The Effect of Curing Temperature on the Properties of Kaolin Geopolymer Paste

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Abstract. This research paper focuses on the effect of curing temperature on the properties of kaolin geopolymer paste. Alternatively, kaolin was used as binders in making concrete paste in order to reduce the emissions of carbon dioxide (CO₂) to the environment. Kaolin geopolymer paste was made by mixture of kaolin and alkaline activators with 0.8:1 of solid to liquid ratio. The alkaline activator solutions were prepared with 0.2:1 ratio of Na₂SiO₃/NaOH. The mixture were placed in cube moulds (50 mm x 50 mm x 50 mm) and left for 24 hours until it hardened. Then, the samples were cured at 60, 70 and 80°C for 24 hours in the oven. The samples were then tested based on compressive strength, porosity, water absorption and morphology after days 28. Kaolin geopolymer paste with 70°C curing temperature is proved to have the highest compressive strength (1.04 N/mm²), lowest percentage of water absorption (1.71%) and lowest percentage of porosity (0.15%) compared to 60°C and 80°C curing temperature. The microstructure of 70°C curing temperature also shows that the geopolimerization was fully occurred with no pore.

1 Introduction

Geopolymer based on aluminosilicates and pozzolonic materials is used nowadays as binders for concretes to replace the Ordinary Portland Cement (OPC) in construction industries. Besides that, geopolymer is a potential materials that could be used in a lots of application such as in pipeline industries, construction, automotive, aerospace and many more. It has superb mechanical, physical, thermal and chemical properties [1]. Geopolymer has been used to reduce the emission of carbon dioxide (CO₂) to the air caused from the production of Ordinary Portland Cement (OPC) hence could also reduced the global warming pollution [2-3]. Geopolymer also has many advantages for instance lower water absorption, lower porosity and high compressive strength [4-5]. Kaolin was chosen in this research study because it is also aluminio silicate inorganic polymer like fly ash and slag which has good durability properties and could reduced CO₂ emission to the environment.

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2 Experimental Methods

2.1 Materials and Sample Preparation

Geopolymer paste in this research was made from a mixture of aluminosilicate (kaolin) and an alkaline activator. The alkaline activator consist of sodium hydroxide (NaOH) solution and sodium silicate (Na$_2$SiO$_3$) solution [6]. Kaolin is the mineral kaolinite which act as binder and a hydrous aluminum silicate formed by the decomposition of minerals. Kaolin is also white in colour. The NaOH pellet was used has the molar mass of 40.00 g/mol. To produce 12M NaOH solution, 1L distilled water was used to dilute 480g of NaOH pellets. The chemical composition for Na$_2$SiO$_3$ consists of 60.5% H$_2$O, 30.1% SiO$_2$ and 9.4% Na$_2$O [1].

Table 1 portrays the research details. The specimens then left for 28 days to be examined through various testing procedures such as compressive strength, water absorption, porosity and morphology.

Table 1. Mix design for this research

| Parameters/Raw Materials          | Value                      |
|----------------------------------|----------------------------|
| Solid/Liquid (S/L) Ratio         | 0.8: 1                     |
| Na$_2$SiO$_3$/NaOH Ratio         | 0.2:1                      |
| Mold Size                        | 50 mm x 50 mm x 50 mm      |
| Curing Temperature               | 60°C, 70°C and 80°C        |
| Curing Time                      | 24 hours                   |

2.2 Testing

2.2.1 Compressive Strength

Compressive strength testing was carried out according to ASTM C109/C109M-16a [7]. The compressive strength result was measured in the N/mm$^2$ or MPa unit.

2.2.2 Porosity

Porosity testings are crucial to determine the proportion of volume occupied by pores and usually state in percentage. The porosity result was obtained by using a pycnometer.

2.2.3 Water Absorption

Water absorption analysis was conducted to investigate the flows of water penetration into the paste. The percentage of water absorption was calculated by Eq. (1) according ASTM C642 [8], where:
Water Absorption (%) = \frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Dry Weight}} \times 100\% \quad (1)

2.2.4 Morphology

The morphological image of kaolin geopolymer samples can be observed by using a Scanning Electron Microscope (SEM). All samples were coated before observed by SEM to improved conductivity and tested at an accelerating voltage of 10-20 kV [9-10].

3 Results and Discussion

3.1 Compressive Strength Analysis

Fig. 1 shows the average compressive strength of kaolin geopolymer paste after 28 days for 60, 70 and 80°C curing temperature. From the Fig. 1, the compressive strength for kaolin geopolymer paste at 70°C stated the highest reading which 1.04 N/mm². At higher temperature of 80°C, the strength was reduced. It is because the occurrence of gel contraction, will affect loss of water molecules and shrinkage may occur in the samples. The granular structure of geopolymer paste mixture will break, hence the samples may not change to a more semi-crystalline form [11].

For this research, the samples were curing only for 24 hours. It is because the longer curing time enhanced the geopolymerization process, hence resulting in good compressive strength properties. However, curing at temperature 90°C and above for prolonged time will distorted the reaction leading to failure of the sample later on. With moderate elevation of reaction temperature, the compressive strength will be increased. To obtain better compressive strength properties, kaolin geopolymer paste need thermal activation [11].

![Fig. 1. Compressive strength for 60, 70 and 80°C of curing temperature.](image)

3.2 Porosity Analysis

Fig. 2 indicates the average porosity of kaolin geopolymer paste after 28 days for 60, 70 and 80°C of curing temperature. From the Fig. 2, the percentage of porosity for kaolin geopolymer paste at 70°C stated the lowest reading which was 0.15%. This shows that the reduction in pore size will lower the water absorption capacity.
However, the porosity of geopolymer samples at 60°C and 80°C stated 0.44% and 0.39% respectively, higher than samples at 70°C curing temperature. The geopolimerization at sample 60°C still not fully occurred which caused a few particles not fully dissolved. These undissolved particles were easily cracked under the action of external forces and will caused smaller pores exists. This will affect the porosity result. The larger pore had seen (Table 2) at 80°C curing temperature sample which caused more water will absorb to the sample and the porosity result was increased.

![Graph showing the porosity of geopolymer samples](image)

**Fig. 2.** Percentage of porosity for 60, 70, and 80°C of curing temperature.

### 3.3 Water Absorption Analysis

The results obtained in the compressive strength and porosity analysis are reflected in the water absorption capacity analysis results. The lower the value of the mechanical resistance (compressive strength result) higher is the absorption. It means that in the case of better cohesion of the consolidated products, the porosity decrease [11]. Fig. 3 shows the average water absorption of kaolin geopolymer paste after 28 days for 60, 70, and 80°C of curing temperature. From the Fig. 3, the percentage of water absorption for kaolin geopolymer paste at 70°C stated the lowest reading which was 1.71%. The percentage of water absorption of geopolymer paste decreases with a longer period of time. Water absorption values basically correlate with the porosity. This could be explained that when the pore present in the sample are low, thus the water that absorb into the sample are also low. Thus, the compressive strength will be high.

However, the water absorption of geopolymer samples at 60°C and 80°C stated 2.72% and 2.56% respectively, higher than samples at 70°C curing temperature. There were a few pores on the sample will cause more water was absorb to the sample and affect the water absorption result. The larger pore exist at 80°C curing temperature sample which caused more water will absorb to the sample and the water absorption result was increased.
3.4 Morphology Analysis

Table 2 shows the microstructure of kaolin geopolymer paste after 28 days for 60, 70 and 80°C of curing temperature. Microstructure for 70°C was highly homogenous and the geopolymerization was fully complete. Table 2 also display that the samples at 70°C became denser, more compact, better binding between kaolin and alkaline activators and shows less porosity present in the microstructure. It is proved that the kaolin geopolymer paste with 70°C of curing temperature had the highest compressive strength (1.04 N/mm²), lowest percentage of water absorption (1.71%) and lowest percentage of porosity (0.15%) compared to 60°C and 80°C of curing temperature. The durability potentially will be improved if the porosity and permeability decreased.

The value of compressive strength for sample at curing 60°C was slightly decreased because the structure less compact, loosely pack, less dense and had bigger precipitates which caused the the porosity and water absorption were increased. The sample at curing 80°C also had lower compressive strength value, higher porosity and higher water absorption value compared to 70°C curing sample because it had larger pore and larger precipitates. The larger pore seen in the Table 2 for samples 80°C curing temperature due to the space left by the dissolved kaolinite particles. This is because the geopolymer provides an exit route for moisture without destructing the geopolymer matrix during the heating process [12].
Table 2. SEM microstructure for X1000 magnification at 60, 70 and 80°C curing temperature.

| Temperature | SEM Microstructure |
|-------------|--------------------|
| 60°C        | ![SEM Image] (Pore; Geopolymerisation not fully occur) |
| 70°C        | ![SEM Image] (Geopolymerisation fully occur) |
| 80°C        | ![SEM Image] (Pore) |
4 Conclusion

Based on this research, the following conclusions were drawn:

a) Kaolin geopolymer paste with 70°C of curing temperature had the highest compressive strength (1.04 N/mm²), lowest percentage of porosity (0.15%) and lowest percentage of water absorption (1.71%) compared to 60°C and 80°C of curing temperature.

b) The microstructure of 70°C curing temperature also shows that the geopolimerization was fully occoured with no pore.

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