Effect of different ratio of geopolymer paste based fly ash-metakaolin on compressive strength and water absorption

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Abstract. This paper presents the effect of different solid to solid (S/S) ratio by mixing the fly ash and metakaolin as geopolymer paste on the compressive strength and water absorption. Fly ash is a waste materials produced by combustion of coal at power plant while metakaolin was obtained by calcining the kaolin at 750 °C for 4 hours. Different weight percentage of S/S ratio of fly ash/metakaolin used were 50%:50% (Mix 1), 70%:30% (Mix 2) and 90%:10% (Mix 3). Alkaline activators that act as binder were sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) solutions. The ratio of Na₂SiO₃/NaOH is 2.5 to 1. The mixture then placed into cylinder mould and left for 7, 14 and 28 days before it be tested. The product obtained were observed and it shows that Mix 2 has highest compressive strength for 28 days that was 41.22 MPa and has lower water absorption. The pore size decreases and permeability also decreases hence the strength potentially improve.

1 Introduction

Geopolymers as an alternative binder systems are becoming one of the main interest in research and development nowadays. Geopolymers can display outstanding technical properties, such as high strength, low water absorption, high acid resistance and also high temperature resistance [1,2]. It has been reported that geopolymer also can give a good performance by utilized the secondary raw materials (industrial wastes like fly ash). This explains the strong interest in this development from countries with growing industrialisation [3]. The use of waste for geopolymer production could not only solve a waste problem, but also reduce the consumption of primary raw materials.

Geopolymers are inorganic aluminosilicate polymers that form solid ceramic-like materials at near ambient temperature. The geopolymer cement is produced as an alternative way to replace the Ordinary Portland Cement (OPC). This is because the production of one ton of Portland cement emits approximately one ton of carbon dioxide (CO₂) an it will lead to global warming [4, 5]. Hence, the use of geopolymer technology not only will reduce the CO₂ emissions by the cement industries, but also enhancing the properties of the geopolymer paste.

One of the possible sources for making geopolymer binders is fly ash because it is available abundantly worldwide, and yet it is rarely used [6]. Therefore, consumption of fly ash in the manufacture of geopolymers is an important strategy in making concrete more environmentally friendly. Other than that, metakaolin is also one of the geopolymer binder. Metakaolin is obtain from the calcination of kaolin at a certain time and high temperature to remove its moisture. There have been some interests in the use of metakaolin as it probably the most effective pozzolanic material for use in concrete [7]. For this reason, fly ash and
metakaolin have been chosen as a base material for this project in order to utilize this industrial waste and to improve the paste properties such as compressive strength value and water absorption percentage.

2.1 Materials Selection

The materials that used to prepare the geopolymer paste was made from the mixture of fly ash and metakaolin. Fly ash used in this study was obtained from Lumut Power Plant, Perak, Malaysia while metakaolin used was purchased from Associated Kaolin Industries Sdn. Bhd. Malaysia. Fly ash and metakaolin undergo X-ray Fluorescence (XRF) analysis to characterize their chemical composition. Table 1 shows the compound composition for both materials.

| Table 1. The compound composition of fly ash and metakaolin. |
|---------------------------------------------|
| Compound | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | Na₂O | TiO₃ | ZrO₂ |
| Fly ash, wt (%) | 65.89 | 24.75 | 3.89 | 1.49 | 2.17 | 1.49 | 0.139 |
| Metakaolin, wt (%) | 55.70 | 38.60 | 2.03 | - | 2.43 | 0.78 | 0.0035 |

2.2 Sample Preparation

Samples were prepared and undergo mixing process for solid to liquid (S/L) ratio that was 1.2:1. Na₂SiO₃ and NaOH solution are most common alkaline activator that had been used in geopolymer paste. Firstly, the NaOH solution was prepared by dissolving the NaOH pellets in 1 L of distilled water and kept it at room temperature for 24 hours [8,9]. Then, the NaOH solution was mixed with Na₂SiO₃ solution with ratio of the mixture of Na₂SiO₃/ NaOH is 2.5:1. This mixture was prepared 24 hours prior to use [9]. The alkaline activator was later mixed with the fly ash and metakaolin as geopolymer based.

Fly ash and metakaolin were weighted and mixed according to the different S/S ratio named as Mix 1, Mix 2 and Mix 3. Table 2 shows the mixing proportion ratio for geopolymer paste based fly ash and metakaolin. The solid and liquid were mixed using automatic mixer then placed it in 50mm x 50mm cylinder mould, compacted and kept in the mould until it hardened and taken out after 24 hours. Then, the samples were cured at 60 °C in the oven for 24 hours [9]. The main purpose of this process was to remove the excessive moisture and give more strength to the sample [10]. The sample were then left for 7, 14 and 28 days at ambient temperature before undergo compressive strength and water absorption testing.

| Table 2. Mixing proportion ratio of fly ash-metakaolin (S/S) geopolymer paste. |
|---------------------------------------------|
| Mix No. | NaOH concentration (M) | Na₂SiO₃/NaOH | S/L ratio | S/S ratio |
| 1 | 12 | 2.5:1 | 1.2:1 | 50:50 |
| 2 | 12 | 2.5:1 | 1.2:1 | 70:30 |
| 3 | 12 | 2.5:1 | 1.2:1 | 90:10 |
2.3 Compressive Strength

Shimadzu machine was used for compressive strength test. Each sample was carried out by pressing the maximum load enforced to it by cross-sectional area, and outcomes were recorded in the unit N/mm$^2$ or MPa by using.

2.4 Water Absorption

The water absorption tests were conducted according to ASTM C642-06 [9]. The percent of water absorption for these samples were determined by Eq. (1).

$$\text{% Water Absorption} = \frac{\text{wet weight} - \text{dry weight}}{\text{dry weight}} \times 100\%$$

3 Results and Discussion

3.1 Compressive Strength

Fig.1 shows the effect of different S/S ratios on compressive strength of fly ash-metakaolin geopolymer paste in units of MPa at 7, 14 and 28 days. From Fig.1, it was observed that the overall results from Mix 1-3 shows increasing value in strength along with the curing time. The compressive strength initially increased up from S/S ratio of 50:50 (Mix 1) to 70:30 (Mix 2) but decreased with the S/S of 90:10 (Mix 3). Mix 2 exhibit highest compressive strength at 7, 14 and 28 days which were 21.65 MPa, 28.57 and 41.22 MPa respectively compared to Mix 1 and Mix 3. The strength increment in the fly ash-metakaolin geopolymer paste at the Mix 1 and Mix 2 might be due to the optimum quantities of binder and alkaline materials content that leads to higher degree of dissolution of source material and the formation of reaction products with lesser pores and more uniform aluminosilicate matrix. Hence, the mixture leads to optimum particle packing [11,12].

At Mix 3, the compressive strength was very low which was 15.61 MPa after 28 days. This is because the amount of liquid content was greater than the solid content which causes the mixture to become watery and the contact becomes limited between activating solution and reactive materials [8,12]. Thus, the dissolution rate of the fly ash-metakaolin became slower and difficult to be harden. Besides, there were many bubbles released which were not able to escape from the mixture may create pores thus producing porous structure which may weaken the geopolymer structure [13]. Hence, low compressive strength was reported at 90:10 (S/S) ratios.
Fig.1. Compressive strength of different ratios of mixed fly ash-metakaolin geopolymer paste at 7, 14 and 28 days.

3.2 Water Absorption

Fig.2 shows the results of water absorption in units of percent (%) for fly ash-metakaolin geopolymer paste after 7 days, 14 days and 28 days. Water absorption can be calculated by using Eq.1 [9].

\[
\% \text{ Water Absorption} = \frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}} \times 100\%
\]  

(2)

Fig.2. Water absorption of different ratios of mixed fly ash-metakaolin geopolymer paste at 7, 14 and 28 days.
From Fig. 2, it was observed that at Mix 2, the percentage of water absorption was the lowest after day 7, 14 and day 28 where the average absorption were 4.59%, 4.24% and 3.87% respectively. It proved that this mixture ratio of geopolymer paste was in good condition because has lower than 5% of water absorption. Low water absorption gives a good indicator of less open porosity [14]. Porosity can lead to sample crack and low compressive strength. Meanwhile, Mix 3 ratio has highest water absorption value compared to Mix 1 and Mix 2, may caused by the excessive alkaline activator content supply for fly ash-metakaolin geopolymer paste. Thus, it leads to low geopolymerization process [15,16].

Conclusions

Based on this research, it can be said that the Mix 2 which was 70:30 (S/S) ratio of fly ash-metakaolin geopolymer paste has achieved its highest compressive strength value and shows acceptable water absorption. It is due to sufficient amount of solid and liquid supplied. With this result, it shows that a balanced ratio between the fly ash, metakaolin and alkaline activator content was very important. This is because to obtain optimum dissolution and less unreacted particles left in the geopolymer matrix also can prevent cracking from occur.

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References

1. M. Weil, K. Dombrowski, A. Buchwald, Geopolymers, (2009)
2. S. N. Fifinatasha et al., Bandung Indones. Adv. Environ. Biol, 7, (2013)
3. D. M. J. Sumajouw, D. Hardjito, S. E. Wallah, and B. V. Rangan, J. Mater. Sci, 42, (2011)
4. N. S. M. F. Ahmed, M. F. Nuruddin, World Academy of Science, Engineering and Technology International Journal of Civil & Environmental Engineering, 5, (2011)
5. Z. F. Farhana, H. Kamarudin, A. Rahmat, M. M. A Bakri and S. R. Shamsudin, AIP Conference Proceedings, (2016).
6. S.E. Wallah and B.V Rangan, (2013)
7. J.M. Khatib, O. Baalbaki, A.A Elkordi, Metakaolin. Waste and Supplementary Cementitious Materials in Concrete, (2018)
8. S. Aparna, D. Sathyan, K. Anand, Materials Today: Proceedings, 5, (2018)
9. Z. F. Farhana, H. Kamarudin, A. Rahmat, M. M. A Bakri, Materials Science Forum, 803, (2014)
10. I.H Aziz, M. M. A Bakri, H.C. Yong, L.Y. Ming, H. Kamarudin, Matec Web of Conferences, 78, (2016)
11. M. Ibrahim, M. A. M. Johari, M. K. Rahman, M. Maslehuddin, Const. Buil. Mater., (2017)
12. N.A. Jaya, L. Y. Ming, H. C. Yong, M. M. A Bakri, (2018)
13. K. Zulkifly, H.C. Yong, L.Y. Ming, M. M. A Bakri, S. F. A. Abdulllah, (2018)
14. F. H. A. Zaidi, R. Ahmad, M. M. A. B. Abdullah, M. F. M. Tahir, Z. Yahya, W. M. W. Ibrahim, A. S. Sauffi, *IOP Conference Series: Materials Science and Engineering*, (2019)

15. F. Zainal, S. Amli, K. Hussin, A. Rahmat and M. Abdullah, *IOP Conference Series: Materials Science and Engineering*, **209**, (2017)

16. N. S. D. M. Azhar, F. F. Zainal, M. M. A. B. Abdullah, *AIP Conference Proceedings*, (2019)