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Effect of Sodium Hydroxide Molarity on Physical, Mechanical and Thermal Conductivity of Metakaolin Geopolymers

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Abstract. In the present work, the effect of different sodium hydroxide (NaOH) molarity (6M, 8M, 10M, 12M and 14M) on the physical, mechanical and thermal conductivity of metakaolin geopolymers (MkGPs) was investigated. Geopolymers were prepared by activating the metakaolin with a mixture of NaOH with sodium silicate (Na_2SiO_3). The products obtained were characterized after 28 days of curing. The density, porosity, compressive strength and thermal conductivity (TC) were determined. In general, the NaOH molarity has a significant effect on the compressive strength of the MkGPs. The highest compressive strength was 14.6 MPa achieved with 10M of NaOH solution. The thermal conductivity of MkGPs measured in this work was low in the range between 0.71-0.97 W/mK. NaOH molarity had a significant effect on compressive strength but a marginal effect on thermal conductivity of MkGPs. The thermal conductivity was mainly affected by the bulk density and thus the total porosity. The results showed that the geopolymer can be considered to be used as the thermal insulating material.

1. Introduction

In 1978, Professor Joseph Davidovits has developed a new class of material called geopolymer [1]. Geopolymers are ceramic-like materials produced by geopolymerisation reaction that uses aluminosilicate materials and an alkaline activating solution as the primary raw materials [1-4]. Depending on the raw materials selection, geopolymers exhibit excellent compressive strength [5], fire resistance [4, 6], toxic waste immobilisation [7, 8] and ability to form insulating foams [9]. The alkaline activator for geopolymer comprises of soluble alkali metal and they influence the kinetics of geopolymerisation [10, 11]. The degree of concentration of the alkaline activator greatly affects the geopolymerisation process as excessive alkalinity can adversely affect the strength of the geopolymer [12]. The concentration of NaOH is very important for the dissolution ability of the aluminosilicate source as low molarity causes insufficient of Na^+ ions to be reacted with Si^{3+} and Al^{4+} to complete the geopolymerisation reaction. However, too high concentration results in a faster setting due to reaction products rapidly forming around the unreacted particles preventing further reaction [13].

The composition of geopolymer greatly affects the mechanical and thermal properties. Thermal conductivity is important when considering thermal performance as geopolymer has the ability to be

thermally insulating materials due to its amorphous structure [14, 15]. In other words, it is a physical property that determines how much heat will flow in a material. Lower TC means better thermal insulation [16-18]. Density and the degree of porosity are expected to control the thermal conductivity and compression strength of the geopolymer [19, 20]. For geopolymer to be categorized as insulating material, certain requirement set by functional classification of RILEM such as thermal conductivity must be met [21].

The current work focus on the effect of NaOH molarity on the physical (bulk density, true density and total porosity), mechanical (compressive strength) and thermal (thermal conductivity) properties of sodium-based geopolymers synthesized from MK. The alkali activator was prepared using different molarity of NaOH and Na_2SiO_3 solution.

2. Experimental method

2.1. Materials
Metakaolin was used as the Si-Al source material. The metakaolin was obtained by calcining the kaolin, purchased from Associated Kaolin Sdn. Bhd. Malaysia, at 850 °C for 6 h in the furnace. The chemical composition of metakaolin determined by X-ray fluorescence (XRF) spectrometer is shown in Table 1. The particle size distribution of metakaolin was obtained using a Malvern particle size analyser. The mean particle size d $_{(50)}$ was 9.465 μm. The sodium hydroxide (NaOH) powder was of caustic soda micropearls and 99% purity with the brand name of Formosoda-P, made in Taiwan. The sodium silicate (Na_2SiO_3) solution was supplied by South Pacific Chemicals Industries Sdn. Bhd., Malaysia, with a chemical composition of 30.1% SiO_2, 9.4% Na_2O and 60.5% H_2O.

Table 1. Chemical composition of metakaolin as determined by XRF analysis

Chemical	Wt (%)
	Metakaolin
SiO_2	55.7
Al_2O_3	38.6
Fe_2O_3	2.03
TiO_2	0.78
CuO	0.03
ZrO_2	0.04
K_2O	2.43
MnO_2	0.04
LOI	1.74

2.2. Preparation of metakaolin geopolymers (MkGPs)
NaOH solution was prepared in a volumetric flask using distilled water. The alkaline activator solution was formed by mixing Na_2SiO_3 and NaOH solutions at a ratio of 0.24 until a clear solution was obtained. The sodium hydroxide molarity used in this study was 6M, 8M, 10M, 12M and 14M. In order to produce geopolymer paste, metakaolin was mixed with the prepared activator solution. Then the paste was poured into steel moulds with a dimension of 50 ×50 × 50 mm. The moulded samples were sealed with a thin film to prevent moisture loss. The samples were cured at the room temperature for 24 hours and in the oven at 60 °C for another 24 hours.

2.3 Test and analysis method

The bulk density of the MkGPs was obtained as the ratio between the mass of the sample and its geometrical volume. The true density of the metakaolin geopolymer was determined by helium pycnometer technique (Multipycnometer, Quantachrome). The total porosity of the MkGPs was calculated according to the following equation:

$$Total\ porosity\ (\%) = \left(1 - \frac{bulk\ density}{true\ density}\right) \times 100$$

The compressive strength of metakaolin geopolymers was measured in accordance to ASTMC109 by using Instron machine series 5569 Mechanical Tester.

Room temperature thermal conductivity was measured on cylindrical specimens (diameter of 4 mm and height of 4 mm) by using a Hot-Disk thermal constant analyzer (Hot-Disk AB Uppsala, Sweden). At least 3 measurements were performed to ensure reproducibility.

The microstructural analysis was carried out using JSM-6460 LA model Scanning Electron Microscope (JEOL).

3. Result and discussion

3.1. Density and porosity

The bulk density and the total porosity of MkGPs are presented in Figure 1. The measured densities were in the range between 1.49 to 1.64 g/cm^3 for MkGPs with NaOH molarity ranged from 6M to 14M. In general, increasing NaOH concentration increases the bulk density of geopolymers. The lowest density is observed at 8M NaOH molarity with 1.49 g/cm^3. After 8M, the bulk density is increased with increasing NaOH molarity.

The porosities of the MkGPs were ranged between 18% and 24%. The highest porosity of 24% was recorded at 8M NaOH solution. This was probably contributed to the lowest bulk density recorded for geopolymers with 8M of NaOH solution. Further increase in the NaOH molarity from 10M to 14M decreased the porosity as the MkGPs become denser as shown by the density values. In general, density is inversely proportional to the porosity [22].
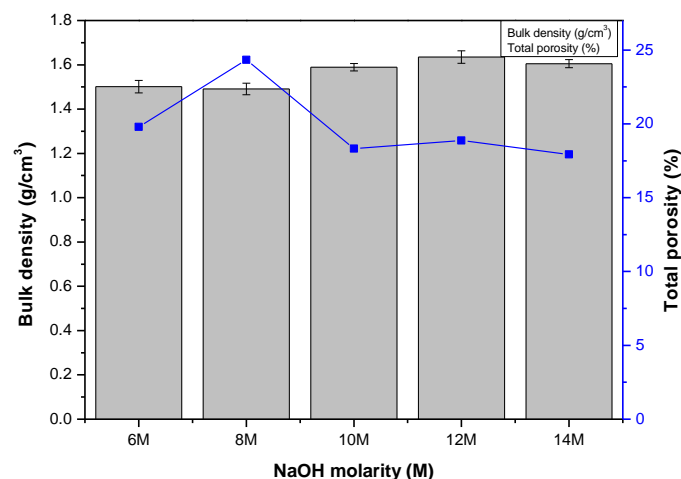

Figure 1: Bulk density and total porosity of MkGPs at varying NaOH molarity.

3.2. Compressive strength

The concentration of NaOH is of great importance affecting the geopolymer properties particularly the compressive strength development. At low molarity (6M), the compressive strength was very low with

only 1.3 MPa. This was because of the lower dissolution ability of metakaolin at low NaOH molarity which causes insufficient Al^{3+} and Si^{4+} ions to allow for complete geopolymerisation process. The compressive strength increased towards an optimum of 14.6 MPa when the NaOH molarity increased from 8M to 10M (Figure 2). The optimum strength at this molarity is likely due to the sufficient Al^{3+} and Si^{4+} ions released from the metakaolin to participate in geopolymerisation with the optimum alkaline medium. The concentration of NaOH solution affects the dissolution ability of the metakaolinite particulates as the higher concentration provides the better dissolving ability of raw materials and accelerate the condensation of the monomer. Hence, the bonding strength of the geopolymer increases [23]. On due course, a dense MkGPs were obtained as shown by the increasing density (Figure 1) with the increasing NaOH molarity. However, at a certain NaOH threshold concentration, the compressive strength of geopolymer decreased [24]. Proper NaOH concentration is needed to facilitate and activate the system in order to develop a proper mechanical property as the compressive strength. As refer to Figure 2, the strength of MkGPs reduced when activated with 12M and 14M of NaOH solution.
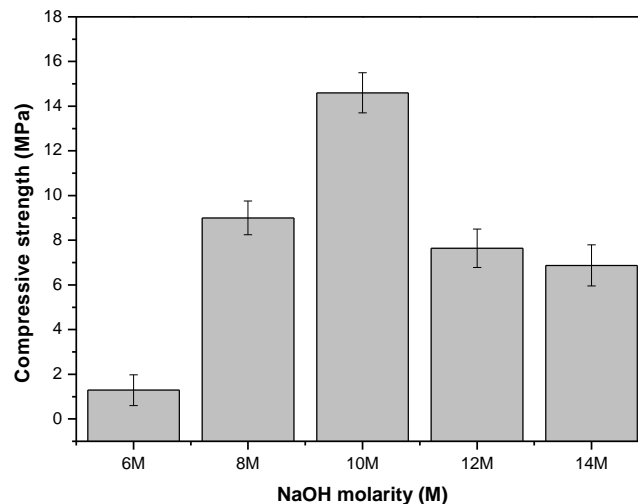

Figure 2: Compressive strength of MkGPs at varying NaOH molarity.

3.3. Thermal conductivity (TC)

The thermal conductivity value was ranged from 0.71 to 0.97 W/mK for MkGPs activated with 6M to 14M of NaOH solution. The lowest value of 0.71 W/mK was recorded at 8M and the highest value of 0.97 W/mK at 14M. The thermal conductivity of MkGPs did not vary significantly with NaOH molarity except for geopolymers activated with 8M NaOH solution. This might most probably due to the highest porosity of geopolymer with 8M NaOH solution (Figure 3). Porosity has the main influence on lowering the TC. The correlation between the TC and compressive strength was not significant because the highest strength was not necessarily attributed to the lowest TC.

Duxson *et al.* [18] obtained a lower value (0.8 W/mK) of TC when activated the geopolymer with potassium-based alkaline activator. The lower value of TC obtained in this work was assumed to be lower because the TC of sodium-based geopolymer is doubled of the potassium-based geopolymer. According to Gouloure *et al.* [25], geopolymers are promising to be used as thermal insulation materials because of the low TC (< 1 W/mK). Further work such as the addition of foaming agent to the MkGPs might help to improve the thermal conductivity to satisfy the criteria to be insulating materials as required by RILEM. Besides, the existence of the crystalline phases in geopolymer affects

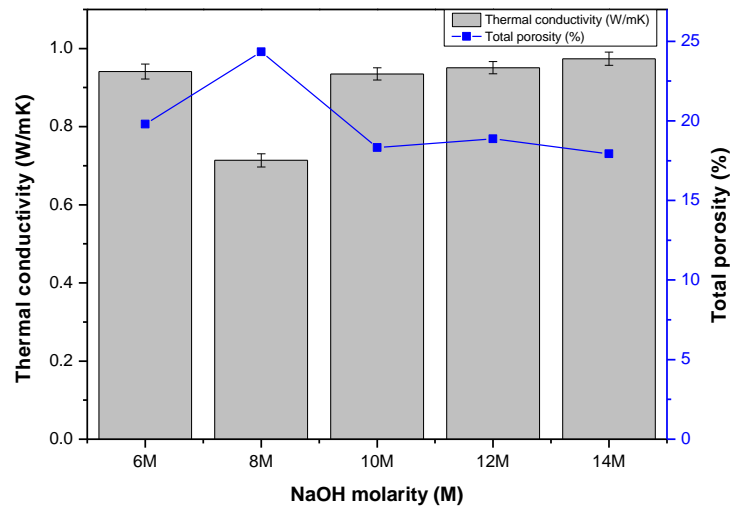the thermal conductivity as the insulating ability is based highly on the amorphous nature of the material [17].

Figure 3: Thermal conductivity and total porosity of MkGPs at varying NaOH molarity.

3.4. Microstructural analysis

From the microstructural image in Figure 4, all samples showed the existence of pores that contributed to the porous structure. The MkGPs at low NaOH concentration (6M and 8M) has more pores compared to the MkGPs at high NaOH molarity (10M, 12M and 14M). Unreacted particles were observed in the samples at 6M and 8M due to the low molarity of NaOH solution to be reacted with metakaolin particles. At 10M, the unreacted particles still exist but become less due to the progress of geopolymerisation reaction. The MkGPs with high molarity (12M and 14M) has a large portion of unreacted particles.

The existence of more pores at 6M and 8M has shown to have high porosity values which correlate well with the lower densities (Figure 1) compared to MkGPs at high NaOH molarity (10M, 12M and 14M). The size of the pores also became smaller at high molarity which is showed by the dense matrix. The existence of pores reduced the density of the geopolymers and subsequently decreased the compressive strength and improved the insulating properties.
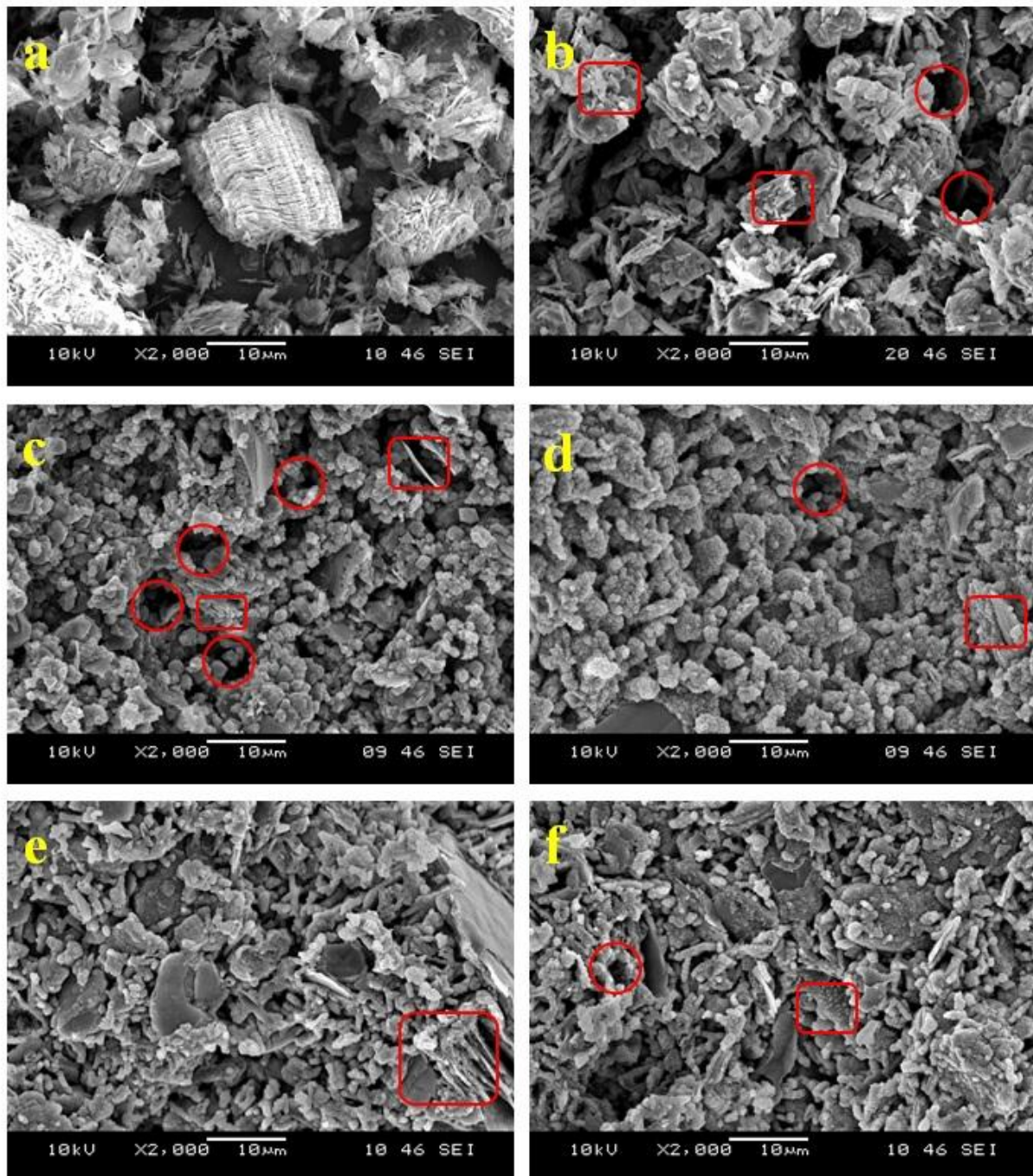
Figure 4: Microstructural images of (a) metakaolin and MkGPs with different NaOH molarity (b) 6M, (c) 8M, (d) 10M, (e) 12M and (f) 14M. (　○pores and 　□unreacted metakaolin particles).

4. Conclusions

The effect of NaOH molarity on physical, mechanical and thermal properties of MkGPs has been carried out. The NaOH molarity has a significant effect on the compressive strength but a marginal effect on thermal conductivity of MkGPs. The highest compressive strength of 14.6 MPa was achieved at 10M NaOH solution. The lowest thermal conductivity of 0.71 W/mK was obtained when activated with 8M NaOH solution. The thermal conductivity was mainly affected by the bulk density and total porosity. The results obtained show that geopolymer here developed can be considered potentially useful to be used as the thermal insulating material.

Acknowledgments

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