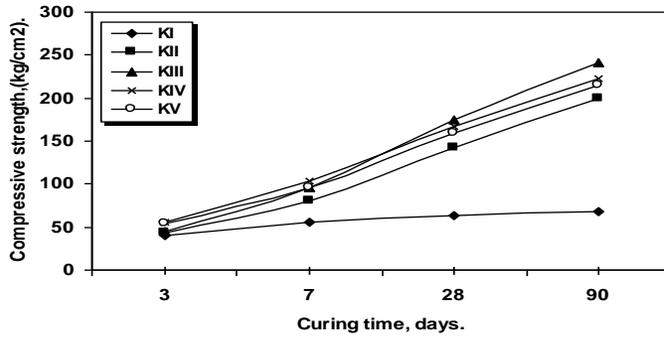


Fig.(3): Compressive strength of hardened specimens ( mixes KI-KV ) made from MK (burnt KK at 900°C) and CaO as a function of curing time

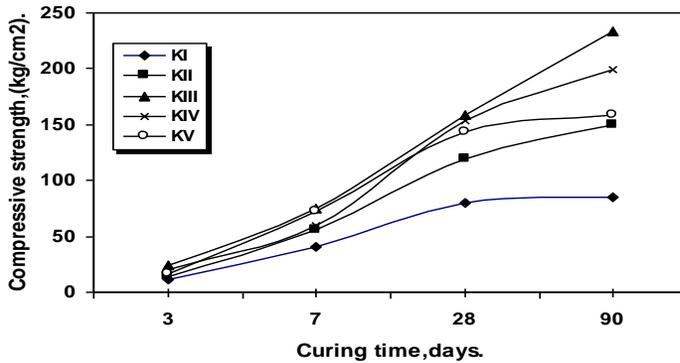
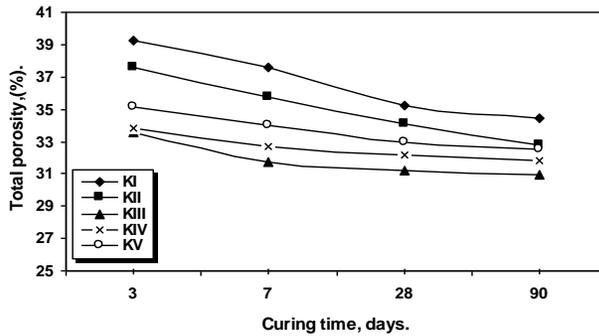


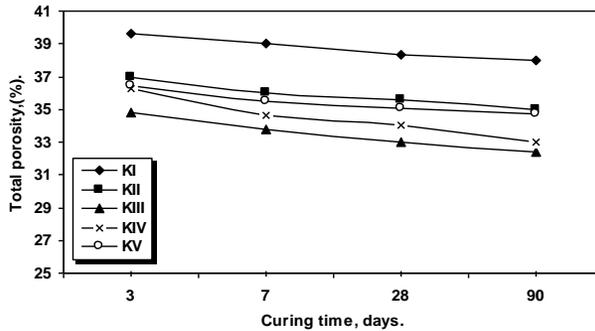
Fig.(4): Compressive strength of hardened specimens ( mixes KI-KV ) made from MK (burnt KK at 1000°C) and CaO as a function of curing time

*Total Porosity:*

The total porosity values of the hardened specimens made from MK fired KK at different temperatures 700,800,900 and 1000°C) and CaO at different proportions are graphically represented in Figs.5, 6, 7 and 8, respectively. The results show that the total porosity decreases with curing time for all MK- CaO pastes at all mix composition and firing temperatures of KK as a result of progress of hydration. The hardened pastes of mixes KIII ( 85% MK:15% CaO ) give the lower porosity values up to 90 days at all firing temperatures, while mixes KI (95% MK:5% CaO) give the highest porosity values. This may be due to the products of the MK-lime pozzolanic reaction, which have high molecular weight silicate chains in presence of high lime contents [13]. This may be also due to the decrease of hydration products formed at lower values of CaO. Generally, the total porosity of metakaolin fired at 1000°C is higher than the other firing temperatures due to the formation of some crystalline phases which retards the hydration of MK-lime mixes.



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



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

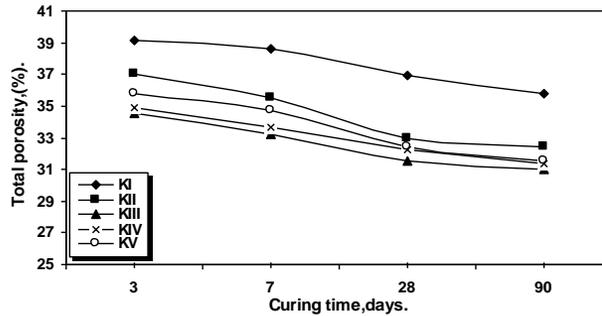


Fig.(7): Total porosity of hardened specimens ( mixes KI-KV ) made from MK (burnt KK at 900°C) and CaO as a function of curing time

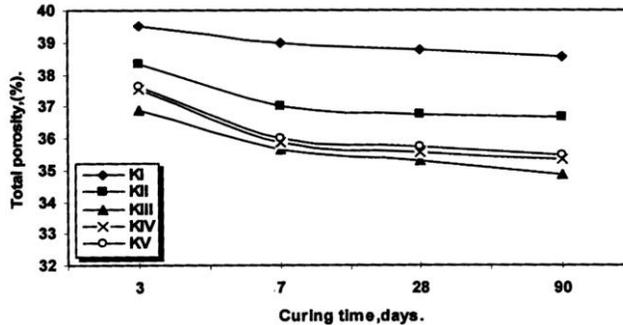


Fig.(8): Total porosity of hardened specimens ( mixes KI-KV ) made from MK (burnt KK at 1000°C) and CaO as a function of curing time

#### Free lime contents:

The free lime (CaO%) values of the hardened pastes made from MK (burned KK at 700, 800, 900 and 1000°C) and CaO as a function of mix composition and curing time are graphically represented in Figs. 9, 10, 11 and 12, respectively. The free lime was consumed gradually during the hydration process of all mixes and at all firing temperatures of KK. Obviously, all of MK-CaO pastes possess extremely very low free lime content values at all stages, a result of the immediately complete reaction of liberated lime with active  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$  of the decomposed MK. Generally, pastes containing mix KI (95% MK: 5% CaO) possess low free lime contents; while that containing mix KV (75% MK: 25% CaO) possess the highest free lime contents at all curing stages and all firing temperatures of KK. This is mainly attributed to the low lime content in mixes KI which is immediately completely consumed with decomposed MK. As the lime content increases in the MK pastes the free lime increases due to the decrease of retarded lime with CaO to form hydration products such as  $\text{CSH}$ ,  $\text{C}_4\text{AH}_{13}$ ,  $\text{C}_2\text{ASH}_8$  and  $\text{C}_3\text{AS}_3\text{H}_6$ .

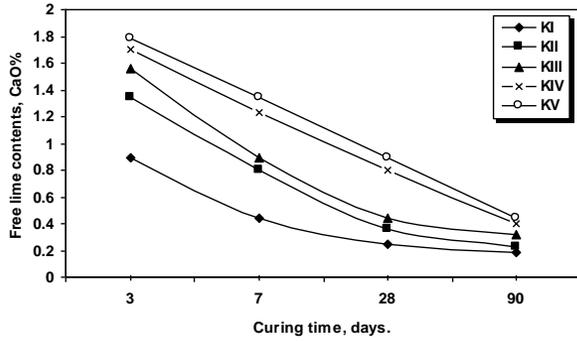


Fig.(9): Free lime contents of hardened specimens ( mixes KI-KV) made from MK (burnt KK at 700°C) and CaO as a function of curing time

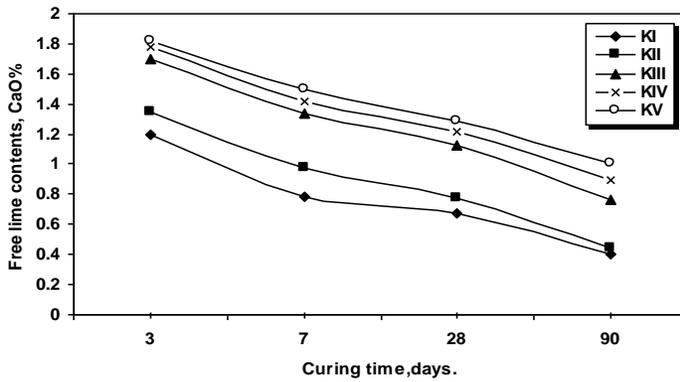


Fig.(10): Free lime contents of hardened specimens ( mixes KI-KV) made from MK (burnt KK at 800°C) and CaO as a function of curing time

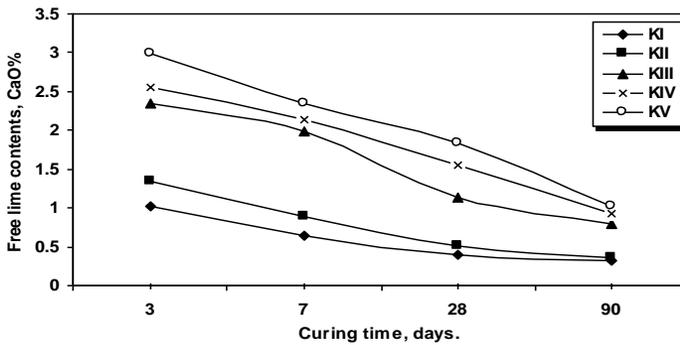
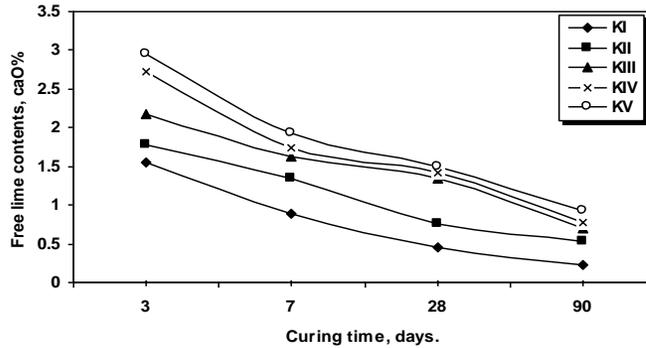


Fig.(11): Free lime contents of hardened specimens ( mixes KI-KV) made from MK (burnt KK at 900°C) and CaO as a function of curing time



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

#### *Infra Red Spectroscopic Analysis:*

The infrared spectroscopic analysis was carried out on some selected samples of hardened MK-lime pastes of mix KIII containing fired KK clay at 800°C as a function of curing time and mixes (KI-KV) containing fired KK clay at 900°C and 1000°C after 90 days to identify the phases coexisting during the hydration process. The IR spectra of hydrated MK-lime paste (KK fired at 900°C) after 3,7,28 and 90 days are shown in Fig.13. It is clear that the intensity of broad band at 3484  $\text{cm}^{-1}$  due to combined water [14] increased with curing time due to acceleration of hydration process with curing time. The intensity of 1438  $\text{cm}^{-1}$  band due carbonation[15] decreases with curing time which gives advantage for the reaction of lime with pozzolana grains in MK. The 1038  $\text{cm}^{-1}$  band which is attributed to CSH (tobermorite) formation increases with curing time and 544  $\text{cm}^{-1}$  band which is attributed to Al-O, hydrogarnet or Ca-O in C-A-H increases with curing time due to accumulation of CSH in the hydration process [16] . Figs.(14&15) illustrated the IR spectra of mixes KI-KV containing fired KK clay at 900°C and 1000°C after 90 days curing respectively. The bands at 3456&1650  $\text{cm}^{-1}$  for 900°C and 3420 &1654  $\text{cm}^{-1}$  for 1000°C due to combined water increased with curing time due to increasing in the rate of hydration process in presence of higher lime content [15]. There are two bands appear due to carbonation[14] at 1438 & 876  $\text{cm}^{-1}$  for 900°C and three bands at 1430,874and 712  $\text{cm}^{-1}$  for 1000°C, the intensities of these bands increased with curing time due to increasing of lime contents causes increase the carbonation . It is clear that there is increasing in the intensities of 1034 & 540  $\text{cm}^{-1}$  bands of 900°C and 1030& 536  $\text{cm}^{-1}$  bands for 1000°C with increasing lime content (KI- KV) due to the accumulation of CSH (tobermorite) and presence of hydrogarnet, Al-O and Ca-O in C-A-H[16]. Mix KIII show higher intensity bands in these cases at 900 & 1000°C. Accordingly, mix KIII improves the hydraulic properties of theses mixes which is in harmony with other measurements in this investigation.

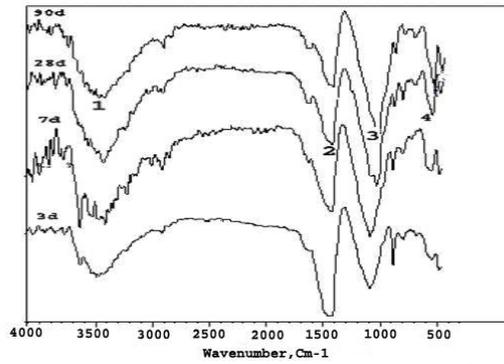


Fig.(13) : IR spectra of mix KIII contains fired KK at 900°C after 3,7,28 and 90 days curing. ( 1= 3484 cm<sup>-1</sup>, 2= 1438 cm<sup>-1</sup>, 3= 1038 cm<sup>-1</sup>, 4= 544 cm<sup>-1</sup> )

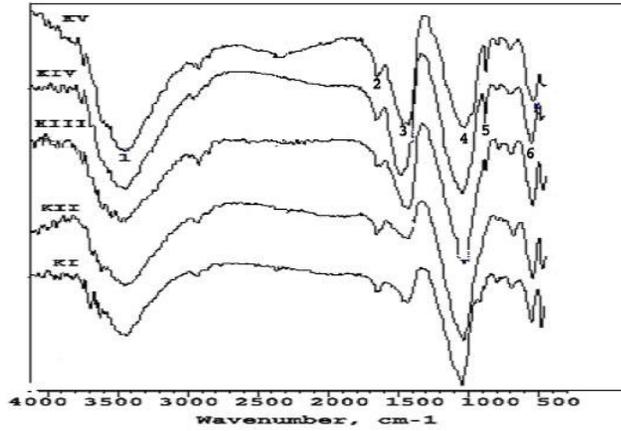


Fig.(14) : IR spectra of mixes (KI-KV) contains fired KK at 900°C after 90 days curing (1= 3456 cm<sup>-1</sup>, 2= 1650 cm<sup>-1</sup>, 3= 1438 cm<sup>-1</sup>, 4= 1034 cm<sup>-1</sup>, 5= 876 cm<sup>-1</sup>, 6= 540 cm<sup>-1</sup>)

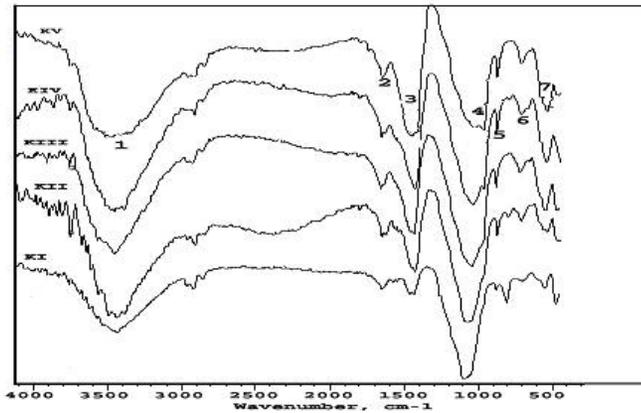
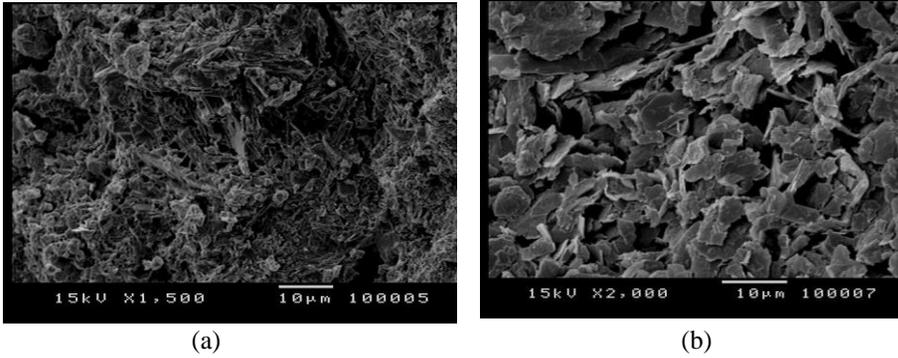


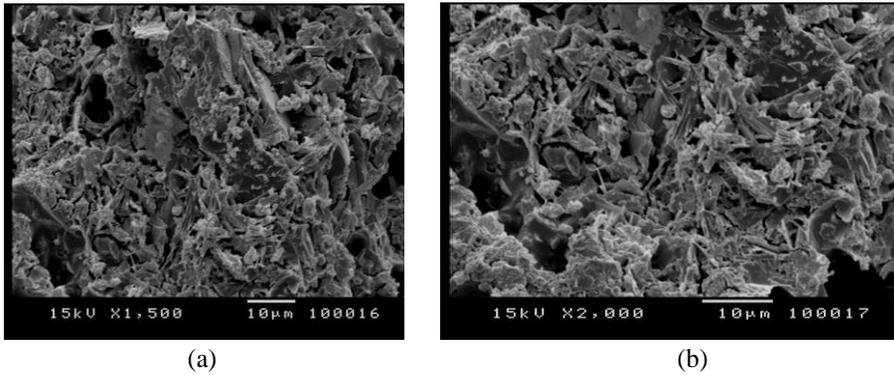
Fig.(15) : IR spectra of mixes (KI-KV) contains fired KK at 1000°C after 90 days curing (1= 3420 cm<sup>-1</sup>, 2= 1654 cm<sup>-1</sup>, 3= 1430 cm<sup>-1</sup>, 4 = 1030 cm<sup>-1</sup>, 5= 874 cm<sup>-1</sup>, 6= 712 cm<sup>-1</sup>, 7= 536 cm<sup>-1</sup>)

*Morphology and microstructure:*

Six samples were investigated using scanning electron microscopy (SEM) as representatives for the hardened MK-lime pastes in this study. Three of these samples were made from mix KI (95% MK: 5% CaO) at the firing temperatures of KK clay 800,900 and 1000°C after 90 days curing; their SEM micrographs are shown in Figs. 16, 18 and 20, respectively. The other three samples were made from mix KIII (75% MK: 25% CaO) at the firing temperature of KK clay 800,900 and 1000 °C after 90 days curing; their SEM micrographs are shown in Figs.17, 19 and 21, respectively. Comparing the SEM micrographs obtained for the specimens of mix KI [Fig.16 (a&b)] and mix KIII [Fig.17 (a&b)] at firing temperature 800°C after 90 days, it is clear that mix KI showed formation of nearly amorphous CSH as well as some cubic crystal of hydrogarnet-like calcium alumino-silicate hydrate, while mix KIII showed formation ill-crystallized and fibrous particles of CSH; which appeared as a clear binder between fibrous clay grains; these hydrates mainly as CSH-(I) were engulfed with small hexagonal particles of Calcium hydroxide. The SEM micrographs of specimens made from mix KI containing burnt KK at 900°C after 90 days indicated formation of irregular hydration products without any interlocking binder [Fig.18 (a&b)]. The micrographs of mix KIII [Fig.19 (a&b)] at the same firing temperature and curing time, showed formation of dense structure and fully crystalline hydrates having close texture structure with interlocking arrangements. Also, there is appearance of hexagonal phases of  $CAH_{10}$  beside mixture of ill-crystallized and amorphous phases which means that these pastes have high hydraulic properties [17]. The SEM micrographs obtained after 90 days curing of specimens made from mix KI containing calcined KK at 1000°C [Fig.20 (a&b)] displayed a dense and massive structure composed of interlocking fibers and crumpled foils as well as particles with irregular appearance. In the same firing temperature and curing time, specimens of mix KIII [Fig.21] displayed mainly ill-crystallized and well-crystallized hydration products; these are a massive calcium silicate hydrates and appreciable amounts of calcium aluminate hydrates ( $C_3AH_{13}$ ) [18]. From the morphology and microstructure studies of MK-lime pastes, it is clear that specimens made from mix KIII (85% MK: 15% CaO) possess the highest hydraulic properties than those made from mix KI (95% MK: 5% CaO) for all firing temperatures of KK and all curing time.. These results are in a good agreement with the obtained physico-chemical measurements of MK- lime pastes.



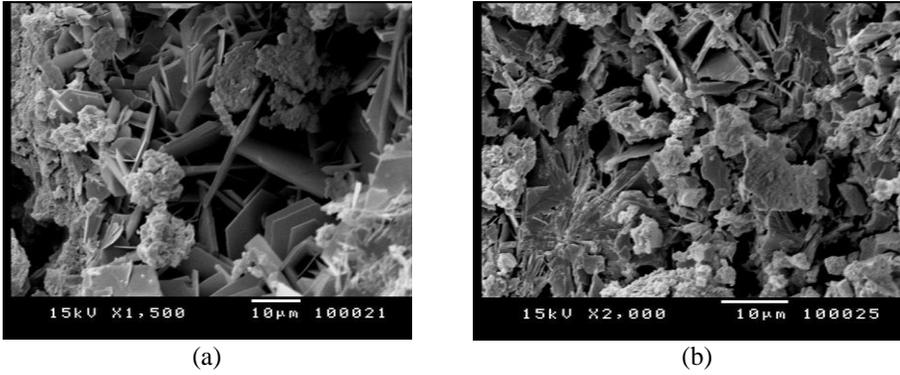
**Fig.(16): SEM micrographs of mix KI contain fired KK at 800°C after 90 days curing**



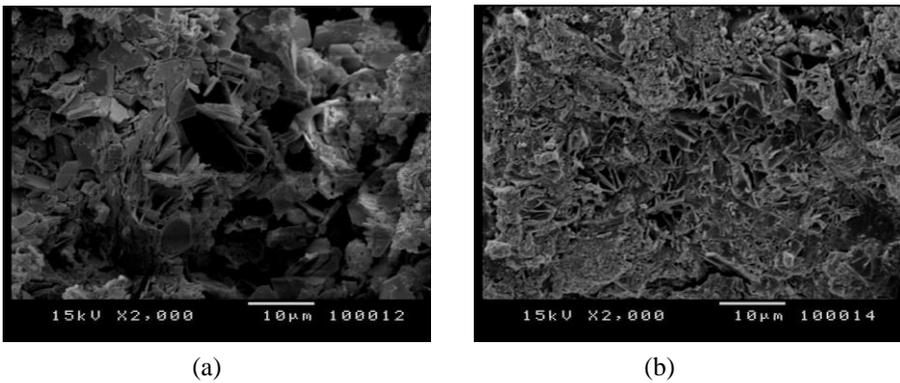
**Fig.(17): SEM micrographs of mix KIII contain fired KK at 800°C after 90 days curing**



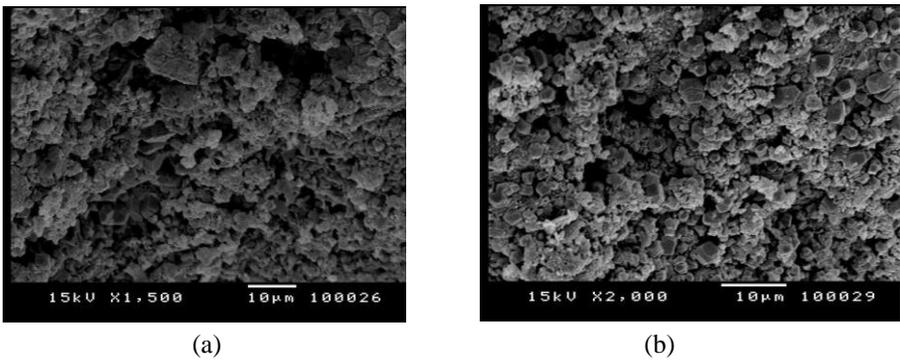
**Fig.(18): SEM micrographs of mix KI contain fired KK at 900°C after 90 days curing**



**Fig.(19): SEM micrographs of mix KIII contain fired KK at 900°C after 90 days curing**



**Fig.(20): SEM micrographs of mix KI contain fired KK at 1000°C after 90 days curing**



**Fig.(21): SEM micrographs of mix KIII contain fired KK at 1000°C after 90 days curing**

**Conclusion:**

- 1- The compressive strength of the various MK-lime pastes increased continuously with increasing age of hydration up to 90 days for all firing temperatures of clay and mix composition. Mix KIII(85% MK:15% lime) has the highest compressive strength values at all curing ages and firing temperatures of clay and the optimum value for this mix was at 900°C after 90 days curing.
- 2- The results of free lime contents indicated that the free lime was gradually consumed with the time of hydration for all MK-lime pastes up to 90 days, which indicate clearly the high pozzolanic reactivity of artificial pozzolana made from burnt Kalabsha kaolinite clay and lime.
- 3- The infrared spectroscopic analysis of some MK-lime pastes indicated the formation of bands at different wave lengths due to the formation of different phases. At 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 KIII (85% MK : 15% CaO) has the hydraulic properties of MK-lime mixes.
- 4- The morphology and microstructure of hardened MK-lime pastes indicated the formation of various hydration products having different degrees of crystallinity. Mixes KI showed formation of nearly amorphous CSH as well as some cubic crystal of hydrogarnet-like calcium alumino-silicate hydrate, irregular hydration products without any interlocking binder and particles with irregular appearance. Mixes KIII showed formation of ill-crystallized and fibrous particles of CSH and fully crystalline hydrates having close texture structure with interlocking arrangements. According to these results; pastes made from mixes KIII (85% MK: 15% lime) show the higher hydraulic properties of prepared artificial pozzolana made from burnt Kalabsha kaolinite clay and lime.
- 5- Finally, we can conclude that, partial substitution of burnt Kalabsha kaolinite clay at 900°C with 15% CaO improves its hydraulic properties and it can be used as building material.

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## الخواص الفيزيوكيميائية لعجائن الميتاكاولين والجير عند درجات حرارة الحرق المختلفة لطفلة الكاولين

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### ملخص البحث

تعتبر دراسة عملية تأثرت العجائن المحتوية على طفلة الكاولين المحروقة (الميتاكاولين) والجير من الدراسات الهامة فى مجال تكنولوجيا مواد البناء. والغرض من هذا البحث هو دراسة الخواص الفيزيوكيميائية للعجائن المحتوية على الميتاكاولين والجير كعامل منشط . وقد تم استخدام الميتاكاولين الناتج من حرق طفلة الكاولين (من منطقة كلابشة بأسوان) عند درجات حرارة 1000 ، 900 ، 800 ، 700 درجة مئوية وذلك لمدة ساعتين كما تم تحضير الجير (CaO) وذلك بـحرق  $CaCO_3$  عند 1000 درجة مئوية. وقد تم تحضير عجائن الميتاكاولين والجير وذلك بالإحلال الجزئى للميتاكاولين بالنسب المئوية الوزنية 5، 10، 15، 20 % من CaO وقد تم تحضير العجائن بالخلط بالماء والصب فى قوالب ثم متابعة عملية التآدرت بالغمر فى الماء حتى 90 يوم . وقد تم دراسة مدى التغير فى الخواص الفيزيوكيميائية للعجائن بقياس قوة الانضغاط والمسامية وكذلك نسبة الجير الحر وذلك بعد ازمئة الغمر المختلفة 3،7،28،90 يوم. كما تم دراسة التغير فى الأطوار المتكونة لبعض العينات باستخدام القياسات الطيفية للأشعة تحت الحمراء (IR spectroscopy) وتم دراسة التغير فى التركيب الدقيق لبعض العينات باستخدام الميكروسكوب الماسح الالكترونى (Scanning electron microscope) .

وقد استنتج من هذه الدراسة أن الإحلال الجزئى لطفلة الكاولين المحروق عند 900 درجة مئوية بنسبة 15% من CaO تحقق أعلى خصائص هيدروليكية لعجائن الميتاكاولين والجير .