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Research paper

An investigation on alkali-activated Egyptian metakaolin pastes blended with quartz powder subjected to elevated temperatures

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ABSTRACT

In this study, the possibility of using quartz powder (QP) to improve the workability as well as the compressive strength of alkali-activated metakaolin (AAMK) paste before and after exposure to elevated temperatures has been investigated. Metakaolin (MK) was partially replaced with QP at levels ranging from 0% to 30% with an increment of 5%, by weight. After curing, specimens were exposed to different elevated temperatures ranging from 400 °C to 1000 °C with an interval of 200 °C for 2 h. Weight and compressive strength of specimens before and after being exposed to different elevated temperatures were thoroughly explored. The various decomposition phases formed upon exposure to temperatures were identified using X-ray diffraction (XRD)-phase characterizations and thermogravimetric (TGA)-thermal characterizations. The microstructure of the formed hydration products was determined using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS). The results showed that the workability as well as compressive strength before and after being exposed to elevated temperatures increased with increasing QP content. The geopolymer formulations developed in this study appeared as promising candidates for high-temperature applications refractory and fire resistant.

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1. Introduction

Geopolymers are new environment friendly inorganic binders, produced by alkaline solutions activating aluminosilicate source materials. Geopolymers and other types of alkali-activated materials could be classified as the third generation cement after lime and Portland cement (PC) (Rashad, 2013a), which are a way to reduce carbon dioxide lunched into the atmosphere (Rashad, 2013b; Rashad, 2014a). Due to the low energy requirement in the production and good mechanical performance, thermal behavior and durability, geopolymers have attracted increasing interest as ecologically friendly fireproof building materials.

Metakaolin (MK), derived from dehydroxylation of kaolin upon thermal treatment, is one of the main common aluminosilicate precursors source material which used in the production of geopolymers. Also, less pure clay and, so, recall the major “family” of calcined clays can be used (Molino et al., 2014; Poowancum and Horpibulsuk, 2015; Pierce et al., 2015). There are several studies in the literature regarding to heating of MK-based geopolymers at elevated temperatures have been carried out, but some studies in this field contradict the others. Some studies reported positive effect of elevated temperatures on MK-based geopolymers such as Lin et al. (2009); Kuenzel et al. (2013) and He et

al. (2010, 2015). On the contrary, other studies reported negative effect, of which Fletcher et al. (2005) reported that geopolymers with higher Si/Al ratio exhibited deteriorated mechanical properties and irregular volume expansion at elevated temperatures. Elimbi et al. (2014) reported a reduction in the initial compressive strength of AAMK pastes after being exposed to 300–900 °C. Zhang et al., (2014a) reported a reduction in the bonding strength of AAMK paste, mortar and concrete after being exposed to 100–800 °C. Whatever, These contradictory results could be related to the change in MK particle size, MK purity, the crystallinity of the kaolinite of which MK was derived, alkaline composition, alkaline concentration, SiO₂/Al₂O₃ ratio, curing conditions etc.

One option to improve some special properties of MK-based geopolymers is to replace part of MK with other materials such as slag, fly ash (FA), micro-sized particles and other supplementary materials. Kamsen et al. (2010) reported that the inclusion of alumina (size 0.1–1 μm) in MK pastes activated with potassium hydroxide and potassium silicate solution can reduce the total shrinkage at 1000 °C from 17% to 12%. van Riessen (2007) reported 1% reduction in the thermal shrinkage with the inclusion of granite filler in MK pastes activated with sodium silicate and NaOH solution after being exposed to temperatures up to 800 °C. Vidal et al. (2015) reported that the inclusion of ammonium molybdate up to 1.57% in MK matrix activated with KOH and amorphous silica decreased the shrinkage at high temperature and increased the temperature of crystallization. Barbosa and MacKenzie (2003) reported that the inclusion 10–20 wt% filler such as kaolinite (size

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<6 μm) and β -sialon (size <6 μm) in MK activated with sodium silicate and NaOH solution exhibited good thermal behavior than the neat AAMK one at elevated temperatures up to 1000 °C. Zhang et al. (2015) prepared reflective heat insulation coating from geopolymer, which was made of MK activated with sodium silicate solution. They modified the MK geopolymer with some additives such as titanium dioxide and hollow glass microspheres. The results showed that adding 12%, by weight, of titanium dioxide to the coating had a positive effect on the application, mechanical, optical and heat insulation properties of the coating. Using 12% titanium dioxide determined the heat insulation effect was best when 6% of microspheres were used. Roviello et al. (2015) reported that the inclusion of epoxy melamine resins in AAMK specimens produced not flammable composites. These composites did not produce smoke in significant amount. On the other hand, Richard William et al. (2013) reported that un-foamed AAMK specimens exhibited higher fire rating than those foamed ones (containing aluminium powder).

Kouamo et al. (2012) reported that the inclusion of 20% alumina-oxide (Al_2O_3) in MK paste activated with sodium silicate and NaOH solution increased the compressive strength by 18.1%. Kamseu et al. (2014) reported that the addition of ladle slag and nepheline syenite in the form of fine powder with Blaine surface area of 12,450 and 21,980 m^2/kg , respectively, to fresh MK-based geopolymer cement in different proportions of 20%, 40% and 60%, by weight, increased the three-points bending strength. The bending strength increased as the content of nepheline syenite increased, whilst the addition of 40% ladle slag exhibited the highest bending strength followed by 20% and 60%, respectively. Yip et al. (2008) reported that the compressive strength of AAMK mortars increased by partially replacing MK with 20% calcite, by weight. Burciaga-Díaz et al. (2010) partially replaced MK with 50% slag in MK pastes activated with sodium silicate and NaOH solution at different Na_2O concentrations of 5%, 10% and 15%. The results showed higher compressive strength with the inclusion of slag, at all concentration levels, compared to the neat AAMK ones. Zhang et al. (2014b) reported that the compressive strength of MK pastes activated with NaOH and sodium water glass solution increased by partially replaced MK with 10% FA, by weight.

According to the literature, the effect of elevated temperatures on compressive strength of AAMK paste blended with QP has not yet been reported so far. Therefore, the prime objective of the current investigation is to study the behavior of AAMK pastes containing different levels of QP before and after exposure to elevated temperatures, and then comparing them with that blends of free QP. In the current study the workability as well as compressive strength before and after firing of alkali-activated MK/QP pastes have been investigated and compared with the neat AAMK paste. MK was partially replaced with QP at levels ranging from 0% to 30% with an increment of 5%, by weight. The specimens were exposed to elevated temperatures ranging from 400 °C to 1000 °C with an increment of 200 °C for 2 h. The current results are intended to add new and valuable development to the field of fire resistance of AAMK system.

2. Experimental details

The source raw MK used in this investigation was produced by calcining kaolin, which brought from Sinai quarry (Northwest Egypt), at temperature of 850 °C for 2 h to enable the formation of amorphous glassy phases (Rashad, 2013c, 2015a). The Blaine surface area of the MK was 460 m^2/kg . The QP was brought from the Egyptian Company for Manufacturing Electrical Insulation. The specific weight of the QP was 2.46, whilst its bulk density was 1.04 g/cm^3 . Table 1 presents the sieve analysis of the QP. The chemical composition of the MK and QP as evaluated by X-ray fluorescence (XRF) spectrometry is presented in Table 2. Liquid sodium silicate which had a density of 1.38 g/cm^3 and a composition comprising of 8.2% Na_2O , 27% SiO_2 and 64% H_2O was used as an alkaline activator. Fig. 1 presents the X-ray diffraction

Table 1
Sieve analysis of QP.

Residue on sieve %	Particle size distribution	
90 μm < = 1	> 90 μm	1
63 μm 2–4	63–90 μm	1–3
45 μm 7–10	45–63 μm	5–6
32 μm 15–20	32–45 μm	8–10
	< 32 μm	80–85

(XRD) analysis results of the raw MK and QP. The wide diffusive amorphous hump between 20° and 30° 2 θ indicates that the MK is mostly amorphous (Fig. 1a). Refractions for quartz and kaolinite were identified. Peaks of quartz were detected in the raw QP (Fig. 1b).

2.1. Mixture proportions

Seven alkali-activated mixtures were prepared. The first mixture was prepared from the neat MK without any addition of QP. This mixture was denoted as QP0. The remaining six mixtures were prepared by partially substituting MK with different ratios of QP ranging from 5% to 30% with an increment of 5%, by weight. These mixtures were denoted as QP5, QP10, QP15, QP20, QP25 and QP30, respectively. Fixed concentration of liquid sodium silicate dosage of 30 wt% from total powders weight was employed to active each mixture. This fixed dosage of activator was chosen to avoid any effect of activator on strength, of which solids-to-liquid affecting the strength as reported by Liew et al. (2012) and Rashad et al. (2016). The water binder (w/b) ratio was fixed for all mixtures at 0.10, by weight. The mixture proportions are presented in Table 3.

2.2. Methods

The starting source supplementary materials were formulated with MK/QP ratios of 100/0, 95/5, 90/10, 85/15, 80/20, 75/25 and 70/30, by weight. The starting materials were mixed for 4 min in mechanical mixer with a speed of 80 rpm to ensure homogenous mixing. The solution of sodium silicate was prepared by dissolving in mixing water then added and mixed with the MK/QP for 5 min, followed by 2 min resting period to scrape off any unmixed powders from the paddle sides of mixer and adding them into the mixing bowl. The mixing was continued for additional 5 min before casting. After complete mixing, the flow value of each mixture was measured and recorded using hand-driven flow table with 10" diameter cast-bronze table/platen (Fig. 2). Included standard H-3622 cast bronze, 2–3/4" top diameter \times 2" high \times 4" bottom diameter cone mould according to ASTM C230-83. Then, the fresh paste was poured into 20 mm cube moulds and vibrated for 1 min to remove air bubbles. Immediately after casting, the moulds were covered with polyethylene sheet to avoid any evaporation. The specimens were cured at 50 ± 1 °C for 7 days then at room temperature.

Table 2
Chemical composition of the starting materials.

Oxide composition	MK (%)	QP (%)
SiO_2	58.52	98.81
Al_2O_3	35.54	0.14
Fe_2O_3	1.15	0.05
CaO	1.24	0.51
MgO	0.19	0.02
Na_2O	0.25	0.04
K_2O	0.05	0.03
SO_3	0.06	0.03
TiO_2	0	0.02
P_2O_5	0	0.32
MnO	0	0
L.O.I.	2.74	0.32

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