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Optimum Metakaolin Content in Cement Pastes

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ABSTRACT

The present paper is directed toward developing a better understanding of the isolated contribution of metakaolin (MK) on pastes and determining its optimum content. An experimental work was carried out over water/binder (w/b) ratios ranging from 0.22 to 0.52 as well as different MK-binder ratios. Cement was partially replaced with MK at levels fluctuated from 0% to 25% with an interval of 5%, by weight. The compressive strength of pastes containing different MK contents at each w/b ratio was measured at age of 28 days. The results was analyzed by thermogravimetric analysis (TGA) and its derivative. The results indicated that the optimum replacement percentage for 28 days compressive strength is not a constant one, but depended on the w/b ratio of the mixture.

Keywords: *Metakoalin, Compressive strength, Water/binder ratio, Replacement level.*

INTRODUCTION

It is well known that pozzolan such as silica fume (SF), fly ash (FA) and blast-furnace slag (slag) are industrial by-products that present great variations of properties, which can sometimes compromise their use. On the other hand, metakaolin (MK) is an artificial pozzolan produced by burning selected kaolinite clay within a specific temperature range of 700- 900 °C [1-3]. The production process is closely controlled and thus higher purity and reactivity can be obtained. MK is basically made up of silica and alumina in an amorphous state, that can react with calcium hydroxide (CH) produced by Portland cement hydration to form calcium hydrosilicate (C–S–H) [4] and calcium hydroalumino silicate (essentially gehlenite – C_2ASH_8) [5].

There is a consensus in the literature that the pozzolanic reaction between MK and CH helps to refine the binder capillary porosity [6] with the direct consequence of improving the mechanical characteristics [7] mainly at early ages [8] and durability e.g. resistance to sulfate attack [9], chloride ingress [10], fire resistance [11,12] and alkali silica reaction [13-15]. But, researchers are not yet to arrive at a unique conclusion regarding the optimum MK replacement level, and different researchers have reported different replacement levels as optimum for obtaining maximum strengths of concrete or mortar or paste [16]. It has been reported that the optimum replacement level is dependent on the cement type and content, type and dosage of admixtures, calcination temperature of kaolin, and also on the age of testing. Strength of cement paste or concrete is considered as an index of its overall quality because an increase in the strength generally leads to an improvement of almost all other properties. Thus, in many of the existing reports, the optimum MK content has been defined with regard to the compressive strength of cement paste or concrete. In this work investigation, the optimum MK replacement level refers to the replacement level that leads to the maximum 28 days compressive strength of cement pastes. The experimental program was designed to determine the isolated effect of MK on cement pastes by keeping other mixture proportion variables constant. Thus, the change in paste strength at any constant w/b ratio occurred due to MK incorporation only.

EXPERIMENTAL DETAILS

Materials

Portland cement, (PC) CEM I: 42.5N, of Blaine surface area 335 m^2/kg and confirming to the Egyptian Standard Specifications (ES 4756-1/ 2006) [17] was used. A sample of kaolin clay collected from Sina quarry with a Blaine specific surface area of 350 m^2/kg was used. The MK used in this research produced by calcining kaolin at a temperature of 850 °C for 2 h. Fig. 1 shows the diffractograms of kaolin and

MK samples analyzed by X-ray diffraction. The details of the chemical and physical properties of PC and MK are given in Table 1.

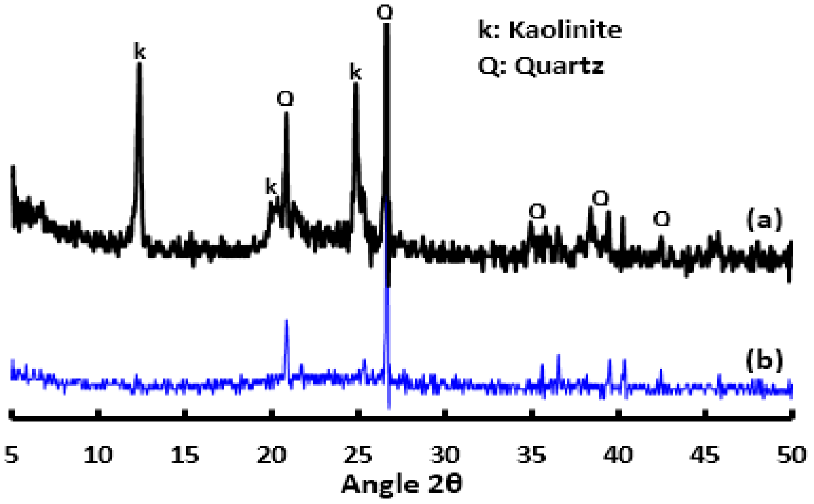

Fig1. *XRD patterns of raw micro-size kaolin (a) and micro-size MK (b)*

Table1: *Chemical composition and physical properties of cementitious materials*

Chemical composition (%)	PC	MK
SiO_2	20.39	58.52
Al_2O_3	5.6	35.54
Fe_2O_3	3.43	1.15
CaO	63.07	1.24
MgO	2.91	0.19
Na_2O	0.38	0.25
K_2O	0.35	0.05
SO_3	0.7	0.06
C_3A	9.04	-
P_2O_5	-	0.09
TiO_2	-	0.04
CaO	-	
L.O.I	2.06	2.74
Physical properties		
Specific gravity	3.15	2.34
Specific surface area (m^2/kg)	335	350

Mixture Proportions

The main objective of this research investigation is to combine different w/b ratios with varying levels of MK in cement pastes to determine the optimum values of each. For this purpose, the experimental program included seven sets of paste mixtures, at w/b of 0.22, 0.27, 0.32, 0.37, 0.42, 0.47 and 0.52 prepared by partial replacement of cement by equal weight of MK. Each set had mixtures at six different levels of MK replaced PC. Cement was replaced with MK at levels of 0%, 5%, 10%, 15%, 20% and 25%. Table 2 refers to the details of mixing proportions of different designed pastes.

Table2: *Details of mixture proportions*

Mix	PC (%)	MK (%)	W/b
M-0.22-0	100	0	0.22
M-0.22-5	95	5	
M-0.22-10	90	10	
M-0.22-15	85	15	
M-0.22-20	80	20	
M-0.22-25	75	25	
M-0.27-0	100	0	0.27
M-0.27-5	95	5	
M-0.27-10	90	10	
M-0.27-15	85	15	
M-0.27-20	80	20	

M-0.27-25	75	25	
M-0.32-0	100	0	0.32
M-0.32-5	95	5	
M-0.32-10	90	10	
M-0.32-15	85	15	
M-0.32-20	80	20	
M-0.32-25	75	25	
M-0.37-0	100	0	0.37
M-0.37-5	95	5	
M-0.37-10	90	10	
M-0.37-15	85	15	
M-0.37-20	80	20	
M-0.37-25	75	25	
M-0.42-0	100	0	0.42
M-0.42-5	95	5	
M-0.42-10	90	10	
M-0.42-15	85	15	
M-0.42-20	80	20	
M-0.42-25	75	25	
M-0.47-0	100	0	0.47
M-0.47-5	95	5	
M-0.47-10	90	10	
M-0.47-15	85	15	
M-0.47-20	80	20	
M-0.47-25	75	25	
M-0.52-0	100	0	0.52
M-0.52-5	95	5	
M-0.52-10	90	10	
M-0.52-15	85	15	
M-0.52-20	80	20	
M-0.52-25	75	25	

Methods

The cement and certain quantity of MK were mixed in laboratory. The mixed powders were added to the water, previously placed in the bowl of Hobart planetary mixer, over 3 min period after which time the mixer was stopped and any unmixed powders was scraped from the sides and the paddle into the mixing bowl. Mixing was then continued for extra 2 min before casting into 20 × 20 × 20 mm lubricated metallic moulds and vibrating for 1 min to remove air voids. The same mixing regime was used for neat PC pastes. Immediately after casting, the samples were sealed by plastic sheets to prevent water loss for 24 h and then specimens were removed from the moulds and cured in water tank for 28 days. All the specimens were cured by immersion in water at room temperature until testing. The curing tank was located at the interior of the laboratory. The temperature of the water in the curing tank has a constant temperature of 30 ± 1 °C. All the specimens were cured in the same curing tank. Because the curing condition was uniform for all the specimens, no adverse effects on the subsequent test results were expected due to temperature variations.

Compressive strength measurements of the pastes were performed in accordance with ASTM C109/C109 M-16a where the strengths were determined on 4 paste cubes of each mixture at age of 28 days. After compressive strength testing, the selected crushed samples were stored in acetone for three days aiming to stop the hydration. Then, the crushed samples were filtered and dried in the desiccators under vacuum. The dried samples was ground and prepared for thermogravimetric analysis (TGA).

RESULTS AND DISCUSSION

The 28 days compressive strength values as obtained for the 42 paste mixtures are presented in Fig 2. The strength values at different w/b ratios have been plotted at each MK replacement level. The results indicated that the MK as well as w/b content directly influence the compressive strength. It was clearly evident that most replacement levels of MK have a positive effect on the compressive strength values at all w/b ratios. When w/b ratio of 0.22 was used, the incorporation of 5%, 10% MK gradually increased the compressive strength by 8% and 12.4%, respectively. Increasing replacement of cement with MK at level of 15% led to further compressive strength improvement, of which it reached its highest value with an enhancement of 15%. Further increasing replacement levels of cement by 20% and 25% MK resulted in a reduction in the compressive strength. When w/b ratio of 0.27 or 0.32 was used, the same trend of the results was observed, of which the compressive strength gradually increased with increasing MK content up to 15%, then decreased when MK content increased to 20% and 25%.

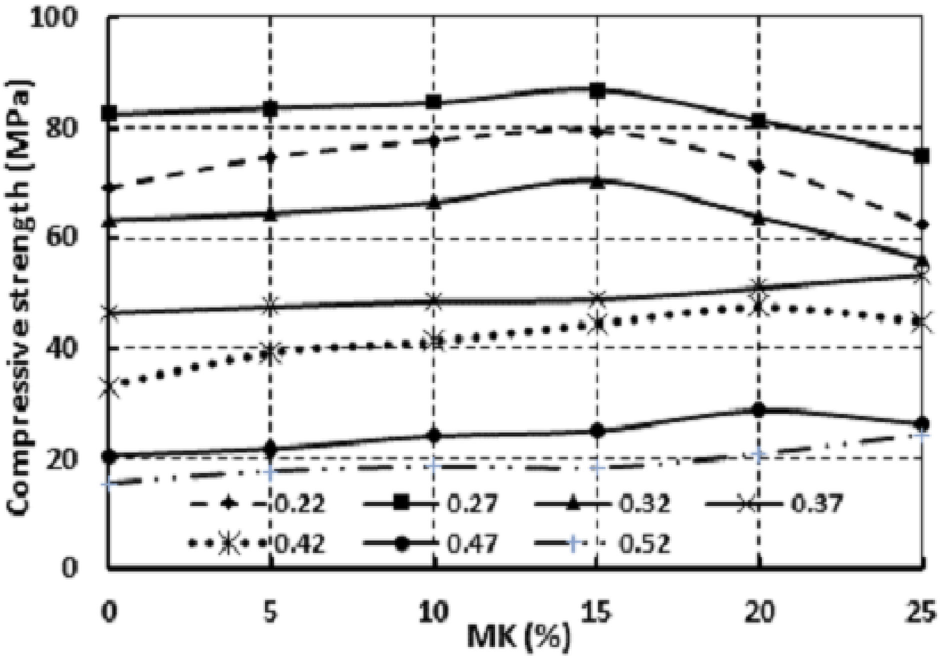

Fig2. *Relationship between 28 days compressive strength and replacement levels of MK at various w/b ratios*

In the case of using w/b ratio of 0.37, an essential change in the optimum content of MK that exhibited the highest compressive strength was observed. The incorporation of 25% MK exhibited the highest compressive strength. The same trend of the results was observed when w/b ratio was 0.52. Whatever, in this case the enhancement in the compressive strength with the incorporation of 5%, 10%, 15%, 20% and 25% MK enhanced the compressive strength by 2.5%, 4.5%, 5.2%, 9.7% and 14.9%, respectively. When w/b ratio of 0.42 or 0.47 was used, an enhancement in the compressive strength at all replacement levels of MK was observed, but the optimum content was 20%. The main features of Fig. 2 are that at the same conditions, the optimum content of MK that exhibited the highest compressive strength depending on the w/b ratio. Furthermore, the compressive strength decreased with increasing w/b ratio.

Fig. 3 shows the weight loss and its derivative curves, in the range of 25-1000 °C for M-0.37-0 which free from MK. As can be seen, the endothermic peak located below 200 °C is attributed to moisture loss, combined water loss came initially from C-S-H. Large endothermic peak located between 400-600 °C can be observed. This peak is attributed to the decomposition of $Ca(OH)_2$. It can be noted that these is an endothermic peak located at 700-800 °C which is attributed to the decomposition crystalline phase of $CaCO_3$. Fig. 4 shows the TGA/DTG curves of M-0.37-5 which containing 5% MK. As can be seen, in addition to the peak of C-S-H, the peak of stratlingite (C_2ASH_8) localized at about 160-190 °C can be observed. This peak resulted from the pozzolanic activity of MK. The endothermic peak located between 400-600 °C is attributed to the decomposition of $Ca(OH)_2$, but the intensity of this peak is smaller than those of M-0.37-0 which confirming the pozzolanic activity of MK, of which some of the CH released from cement hydration can be consumed by the pozzolainc activity of MK. Finally, the crystalline phase of $CaCO_3$ can be observed through the endothermic peak located between 700-800 °C. Similar peaks identifications for M-0.37-15, M-0.37-20 and M-0.37-25 presented in Figs 5,6,7, respectively, were obtained, but the CH peak decreased with increasing MK content to reach its lowest intensity when MK content was 25% (Fig. 7). In addition the peaks of C-S-H and C_2ASH_8 slightly increased with increasing MK content. These confirming the pozzolanic activity of MK.

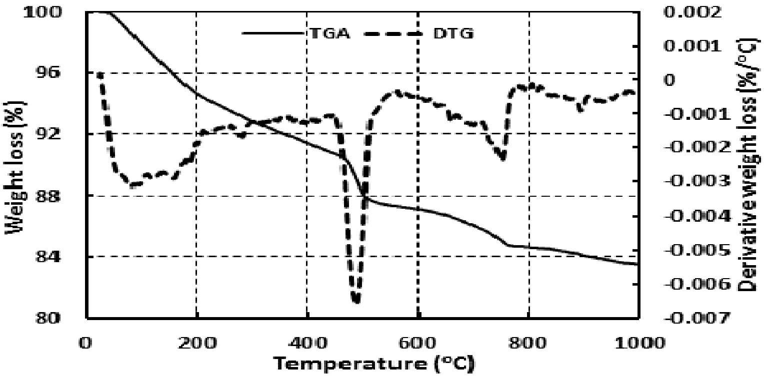

Fig3. *TGA/DTG curves for M-0.37--0 after 28 days of hydration*

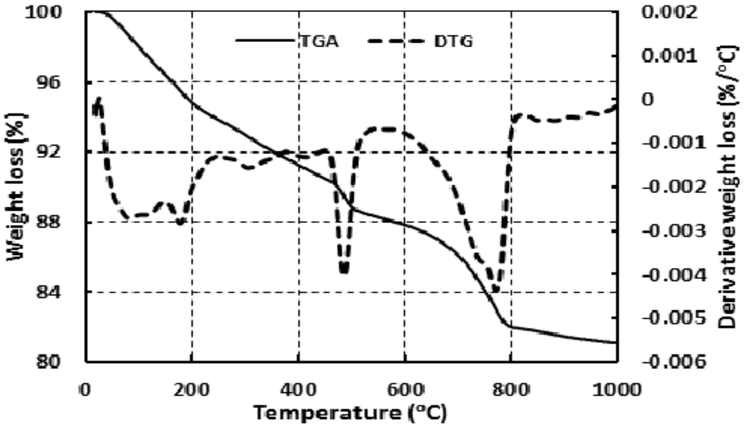

Fig4. *TGA/DTG curves for M-0.37--5 after 28 days of hydration*

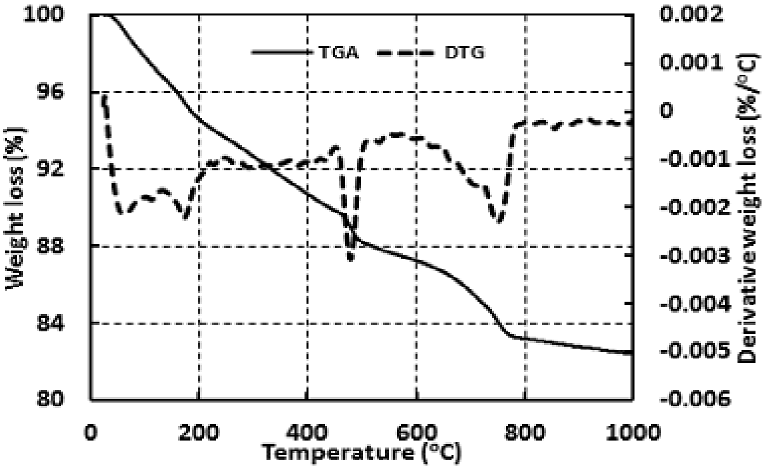

Fig5. *TGA/DTG curves for M-0.37--15 after 28 days of hydration*

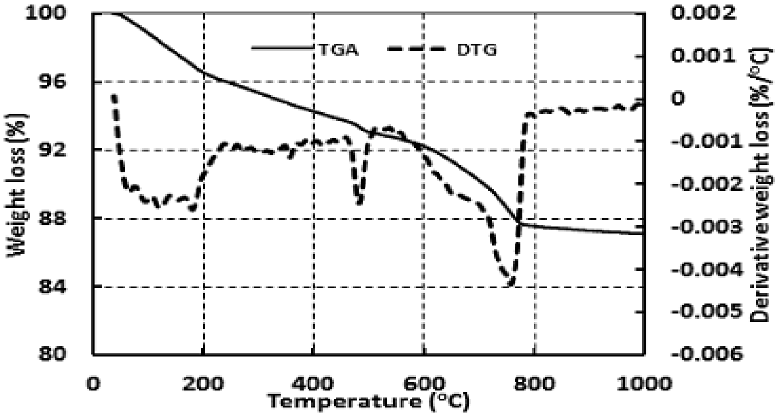

Fig6. *TGA/DTG curves for M-0.37--20 after 28 days of hydration*

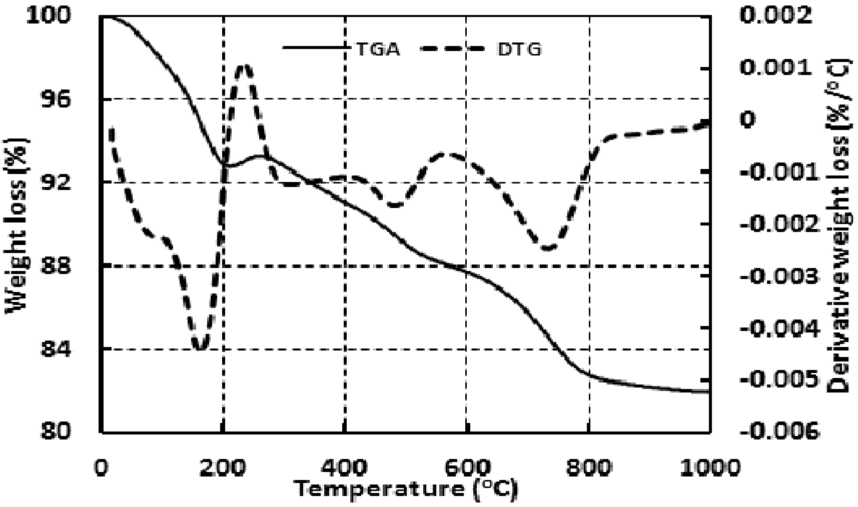

Fig7. *TGA/DTG curves for M-0.37--25 after 28 days of hydration*

In short, the improvement in the compressive strength with the incorporation of MK is attributed to its filling effect as well as its pozzolanic reaction with CH. MK can reach with portlandite released from cement hydration to form C-S-H supplementary to that produced by cement hydration. The elimination of CH and excess C-S-H is the key of increasing compressive strength and durability. Furthermore, MK strongly influences the pore structure in the matrix by pore refinement, leading to significant delays in the transport of harmful substances. In addition, the optimum content of MK is not fixed, but changed according to w/b ratio.

CONCLUSIONS

The present paper studies the effect of MK on cement pastes compressive strength over a wide range of w/b ratios fluctuated from 0.22 to 0.52 and cement replacement levels from fluctuated from 0% to 25%. The optimum MK replacement level for each w/b ratio is determined at age of 28 days. The following conclusions can be drawn from the results presented in this paper:

1. The results of the present investigation indicate that other mixture proportioning parameters remaining constant, the optimum MK replacement level is not a constant one, but a function of the w/b ratio of the mixture
2. Both the filler and pozzolanic effects of MK are highly significant in improving the compressive strength.
3. The TGA/DTG curves confirm that the optimum MK content can completely consume the portlandite released during the hydration of cement producing more amount of C-S-H (the main source of improving compressive strength).

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