Correlation of Thermal Conductivity Versus Bulk Density, Porosity and Compressive Strength of Metakaolin Geopolymer

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Abstract. This paper investigates the correlation of thermal conductivity versus bulk density, porosity and compressive strength of metakaolin geopolymer for different mixing parameters (that are, alkali concentration, activator (AA) ratio and metakaolin/activator (MK/AA) ratio). Metakaolin was alkali-activated with NaOH and Na2SiO3 solution to produce geopolymer. Varying NaOH concentration (6M, 8M, 10M, 12M and 14M), AA ratio (0.4, 0.6, 0.8, 1.0 and 1.2) and MK/AA ratio (0.6, 0.7, 0.8, 0.9 and 1.0) were used to study the effect on bulk density, porosity, compressive strength and thermal conductivity. Result showed that metakaolin geopolymer with maximum compressive strength (33 MPa), bulk density of 1680 kg/m3, porosity of 18% and thermal conductivity of 0.40 W/mK is achieved with alkali concentration of 10M, AA ratio of 1.0 and MK/AA ratio of 0.8. From the gradation analysis of the strength result, AA ratio is the most influential mixing parameter in determining the compressive strength. In contrast, MK/AA ratio significantly affected the thermal conductivity. From the Pearson correlation coefficient, TC had strong relationship with bulk density and porosity and poor relationship with compressive strength.

1. Introduction

Geopolymers are made from different aluminosilicates that have received significant attention in recent years [1-4]. Geopolymers are produced by geopolymerisation reaction that uses aluminosilicate materials and an alkaline activating solution as the primary materials. Geopolymerization reaction involves the dissolution and condensation process that occurs at room temperature or slightly higher temperature [5-8].

The properties of geopolymer strongly dependent on synthesis parameters, such as raw materials, mixing proportions and curing conditions [9, 10]. This work focuses on the mixing proportion which is the NaOH concentration, alkali activator (AA) ratio and metakaolin/activator (MK/AA) ratio. The concentration of NaOH is very important for the dissolution ability of the aluminosilicate source as Na+ ions reacted with Si3+ and Al4+ to complete the geopolymerisation reaction [11]. Geopolymer with high S/L ratios had a low viscosity and vice versa, which accelerates the dissolution of source materials [9].

Thermal conductivity is important when considering thermal performance as geopolymer has the ability to be thermally insulating materials due to its amorphous structure [11, 12]. In other words, it is a physical property that determines how much heat will flow in a material. Lower TC means better
thermal insulation [13-16]. Density and the degree of porosity are expected to control the thermal conductivity and compression strength of the geopolymer [16-19].

In this work, mixing parameters such as NaOH concentration, AA ratio and MK/AA ratio was investigated for the optimization of metakaolin geopolymer. Their effect on compressive strength, thermal conductivity, bulk density and porosity were studied to satisfy the correlation study.

2. Experimental work

2.1. Materials

Metakaolin was used as the Si-Al source material in this work. The metakaolin was obtained by calcining the kaolin at 900 ºC for 6 h in the furnace. The chemical composition of metakaolin determined by X-ray fluoroscene (XRF) spectrometer is shown in table 1. The activator solution was a mixture of sodium hydroxide (NaOH) and liquid sodium silicate (Na₂SiO₃). The NaOH powder has 99% purity with a density of 2.13 g/cm³ while the liquid Na₂SiO₃ contains 30.1% SiO₂, 9.4 Na₂O and 60.5% H₂O with a density of 2.4 g/cm³.

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

| Factors  | Weight percent (wt.%) |
|----------|-----------------------|
| SiO₂     | 55.7                  |
| Al₂O₃    | 38.6                  |
| Fe₂O₃    | 2.03                  |
| TiO₂     | 0.78                  |
| CuO      | 0.03                  |
| ZrO₂     | 0.04                  |
| K₂O      | 2.43                  |
| MnO₂     | 0.04                  |
| Others   | 0.38                  |

2.2. Preparation and optimization of metakaolin geopolymer

Metakaolin geopolymer was prepared by mixing metakaolin with alkali activator for 5 minutes by using a mechanical mixer to from a homogenous slurry. The fresh geopolymer paste was poured into high-density polyethylene (HDPE) mould with dimensions of 50 × mm × 50 mm. The moulded samples were vibrated for 2 minutes on the vibration table to remove entrained air and sealed with a thin film to prevent moisture loss. The geopolymer paste were left to cure at room temperature (29 °C) for 24 hours and subsequently place in an oven at 60 °C for another 24 hours. After curing, the samples were demoulded and kept under room temperature until the day of testing. The NaOH concentration, alkali activator (AA) ratio and metakaolin/activator (MK/AA) ratio were varied as showed in table 2. The optimum mixing parameter was selected based on the highest compressive strength.

Table 2. Mixing parameters of metakaolin geopolymers in order to investigate the optimum formulation.

| Investigated Parameter | NaOH concentration | AA ratio          | MK/AA ratio          |
|------------------------|--------------------|-------------------|---------------------|
| NaOH Concentration     | 6M, 8M, 10M, 12M and 14M | 0.24              | 0.80                |
| AA Ratio               | 10M                | 0.40, 0.6, 0.8, 1.0 and 1.2 | 0.80 |
| MK/AA Ratio            | 10M                | 1.00              | 0.60, 0.7, 0.80, 0.90 and 1.00 |
2.3. Testing and analysis method

The true density of the metakaolin geopolymers were determined by the pycnometer (AccuPyc II 1340 Helium Pycnometer, Micromeritics). The bulk density was measured by the geometric method. The total porosity of the geopolymers were obtained from the bulk density to the true density with equation 1.

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

Compressive strength after 28 days was tested based on the ASTM C109 using Instron machines series 5569 Mechanical Tester. The samples were tested for each parameter to obtain the average compressive strength value. Room-temperature thermal conductivity ($\lambda$) was measured using a KD2 Pro Thermal Properties Analyzer (Decagon Devices Inc) which utilized the transient line heat source method according to IEEE 441981 and ASTM D5334. As least 3 measurements were performed to ensure accuracy.

3. Results and Discussions

Figure 1 presents the compressive strength of metakaolin geopolymers with different mixing parameters. It was in evidence that the NaOH concentration, AA ratio and MK/AA ratio are influential to the compressive strength measured. Besides that, increasing each mixing parameter increased the compressive strength. The optimum compressive strength of 33 MPa was achieved at 10M NaOH solution, AA ratio of 1.0 and MK/AA ratio of 0.8. To identify the significant parameters affecting the formation of geopolymers, a gradation analysis was carried out (table 3). From this analysis, AA ratio has the most critical influence to the strength of geopolymers indicating by the highest range, followed by MK/AA ratio and NaOH concentration. This conclusion complied with the bulk density and porosity values recorded in table 4. The proportion of Na$_2$SiO$_3$ and NaOH in the activator solution is crucial as high AA ratio contributes more soluble Si while low activator ratio contributes more OH- for dissolution purposes. The presence of soluble Si by Na$_2$SiO$_3$ could modify the reaction kinetics by enhancing the condensation process and thus improving the compressive strength [20, 21].

Figure 1. Compressive strength of metakaolin geopolymers.
Table 3. Gradation analysis of various mixing parameters.

| Investigated Parameter | Compressive strength (MPa) | Range* |
|------------------------|----------------------------|--------|
| NaOH concentration     |                            |        |
| 6M                     | 13.31                      | 13.31  |
| 8M                     |                            |        |
| 10M                    |                            |        |
| 12M                    |                            |        |
| 14M                    |                            |        |
| AA ratio               |                            |        |
| 0.4                    | 20.70                      | 20.70  |
| 0.6                    |                            |        |
| 0.8                    |                            |        |
| 1.0                    |                            |        |
| MK/AA ratio            |                            |        |
| 0.6                    | 14.68                      | 14.68  |
| 0.7                    |                            |        |
| 0.8                    |                            |        |
| 0.9                    |                            |        |
| 1.0                    |                            |        |

Table 4. Values of the thermal conductivity, bulk density and porosity of metakaolin geopolymers.

| Investigated Parameter | Thermal conductivity (W/mK) | Bulk density (kg/m³) | Porosity (%) |
|------------------------|----------------------------|----------------------|--------------|
| NaOH concentration     |                            |                      |              |
| 6M                     | 0.364                      | 1491                 | 24.33        |
| 8M                     | 0.383                      | 1501                 | 19.79        |
| 10M                    | 0.412                      | 1635                 | 17.93        |
| 12M                    | 0.402                      | 1605                 | 18.33        |
| 14M                    | 0.395                      | 1589                 | 18.87        |
| AA ratio               |                            |                      |              |
| 0.4                    | 0.327                      | 1603                 | 26.96        |
| 0.6                    | 0.332                      | 1629                 | 26.17        |
| 0.8                    | 0.345                      | 1651                 | 24.75        |
| 1.0                    | 0.400                      | 1675                 | 19.76        |
| 1.2                    | 0.350                      | 1493                 | 30.07        |
| MK/AA ratio            |                            |                      |              |
| 0.6                    | 0.348                      | 1528                 | 1528         |
| 0.7                    | 0.361                      | 1547                 | 1547         |
| 0.8                    | 0.400                      | 1675                 | 1675         |
| 0.9                    | 0.384                      | 1648                 | 1648         |
| 1.0                    | 0.372                      | 1637                 | 1637         |

Figure 2 shows the Pearson correlation coefficient study which was performed to determine the correlation between TC, bulk density, porosity and compressive strength of metakaolin geopolymers with varying mixing parameters. Pearson correlation coefficient is used to measure the strength of two linear dependable variables. If the correlation coefficient is nearer to value of 1.0, it indicates a strong functional relationship.

Referring to figure 2, TC had strong relationship with bulk density and porosity. It is most significantly affected by the MK/AA ratio. As aforementioned, the solid and liquid contents in geopolymer mixture determined the efficiency of dissolution and formation of dense matrix. According to Rickard [21], liquid content is substantial part of the mixture and influence the properties such as density and porosity. It was observed that regardless of the mixing parameter, the TC value would not differ much if the porosity was similar.

Besides that, it is worth to note that TC has poor correlation with compressive strength regardless of the different mixing parameters. The high compressive strength did not contribute to the better insulating properties [22, 23]. This is because the denser the geopolymer, the higher the compressive strength and thus higher the TC value [24]. The structure of the geopolymer became compact and less porous due to the geopolymerisation reaction that produces amorphous and porous interconnected polysialates that restricted heat transfer [25-27]. This was well-supported by Fongang et al. [28].
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Figure 2. Correlation coefficient (R2) of TC versus bulk density, porosity and compressive strength with varying mixing parameters: (a-b) NaOH concentration, (c-d) AA ratio and (e-f) MK/AA ratio.

4. Conclusions
From the research, we can draw several conclusions, which are;
- Optimized metakaolin geopolymers had a compressive strength of 33 MPa after 28 days prepared using NaOH concentration of 10M, AA ratio of 1.0 and MK/AA ratio of 0.80. The strength determining factor was AA ratio > MK/AA ratio > NaOH concentration.
- From the Pearson correlation coefficient, TC had strong relationship with bulk density and porosity and poor relationship with compressive strength. The solid and liquid contents in geopolymer mixture plays a substantial part in determining the properties of geopolymers.

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