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PHASE ANALYSIS OF DIFFERENT LIQUID RATIO ON METAKAOLIN/DOLOMITE GEOPOLYMER

Geopolymer is widely studied nowadays in various scope of studies. Some of the ongoing studies are the study of the various materials towards the geopolymer strength produced. Meanwhile, some of the studies focus on the mixing of the geopolymer itself. This paper discussed the phase analysis of metakaolin/dolomite geopolymer for different solid to the liquid ratio which was, 0.4, 0.6, 0.8, and 1.0, and the properties that affected the geopolymer based on the phases. The constant parameters in this study were the percentage of metakaolin and dolomite used. The metakaolin used was 80% meanwhile dolomite usage was 20%. Besides that, the molarity of NaOH used is 10M and the alkaline activator ratio used is 2.0. All the samples were tested at 28 days of curing. The results show that the 0.8 solid to the liquid ratio used gave better properties compare to other solid to liquid ratio. The phases analyzed were quartz, sillimanite, mullite, and faujasite. The 0.8 S/L ratio shows the better properties compared to others by the test of phase analysis, compressive strength morphology analysis, and functional group analysis.

Keywords: Geopolymer; Metakaolin; Dolomite; Phase Analysis

1. Introduction

Many studies show that the methods of XRD help to understand the behavior of geopolymers and for this reason, they were of great significance to scientists in the latest studies [1,2]. XRD is also used, considering the considerable amorphous nature of geopolymers, to classify newly developed phases, to describe the degree to which the starting materials have reacted, and to determine the amorphous level of the final products.

Geopolymers are synthetic alkali aluminosilicate materials produced under extremely alkaline conditions by the reaction between aluminosilicate materials with alkali or alkali silicates [3-5]. Synthesis of geopolymers implies polymerization of dissolution and condensation. Cementitious materials are also known as condensation materials at room temperature, the reaction takes place. Highly alkaline solutes, such as potassium and sodium hydroxide (NaOH) potassium hydroxide (KOH) are integrated into SiO₂ and Al₂O₃ rich source materials [6].

One of the factors that affect the rate of geopolymer formation is influenced by the initial solid content. The activator concentration was increased, a delay in geopolymer formation occurred in geopolymer bricks, whereas temperature acceler-

ated its formation. The solids ratio did not influence the rate of geopolymer formation, but it was proved that larger product precipitation was observed when the ratio of the solid was increased because of the increase in dissolved species [7].

Metakaolin was chosen to be used in this study due to increased reactivity over raw kaolin. Metakaolin can be obtained from calcination or de-hydroxylation of kaolin clay at 500-900°C [8]. It extracts chemically bonded water and changes a significant portion of the kaolin-coordinated octahedral aluminum to a four and five-fold configuration [9]. This makes metakaolin useable as geopolymer source materials. Also, metakaolin geopolymer has demonstrated an increase in intensity and tolerance to fire behaviors [10,11].

Aside from that, dolomite is one of the materials that can be used in geopolymer production. However, limited studies were done towards the dolomite in terms of stand-alone geopolymer material or as a blended geopolymer material [12]. Dolomite was used as a source for the manufacture of magnesium oxide in concrete materials, which is a well-recognized and already commercialized shrinkage-compensating additive [13]. Thus, the dolomite was used as an addition to the metakaolin to produce metakaolin/dolomite blended geopolymer.

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2. Materials

There are four main materials used in this study. Two of them act as solid precursors and meanwhile, the other two act as the alkaline activator. There were metakaolin, dolomite, sodium silicate (Na_2SiO_3), and sodium hydroxide (NaOH).

The metakaolin was obtained from the calcination of kaolin at 900°C for 8 hours. The kaolin used was purchased from Associated Kaolin Industries Sdn. Bhd. Malaysia. The physical form of kaolin used was powder type and has a minimum of 40% of particles sized less than $2\ \mu\text{m}$ and a maximum of 2% of moisture content. Dolomite was supplied by the Perlis Dolomite Industries Sdn. Bhd. Perlis, Malaysia. The dolomites undergo a crushing process for 30 minutes and were sieved to obtained $150\ \mu\text{m}$ size.

The technical grade liquid Na_2SiO_3 was supplied by South Pacific Chemical Industries Sdn. Bhd. (SPCI), Malaysia with a chemical composition of 30.1% SiO_2 , 9.4% Na_2O , and 60.5% H_2O ($\text{SiO}_2/\text{Na}_2\text{O}$ ratio of 3.20). The liquid Na_2SiO_3 is colorless and dissolves readily in water. NaOH was obtained from the brand name of Formosoda-P supplied by Formosa Plastic Corporation, Taiwan with a purity of 99%. The 10M NaOH was prepared by dissolving pallet NaOH with distilled water. After dissolved it with the distilled water, the NaOH was left at room temperature for 24 hours.

3. Methodology

The metakaolin/dolomite geopolymers were done by mixing different solid-liquid ratio of 0.4, 0.6, 0.8, and 1.0. The metakaolin/dolomite mixture was fixed to 80/20 by mass and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio used was 2.0 by mass. Samples preparation was done by mixing the solid precursor followed by the addition of alkaline activator by using the mechanical stirrer. After mixing for 5 minutes, it was poured into $50 \times 50 \times 50\ \text{mm}$ mold and was removed 24 hours later. The samples were tested after 28 days of curing.

4. Results

Phase Analysis. Figure 1 shows the phase analysis of metakaolin/dolomite geopolymer for different solid to liquid ratio. In the XRD diffractogram, the major crystalline components were quartz (Q), sillimanite (S), and mullite (T). Similar observations have been reported by [14]. The XRD patterns of the geopolymers for metakaolin manifested the formation of sillimanite. The patterns also represented the formation of silicate-based compounds, particularly when increased in solid content.

Quartz had the highest peak compared to sillimanite and mullite. This can be seen that quartz is usually detected as an impurity of kaolin clays. The role and behavior of quartz particles in geopolymerization and its probable effects on the mechanical behavior of geopolymer products. Some study concludes that using high content quartz kaolin causes higher

compressive strength [15]. A study concludes that the scanning electron microscopy confirmed that quartz does not participate in geopolymerization and completely remains in the structure as fine particles. High-hardness quartz particles by retarding crack growth in the geopolymer matrix enhance the mechanical behavior of the samples [16,17]. Although quartz particles could potentially increase the compressive strength of geopolymers, the main factor determining the mechanical response of geopolymers is geopolymer gel, and to obtain the maximum compressive strength, appropriate thermal and alkaline activation of the source material is necessary [18].

Quartz was the phase most widely found at (2θ) of 26.85° and 31.47° . The other phases found in geopolymer was mullite shown from the peak at (2θ) of 48.20° and 44.47° . Another phase was sillimanite which found at (2θ) 19.15° , 42.2° , and 53.66° . Quartz and mullite were originally present in the starting material. This indicates that the phases were not attacked during the geopolymerization process [19].

With the change of solid to liquid ratio. The peak of metakaolin dolomite geopolymer would not show significant change. To observe the phase changing in this geopolymer, a heat exposure needs to be done. Exposing the geopolymer to a high temperature would make a proper way to study the phase transition in the geopolymer.

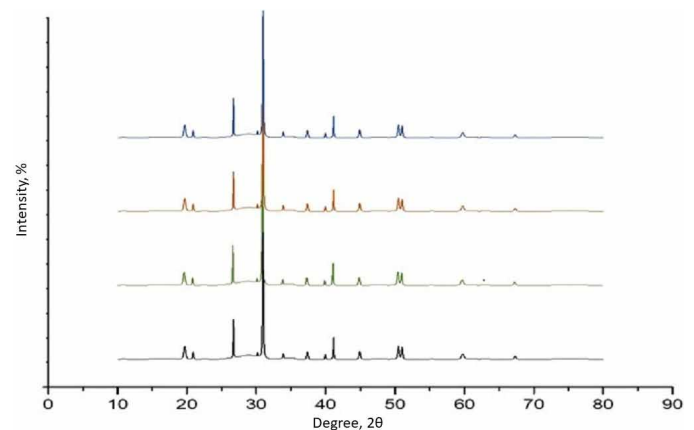


Fig. 1. Phase analysis of the metakaolin/dolomite geopolymer

Compressive Strength. Fig. 1 shows the compressive strength for metakaolin/dolomite geopolymer with 0.4, 0.6, 0.8 and 1.0 solid to liquid ratio. Most of the ratio produced lower than 20 MPa of compressive strength except for solid to liquid of 0.8. The highest compressive strengths obtained from S/L of 0.8 was 25.36 MPa at. The lowest compressive strength recorded was the usage of 0.4 with 2.03 MPa. The compressive for 0.6 S/L ratio was 12.54 MPa and 1.0 S/L was 4.47 MPa respectively.

The different of Si and Al content in a geopolymer material will usually create different strength. The higher Si and Al can create bonds will improve the strength of the geopolymer [18]. On the other way, higher volume of solid content compared to fluid which make it hard to be mixed and reduce the workability. Due to the reasons above, the geopolymer will not mix well and the consequently lowered the compressive strength [20].

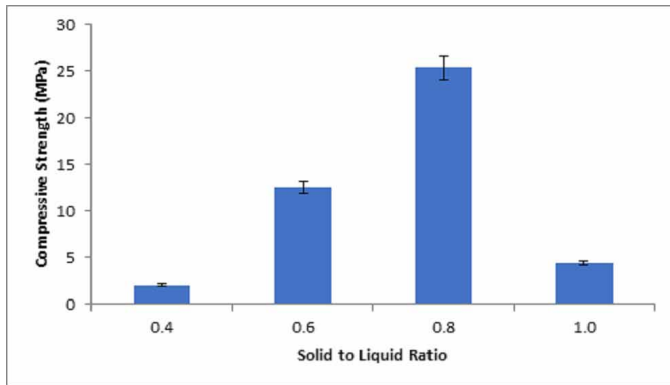


Fig. 2. The compressive strength for metakaolin/dolomite geopolymer with various solid to liquid ratio

Morphology Analysis. Fig. 2 shows the morphology of metakaolin with different S/L ratio. The $\times 500$ magnification was used to observe all the geopolymer samples. Fig. 2(a) shows a few crack and unreacted material observed in the geopolymer sample. This was the reason which gave low compressive strength which was lower than 20 MPa. The unreacted materials in the geopolymer will disturb the geopolymer chains. The disturbance of the chains will reduce the properties of the metakaolin geopolymer itself [21].

Fig. 2(c) shows the morphology of metakaolin/dolomite geopolymer for ratio 0.8 S/L. The morphology of the geopolymer is the almost the same as Fig. 2(b). It can be observed that better surface can be observed due to better reaction between the solid

precursor and the alkaline activator. This resulted better compressive strength compared to others S/L ratio that showed before.

Fig. 2(d) shows the geopolymer for 1.0 S/L ratio. The surface of the geopolymer become more uneven which was almost the same as Fig. 2(c) but with more geopolymer gel can be observed. However, the compressive strength of this geopolymer was lower than S/L ratio of 0.6 due to crack created on the surface of the geopolymer. The presence of cracking in geopolymer will affect the loading ability of the product. The bigger of the higher crack size of content will reduce the strength ability of the concrete or geopolymer [22].

Functional Group Analysis. Fig. 4 displays the FTIR spectrum of metakaolin/dolomite geopolymer with different solid to liquid ratio. The broad band which clearly appeared at the band at around 3400 to 3300 cm^{-1} was attributed to the presence of the OH stretching frequency of silanol groups (Si-OH) bonded to the inorganic structure [23]. This band also attributed to the occurrence of hydrogen bonds that occurred between absorbed water and silanol group. The existence of those bonding was due to entrapped water molecule in geopolymer network after the geopolymerization process [24]. Normally water molecule was condensed during polycondensation process in geopolymerization. However, the water content still can entrap in the geopolymer structure.

At band 1650 to 1610 cm^{-1} OH^- bending was detected which implies the same interaction as for the silanol group. The vibration of ion CO_3^{2-} occurred at band 1400 to 1300 cm^{-1} which was attributed by carbonation reaction. During this pro-

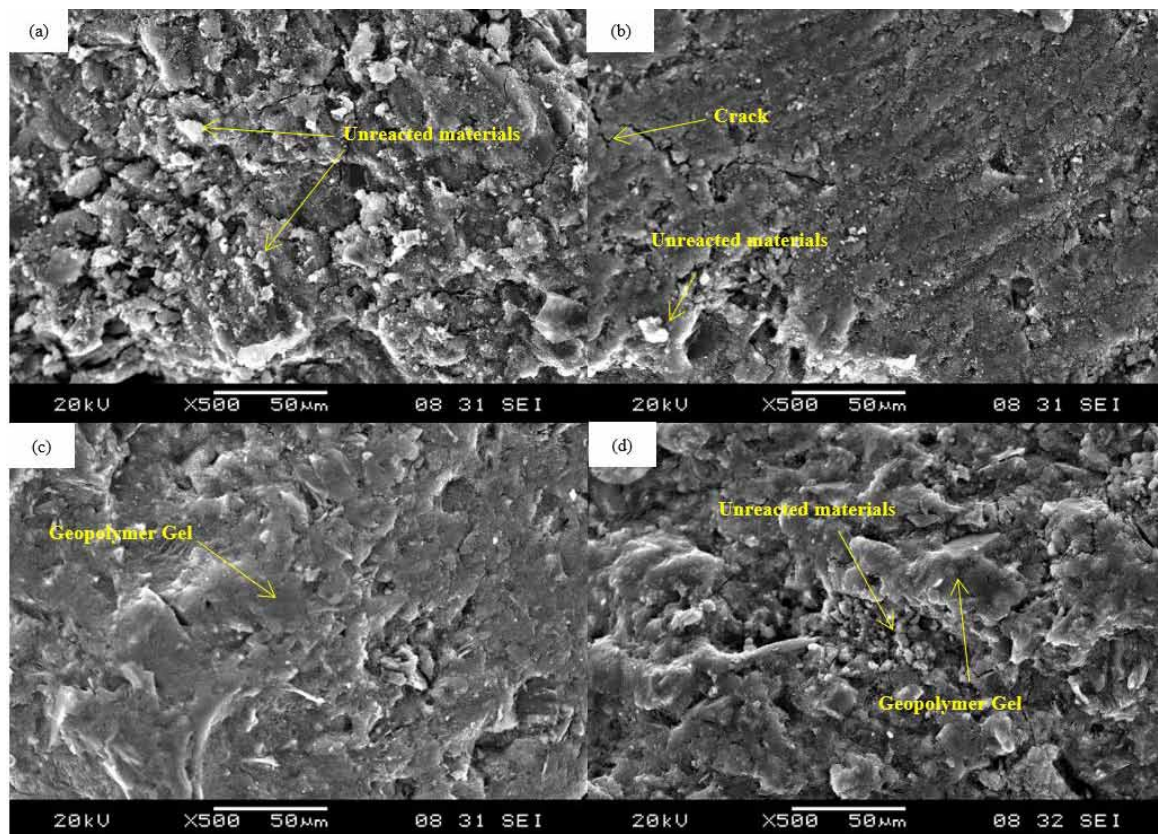


Fig. 3. The morphology of metakaolin/dolomite geopolymer for different solid to liquid ratio (a) 0.4, (b) 0.6, (c) 0.8 and (d) 1.0

cess, the penetration of carbon dioxide (CO₂) on the surface of the geopolymer took part. At this stage, CO₂ reacted with moisture in the pores and hydroxide (OH) from NaOH thus forms calcium carbonate (CaCO₃) [25]. The band attributed to asymmetric stretching vibration of Si-O-Si and Si-O-Al around the area 970 to 950 cm⁻¹. At this band, indicated the formation of aluminosilicate matrix. The peak of this band is sharp compared to other was due to the metakaolin usage which high of Si and Al content. The metakaolin used were 80% by mass.

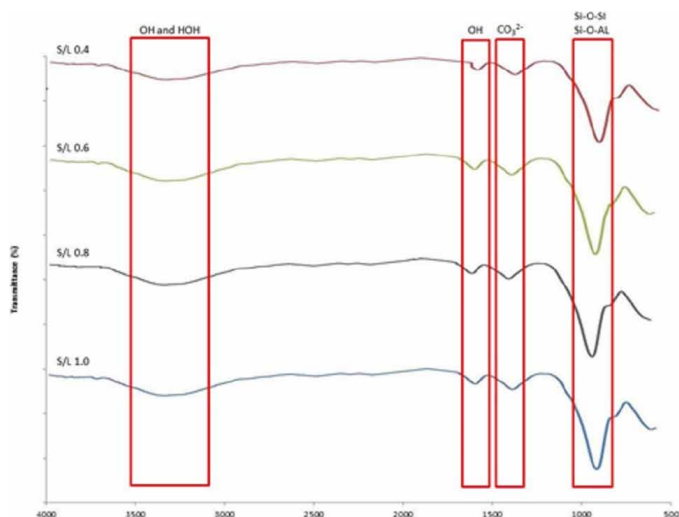


Fig. 4. FTIR spectrum of metakaolin/dolomite geopolymer with different S/L ratio

5. Conclusion

In a conclusion, the solid to liquid ratio of 0.8 has significant properties compared to other solid to liquid ratios. The phase analysis shows that the results are parallel to the compressive strength, morphology analysis, and functional group analysis. Phase analysis can help to elaborate the properties of geopolymer by detecting the major crystalline components which can exist in geopolymer systems. Besides that, a solid to liquid ratio of 0.8 is also shown to give better properties in compressive strength by achieving the highest strength. Lastly, in the morphology analysis solid to liquid 0.8 gave better surface observation..

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