

Research paper

Effect of alkali-activated metakaolin cement on compressive strength of mortars

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ABSTRACT

This paper reports the compressive strength and microstructure characteristic of alkali-activated metakaolin (MK) cement under two curing methods. MK was used to replace part of Portland cement (PC) at 70, 80, 85, 90, 95 and 100% by mass of binder. Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were used as activating agents (Na_2SiO_3 to NaOH ratio of 0.67). NaOH with 10 molar concentrations, liquid alkali/binder ratio of 0.8 and water base was used. Mortar and paste specimens were cured at $23 \pm 2^\circ\text{C}$ (55% RH) and 60°C (95% RH). The results indicated that calcium silicate hydrate (C–S–H) and calcium carbonate (CaCO_3) phases were detected by thermogravimetry (TGA). Setting time and drying shrinkage decreased when metakaolin replacement level increased, the compressive strength of alkali-activated metakaolin cement increased with the increase of PC content and increased with temperature. The curing of specimens at 60°C (95% RH) enhanced the compressive strength, matrices appeared denser than when cured at $23 \pm 2^\circ\text{C}$ (55% RH) and phases of (C, N)–A–S–H gel and or C–S–H gel can be seen clearly.

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1. Introduction

Portland cement production is a high energy demand process (Puertas and Torres-Carrasco, 2014) that emits 900 kg CO_2 into the atmosphere per tonne of cement (Benhelal et al., 2013) which contributed to global warming and climate change. Therefore, in looking for ways to reduce CO_2 emissions associated with cement production (Gartner, 2004) by the use of supplementary cementitious materials (SCMs) to replace part of Portland cement. Common SCMs include metakaolin, another pozzolan material which is produced from selected kaolins after customizing and calcination under specific conditions (Courard et al., 2003). MK are high reactivity (Wang et al., 2012) improves concrete mechanical properties and durability (Rashad, 2013).

“Geopolymer” is an alternative cementitious binder, defined by Davidovits (1979) as alkali activators react with source of silicon (Si) and Aluminium (Al) to become cementitious. Hence, geopolymer is fundamentally aluminosilicate inorganic polymers consist of chains with three dimensional replicate structures of $(\text{Si}-\text{O}-\text{Al}-\text{O})_n$ and $(\text{Si}-\text{O}-\text{Al}-\text{Si}-\text{O})_n$ units (Hermann et al., 1999; Barbosa et al., 2000). Alkaline activators used for geopolymers are usually combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate (Davidovits, 1991; Palomo et al., 1999; Xu and Van Deventer, 2000; Barbosa and MacKenzie, 2003; Hardjito et al., 2004;

Duxson et al., 2007). Typically NaOH and sodium silicate are used due to cost and availability to accomplish comparable strength to PC concrete (Palomo et al., 1999; Hardjito et al., 2004; Duxson et al., 2007).

Curing is very important for cement, mortar or concrete specimens, which influence the strength and durability development (Wongkeo et al., 2013). Similarly, geopolymer requires the sufficient curing to achieve high mechanical properties and elevated durability (Aredes et al., 2015; Slaty et al., 2015). Metakaolinite - based geopolymers treated at 60°C for 24 h were found to have lower porosity and higher compressive strengths compared to specimens cured at 30°C (Muñiz-Villarreal et al., 2011; Kuenzel et al., 2013). Curing temperatures in between 50 and 80°C are widely accepted values used for successful geopolymer hydration (Kurtz and Balaguru, 2001; Bell et al., 2005; Fernández-Jiménez et al., 2008; Petermann et al., 2010; Menna et al., 2013; Bing-hui et al., 2014; Villaquirán-Cacedo et al., 2015). Rovnaník (2010) reported that the strength developments by curing treatment of fresh mixture at higher temperatures of 60°C and 80°C were accelerated at early ages but the 28 day mechanical properties were deteriorated in comparison with the mixtures those were treated at an ambient or at lower temperatures.

Very little research work has been conducted to study alkali activated cement, especially metakaolin and with different amount of Portland cement. Alkali-activated cement produced from a combination of metakaolin and Portland cement. Therefore, the objectives of this research work was to investigate the initial setting time, compressive strength, drying shrinkage, characteristics of alkali activated metakaolin

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cement obtained in curing conditions at $23 \pm 2^\circ\text{C}$ (55% RH) and 60°C (95% RH).

2. Materials and methods

2.1. Materials

Chemical and physical characteristics of Portland cement and metakaolin used are given in Table 1. Portland cement (ASTM type I) was used with the particle size distribution plotted as shown in Fig. 1 (median particle size = $14.39 \mu\text{m}$). Metakaolin supplied by MetaMax (Centasia Co., Ltd.) with median particle size of $2.55 \mu\text{m}$ was used. Scanning electron micrographs of Portland cement and metakaolin particles are shown in Fig. 2 where the plate-like metakaolin particles can be seen having the particle size in the range of $0.5\text{--}9 \mu\text{m}$. The activators used in the experiment were prepared by mixing 10 M NaOH (97% purity from RCI Labscan Co. Ltd.) with Na_2SiO_3 solution (8.2% Na_2O , 27% SiO_2 and 64.8% H_2O , $\rho = 1.38 \text{ g/cm}^3$) from PANREAC S.A. and Ottawa sand from TEQ equipment limited partnership.

2.2. Specimens preparation

In this study, PC was used to replace part of MK at 0, 5, 10, 15, 20 and 30% by mass of binder. $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios of 0.67 and water base were adopted in this study (see Table 2 for mix proportions). The activator solutions (NaOH + Na_2SiO_3) were prepared 24 h prior to use, due to high heat released dissolving sodium hydroxide flakes. Mortars were prepared by mixing PC + MK with activators solution and Ottawa sand (sand to binder ratio of 2.75 was used, see Table 2) for 4 min (a: low speed, 1.5 min; b 1.5 min stopping to rest and homogenize; c: high speed, 1 min) (Pelisser et al., 2013) using a mortar mixer then homogeneous mortar was cast into $50 \times 50 \times 50 \text{ mm}$ cube molds ASTM C 109/C109M (ASTM standard, 2005) and $25 \times 25 \times 285 \text{ mm}$ ASTM C 596 (ASTM standard, 2009), compacted using a vibrating table. All the mortar specimens were cured at $23 \pm 2^\circ\text{C}$ (55% RH) and 60°C (95% RH) after were stored in molds for 72 h due to adhesion of binder and aggregate (sand) was not complete, cause fracture of specimens easily when demolds previous 72 h and then compressive strengths of alkali-activated metakaolin cement mortars were tested at the ages of 3, 7, 28, and 60 days (three samples were investigated for each age) and drying shrinkage were tested at 1 day (initial value), 7, 14 and 28 days (four samples were investigated for each age). Initial setting time of mortar was measured with a Vicat apparatus, as described in ASTM C 191 (ASTM standard, 2004).

The microstructural characteristics and mineralogical of materials were studied by SEM/EDX and X-ray diffraction (XRD) respectively. JEOL JEM-5910LV Scanning Electron Microscope (SEM) was used for microstructural characterization. Thermogravimetry (TG) and derivative thermogravimetric (DTG) were carried out using a Mettler-

Table 1
Chemical and physical characteristics of Portland cement and metakaolin.

Chemical	Component (%)	
	Portland cement	Metakaolin
SiO_2	19.13	51.30
Al_2O_3	4.98	44.42
Fe_2O_3	3.20	0.42
CaO	64.47	0.05
MgO	1.88	<0.01
K_2O	0.61	0.14
Na_2O	0.09	0.33
TiO_2	–	1.69
SO_3	3.23	0.13
LOI	2.30	1.30
Specific gravity	3.15	2.40
Median particle size (μm)	14.39	2.55

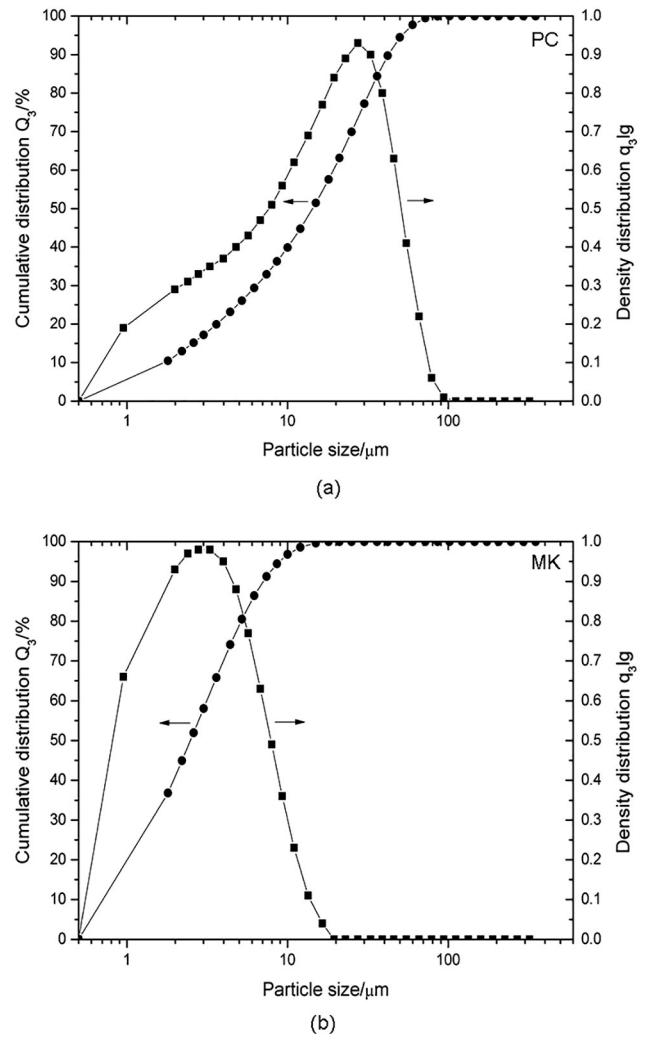


Fig. 1. Particle size distribution of (a) Portland cement and (b) metakaolin.

Toledo TGA/SDTA 851^e. The specimens were heated from 25 to 1000°C at a heating rate of $20^\circ\text{C min}^{-1}$ under nitrogen atmosphere.

3. Results

3.1. Setting time

The setting time tests were carried out in a controlled temperature of $23 \pm 2^\circ\text{C}$ (55% RH). Fig. 3 shows the setting time of alkali-activated metakaolin cement. The initial and final setting time of alkali-activated metakaolin cement pastes is influenced by the Portland cement content, as Portland cement replacement level increased, setting time decreased. This is due to amount of calcium in Portland cement (Nath and Sarker, 2015) but with metakaolin replacement level at 100%, setting time decreased. This is due to high fineness of metakaolin (Badogiannis et al., 2005).

3.2. Density

The density of alkali-activated metakaolin cement mortar specimens cured at $23 \pm 2^\circ\text{C}$ (55% RH) is shown in Fig. 4. At this curing condition, content of PC significantly affects the density of PC-MK mixes and tended to increase with increased amount of PC. This is due to PC reaction with water base liquid resulting to hydration products. The hydration products improve pore structure due to water-filled capillary porosities (Neville, 1995) and there may not be sufficient alkali

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