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MORPHOLOGY ANALYSIS ON METAKAOLIN/DOLOMITE GEOPOLYMER UNDER HIGH TEMPERATURE EXPOSURE

This paper presents the morphology of the metakaolin/dolomite geopolymer after being exposed to high temperature and compare the findings with the morphology that has not been exposed to the temperature. The geopolymers were exposed at temperatures from 200°C up to 800°C. The geopolymer was a constant mix of 90% metakaolin and 10% dolomite with 10 NaOH molarity, 0.8 solid to liquid ratio, and 2.0 by mass of alkaline activator. The morphology of geopolymer exposed to high temperature contains many pores and as the temperature rises, the pores become huge, and a higher quantity of pores can be observed. The surface analysis, compressive strength, and water absorption test were also done to support the findings. The compressive strength calculation was based on weight loss. The lowest compressive strength loss was at 200°C temperature exposure with 10.98%. Meanwhile, the highest compressive strength loss was at 800°C temperature exposure with 48.54%. In comparison, this metakaolin/ dolomite geopolymer archive better properties compared to the concrete and metakaolin past studies.

Keywords: Geopolymer; Dolomite; Metakaolin; High Temperature

1. Introduction

Geopolymer analysis is currently on the way to international research booming scene 2010 towards the field of civil engineering. This was due to their capability in terms on strength, low density, low CO₂ emission, better heat exposure and lower cost [1]. Geopolymer can be made from metakaolin, fly ash, kaolin, slag, dolomite, and red mud through a process called geopolymerization [2,3]. Geopolymerization reaction includes the process of dissolution and condensation occurring at room temperature or slightly higher temperature [4]. The mechanism of geopolymerization involves three steps. Firstly, dissolution of aluminosilicate raw material. Next is decrease in the content of polymeric aluminosilicate species with increase in orthosilicate phase and lastly, condensation of tetrahedra and disappearance of polarized forms [5,6].

Previous studies have shown that the accumulation of appropriate amounts of Ca containing substances into geopolymers will have a significant impact on the structure and performance of geopolymers [7,8]. In some cases, the combination of Ca with the geopolymer system may trigger the development of C–S–H gels, thereby enhancing the structure of the geopolymer,

as indicated by the increase in compressive strength [9,10]. Thus, the mixture of metakaolin and dolomite will highly contribute to the recent studies about geopolymer applications.

Many researchers have conducted studies on the thermal properties of geopolymers and the mechanical properties at high temperatures. However, there were less studies on metakaolin dolomite geopolymer in high thermal applications. Therefore, the study of metakaolin and dolomite into the high thermal exposure can gain a great interest. Metakaolin/dolomite geopolymer can be studied to focus on high temperature exposure. This is because the strength of metakaolin geopolymer decreased by 51% when exposed to 800°C compared to Portland based material which was 80% [11]. With the addition of dolomite, the strength reduction can be reduced when exposed to the high temperature. In addition, the metakaolin/dolomite geopolymer can be applied as plastering applications.

2. Methodology

The materials used in this study were solid precursor which were metakaolin and dolomite, alkaline activator which were

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sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃). The metakaolin was obtained from the calcination of kaolin at 900°C for 8 hours. The physical form of kaolin used was powder type and has minimum 40% of particles sized less than 2 μ m and maximum 2% of moisture content. Dolomite was supplied by the Perlis Dolomite Industries Sdn. Bhd. Perlis, Malaysia. The dolomites undergo crushing process for 30 minutes and were sieved to obtained 150 μ m size.

The ratio of the metakaolin to dolomite was 90 : 10 and the ratio of solid precursor to alkaline activator was 0.8 which also called solid to liquid ratio. Samples preparation was done by mixing the solid precursor followed by addition of alkaline activator by using the mechanical stirrer. After mixed for 5 minutes, it was poured into $50 \times 50 \times 50$ mm mold and was removed 24 hours later. After that, the samples will undergo a heat exposure at 200, 400, 600 and 800°C. All the samples were tested and analyzed for further discussions.

3. Results and discussions

The results focus on the after effect of metakaolin/dolomite geopolymer after being exposed to 200, 400, 600 and 800°C temperature. The sample that did not undergo temperature exposure was also studied to compare the after effect.

3.1. Surface Analysis

TABLE 1 shows the appearance of metakaolin/dolomite geopolymer before exposed to the high temperature (control) and after exposed to 200, 400, 600 and 800°C temperature. Most of the samples change the colour from control sample. The colour changes from brown to dark grey. Besides that, there were also cracks observed on every sample after heat was applied to it. The cracks increase as the higher heat was applied. The observation towards the colour of the metakaolin/dolomite geopolymer also changes from light brown before temperature to dark brown as the heat increasing applied to it. The 800°C temperature exposure samples resulted the darkest brown colour among other temperature exposure.

Other than that, the 600°C and 800°C samples shrink compared to other samples. These shrinkages resulting from the

TABLE 1

Visual appearance of metakaolin/dolomite geopolymer before and after fire exposure at different temperature

Samples	Visual Observations
Control	
200°C	
400°C	
600°C	
800°C	

dehydration and dihydroxylation of the binders during heating. As the temperature increased to some elevated point, dihydroxylation process took part. The release of hydroxyl group (OH^{-}) from polyanion structure had occurred at dihydroxylation stage thus disturbing the geopolymer structure. This is supported by Zulkifly et al. [12] who reported that dihydroxylation tend to increase as the heating temperature to sample increased.

3.2. Morphology analysis

Fig. 1 shows the results for morphology analysis for metakaolin/dolomite geopolymer with (a) room, (b) 400°C and (c)



Fig. 1. The morphology analysis for metakaolin/dolomite geopolymer with (a) room, (b) 400°C and (c) 800°C temperature exposure

800°C temperature exposure. The pores and crack can be mainly seen in the 400°C and 800°C. This was due to the temperature exposed to the geopolymer will create pore spaces that provide escape routes for moisture in the matrix thereby decreasing the compressive strength of the geopolymer [13].

The pores mainly can be seen at the 800°C temperature exposure. Besides that, the cracks were also visible in the surface. The 400°C temperature exposure resulted a crack which was almost the same as 800°C temperature exposure however, there seems to be less pores created on the geopolymer surface. Compared to the room temperature exposure, the morphology of the geopolymer better. There were no cracks observed and only a few pores can be seen.

Study form Thomas et al. [14] conclude that the degradation of the paste begins to be visible and numerous pores appear because of the discontinuities generated by the dehydration. With the bound water completely lost, the C–S–H degrades and at these temperatures the portlandite decomposes [10,15]. Increased cracking and porosity indicated weakening of the matrix, which was responsible for the reduced strength at high temperatures [16].

3.3. Water Absorption Analysis

Fig. 2 shows the water absorption results after the samples were exposed to water for 24 hours. The metakaolin/dolomite geopolymer resulted an increment for the water absorption results. This was due to the product of the samples after exposed to the high temperatures.



Fig. 2. The rate of water absorption for metakaolin/dolomite geopolymer with different temperature exposure

The rate of water absorption for the 7 days curing of metakaolin/dolomite geopolymer increased from 0.43 at room temperature to 1.56% at 200°C. After that, the samples increased more at 400°C with value of 2.68%. The percentage keep increasing from 4.97% at 400°C and 6.75% at 800°C. Referred to the visual observation we can see that the temperature exposure towards the geopolymer produce cracks and shrinkage. This was the main reason for the increment of the rate of water absorption

[17,18]. The cracks enable the waters to flow through the samples and be absorbed into it. With the higher crack produced as the increment temperature exposure, making that the rate of water absorption increased [19].

Besides that, the porosity can be mainly observed in the morphology analysis before. The 800°C geopolymer samples observed to have manly small pores and a large crack. Compared to the control sample, the porosity and cracks were huge difference.

3.4. Density Analysis

The density of metakaolin/dolomite geopolymer after exposed to different high temperature was recorded in Fig. 3. The density of metakaolin/dolomite geopolymer decreased proportional to the increment of temperature exposure. The density of the geopolymer at room temperature was 4.15 g/cm³. After exposed to 200°C, the density of metakaolin/dolomite geopolymer became 3.05 g/cm³. The value of density keeps on deceasing as for the geopolymer exposed to 800°C gave the lowest values which were 1.41 g/cm³.



Fig. 3. The density for metakaolin/dolomite geopolymer with different temperature exposure

Based on the density result that gained, the reducing value of density was due to the weight loss of the geopolymer after exposed to the high temperatures. When the metakaolin/dolomite geopolymer exposed to the high temperature, the mass tends to decrease. Commonly, evaporation and dihydroxylation are the two properties that are supposed to be accountable for the weight loss of geopolymer at high thermal exposure [5,20]. To strengthen this statement, Fig. 4 shows the weight loss of the metakaolin/dolomite geopolymer.

These weight losses were due to the during the heating period, the moisture escaped from the geopolymer for more than half of the total mass loss. The heating of below 100°C the evaporate waters mainly came from the substantially attached water or free water set in the pores of the geopolymers which contributes about 55 to 60% of the whole water content in the geopolymer structure [21,22]. Meanwhile, the more heating



Fig. 4. The weight loss for metakaolin/dolomite geopolymer with different temperature exposure

above 100°C leads to disappearance of the chemically bonded water and the hydroxyl group OH inside the gel pores [23].

4. Conclusions

In a conclusion, the metakaolin/dolomite geopolymer shows increment of porosity and cracks when exposed to higher temperature. The water absorption and density analysis enhance the morphology results which causes the higher crack produced as the increment temperature exposure, making that the rate of water absorption increased. However, the compressive strength has not been discovered yet which will result better strength compared to Portland cement.

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