

Potassium alkali concentration and heat treatment affected metakaolin-based geopolymer



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HIGHLIGHTS

- Using KOH and K_2SiO_3 as alkali solution instead of NaOH and Na_2SiO_3 .
- Very high concentrations of KOH in used.
- Heat-treated at 550 °C.

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ABSTRACTS

Geopolymers prepared using KOH and K_2SiO_3 alkali solution instead of the more common NaOH and Na_2SiO_3 are reported. The influence of KOH concentration, curing temperature and heat treatment on the properties of metakaolin-based geopolymers were studied. The KOH concentrations were varied at 6, 8, 10, 20, 30 and 40 M, the K_2SiO_3 /KOH ratios used were 1 and 1.5 and curing temperatures of 40 °C and 60 °C for 24 h were applied. Results showed that geopolymer with 10 M KOH, cured at 40 °C, 24 h and heat treated at 550 °C gave the highest compressive strength at 28 days. The heat treatment process caused the porosity of geopolymer increased because water was eliminated from the geopolymer structure by the applied heat, therefore, the density decreased whereas the water absorption and the porosity of geopolymer specimens increased. The microstructure of heat-treated specimens showed more mature geopolymer matrix than that of non heat-treated ones leading to higher compressive strength in the former geopolymers and had ceramic-like property.

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1. Introduction

Alkali activation of a large number of natural aluminosilicates as well as slag, mine tailings and industrial by-products generate an interesting sustainable cold-setting cement [1,2]. Among these aluminosilicate starting materials, metakaolin has been frequently used as a reference system to easily investigate consolidation reaction [3,4]. The resultant alternative binders, primary used in the construction industry, are also known as geopolymers or geopolymeric binders [1] and are prepared by mixing the metakaolin solid powder, or another aluminosilicate rich in silica and alumina, and alkali activators such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3) or potassium sili-

cate (K_2SiO_3). In general, sodium based alkali activators are commonly used due to their availability, low cost and high reactivity [5], but potassium hydroxide and silicate are preferable for high temperature applications [6–9].

Metakaolin is a popular source material used in the production of geopolymer since it can solidify at ambient temperature and gives high compressive strength [1,10–14]. Kaolin is transformed to metakaolin after calcination at 550–800 °C [9,15–18]. The optimum calcination condition of Thai kaolin is 600 °C for 2 h [12]. The strength of geopolymer is dependent on the concentration of KOH or NaOH as it affects the dissolution of Si^{4+} and Al^{3+} ions in metakaolin particles. The compressive strength thus increases with increasing alkali activator concentration. Consequently, the formation of the geopolymer structure is generated by these ions [15]. However, Chindaprasirt et al. reported that the compressive strength of geopolymers with variations of NaOH concentration

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(10, 15 and 20 M) was not significantly different [10]. The common alkali activators are the mixtures of sodium hydroxide or potassium hydroxide and sodium silicate or potassium silicate [19]. A number of findings indicates that the use of sodium hydroxide and sodium silicate give better performance in terms of strength development than the potassium activators. However, Hounsi et al. [20] varied the concentration of NaOH and KOH from 4 to 12 M and they reported that at the same concentrations of NaOH and KOH, compressive strength of potassium based geopolymers are higher than that of sodium based geopolymers. In addition, potassium based geopolymers are popular for high temperature applications. Davidovits [21] suggested the ratios of $M_2O/SiO_2 = 0.25\text{--}0.28$, $SiO_2/Al_2O_3 = 4.0$, $H_2O/M_2O = 16\text{--}17.5$, and $M_2O/Al_2O_3 = 1.0\text{--}1.14$ to obtain geopolymers with good properties. Khale and Chaudhary [22] extended these ratios to a wider range $M_2O/SiO_2 = 0.20\text{--}0.48$, $SiO_2/Al_2O_3 = 3.3\text{--}4.5$, $H_2O/M_2O = 10\text{--}25$ and $M_2O/Al_2O_3 = 0.8\text{--}1.6$.

In this research, potassium alkali was thus used as the alkali activator instead of sodium alkali in order to study the effect of K/Al (0.13 to 0.7) [23] and curing temperature (40 °C and 60 °C) on the compressive strength and microstructure. The samples were heat-treated at high temperature 550 °C with 6 h soaking time. The properties of heat-treated and non heat-treated geopolymers such as chemical properties, microstructures and compressive strength were investigated and discussed.

2. Experimental

2.1. Materials preparation

Metakaolin (MK) (53.71 wt% SiO_2 , 43.52 wt% Al_2O_3 and 1.36 wt% K_2O) was received by calcining kaolin (from MRD-ECC Co., Ltd., in Ranong province, Thailand) at 600 °C for 2 h [12]. The MK was then sieved through 325 mesh (45 μm). The main alkali activators used were KOH and K_2SiO_3 (10.83 wt% K_2O , 23.31 wt% SiO_2 and 65.86 wt% H_2O). K_2SiO_3 and KOH solutions with concentrations of 6, 8, 10, 20, 30 and 40 M. The ratios of K_2SiO_3/KOH were 1 and 1.5.

2.2. Sample preparation and characterization

Two series of mixtures with varied KOH concentrations, K_2SiO_3/KOH ratio and curing temperatures (40 °C and 60 °C), as given in Table 1, were prepared. The alkali/metakaolin ratio of the mixture formulations was kept at 0.67. The Si/Al, K/Al and K/Si molar ratios are also shown in Table 1. The K/Al and K/Si were changed by varying the KOH concentrations and K_2SiO_3/KOH ratio. For charge balance in the tetrahedral aluminosilicate structure, the ideal K/Al is 1 [1,21]. For this research, the Al_2O_3 content was very high at

Table 1
Mixture formulations.

Sample	K_2SiO_3/KOH	KOH concentration	Si/Al	K/Al	K/Si
6ML1	1	6	2.40	0.15	0.36
8ML1		8	2.40	0.18	0.44
10ML1		10	2.40	0.21	0.51
20ML1		20	2.40	0.38	0.91
30ML1		30	2.40	0.54	1.30
40ML1		40	2.40	0.70	1.69
6ML1.5	1.5	6	2.46	0.13	0.33
8ML1.5		8	2.46	0.16	0.39
10ML1.5		10	2.46	0.18	0.45
20ML1.5		20	2.46	0.31	0.77
30ML1.5		30	2.46	0.44	1.08
40ML1.5		40	2.46	0.57	1.39

Remark: the curing temperatures were varied between 40 °C and 60 °C for 24 h, and each formulation was heat-treated and non heat-treated.

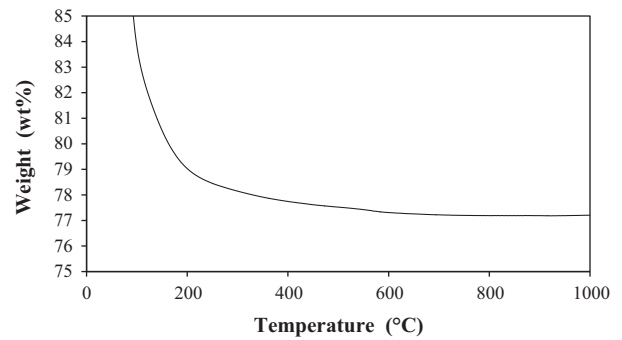


Fig. 1. Weight loss of non heat-treated specimen by TGA.

43.52 wt% whereas MK from other sources are generally lower at around 30.00 wt% [20,24] and thus the K/Al ratio is thus low at between 0.13 and 0.70.

Metakaolin-based geopolymers were prepared by manually mixing metakaolin and potassium alkali activators in a bowl. Then, the geopolymer paste was rapidly poured into cubic acrylic molds of $25 \times 25 \times 25 \text{ mm}^3$ and put on a vibrator for 1 min to remove entrapped air. In order to prevent the evaporation of water and reactivity with air, the specimens were covered with plastic films during the setting and hardening process. The molds were removed after 24 h. Then, the specimens were cured at temperature of 40 °C and 60 °C for 24 h. Specimens were stored at an ambient temperature (25 °C), called “non heat-treated specimens” or “NH”. The other series were fired at 550 °C for 6 h and after that stored at an ambient temperature, called “heat-treated specimens” or “H”, until testing. The temperature of heat treatment at 550 °C was determined by Thermogravimetric Analysis (TGA) (Mettler Toledo, TGA/DSC1, England) as the temperature at which reactivity, hence no weight loss, is no longer recorded over a measuring range from 50 °C to 1000 °C, as shown in Fig. 1.

The compressive strength was tested at 28 days (UTM) (Universal Testing Machine, Hounsfield, H50KS, England). The fragments after compressive strength tests were collected and used for the microstructural observation. A Scanning Electron Microscope (SEM) (Philip, XL30, the Netherlands) with an accelerating voltage of 15 kV, a probe current of 15 mA and a working distance of 10 mm was used. The samples were immersed in acetone for 1 day to stop any further geopolymerization reaction and then ground to very fine particles ($\leq 45 \mu m$) with an agate mortar for phase characterization by using X-ray diffraction (XRD) (X'pert, Philips, the Netherlands) with 40 kV, 35 mA, CuK_{α} radiation, scanning rate of 1° per min from 5° to 80° (2θ), scans of 45 min long and steps of 0.02° (2θ). The functional groups of geopolymer were determined by Fourier Transform Infrared Spectroscopy (FTIR) (Bruker, Alpha-E, US). The water absorption, density and porosity were measured at 28 days. Finally, the best mixture formulation was chosen to test the compressive strength at 7, 14, 21 and 28 days.

3. Results and discussion

3.1. Compressive strength

The compressive strength values of all samples at 28 days are shown in Fig. 2. The results show that the cured L1.5 specimens at 40 °C gave the higher strength value than those of L1 approximately 3–7 MPa. The cured specimens at 40 °C showed the same trend both L1 and L1.5. The compressive strength increased as a function of KOH concentration increasing from 6 to 10 M but when the KOH concentration increased more than 10 M, the compressive

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