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Effect of Particle Size on Properties of Sidoarjo Mud-based Geopolymer

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Introduction

Effort to utilize the abundant material from mud volcano eruption in Sidoarjo, East Java, Indonesia has been going on since its eruption in May 2006. The volcanic mud has covered not less than 640 hectares of fertile land. In some areas, the height of the embankment is up to 12 m. The disaster needs to be mitigated. It has been established that the mud having a crystalline microstructure and high shrinkage. It cannot be used as construction material in its initial condition [1]. The total content of SiO₂, Al₂O₃ and Fe₂O₃ in Sidoarjo mud is higher than 80%. This makes it possible to be used, but only after heat treatment is applied to convert its crystalline structure.

Calcination of the Sidoarjo mud was found to change its microstructures, and render it possible to be used as pozzolanic material and geopolymer precursor [2-5]. Calcination temperature at 700° C-900°C is considered enough to change its crystalline microstructure to be amorphous, and hence to make it more reactive [3]. The use of Sidoarjo mud as pozzolanic material can replace up to 60% of cement content with the Strength Activity Index (SAI) of 85% at 28 day, and more than 90% at 56 day [5]. As a geopolymer precursor, the use of 100% calcined Sidoarjo mud as cementing material has been made possible [3-4]. However, to date no report is available on the influence of particle size of the calcined mud on the properties of geopolymer mortar. This paper reports the effect of particle size affected by milling time on the calcined Sidoarjo mud to make geopolymer mortar. The durability properties is also reported in this paper, i.e. sulphate acid resistance, chloride penetration resistance, and shrinkage of the Sidoarjo mud-based geopolymer.

Experimental Program

Materials. Sidoarjo Mud used in this study were taken directly near the centre of eruption. Sidoarjo mud should undergo several processes before it can be used as reactive material. The mud was

formed into brick-shape for easy handling, dried in the laboratory oven at 110°C for 24 hours to reduce its water content (Fig. 1). Then, the bricks were transported to local roof tile manufacture kiln to be heated to peak temperature of 945°C for 5 hours and gradually cool to room temperature for 36 hours. It was observed that the cross section of the brick have some unevenness of the color showing that there were some un-uniformity of the calcination process. However, the calcined mud was mixed uniformly once it was ground in the milling machine. The XRF test results and the LOI content are shown in Table 1.

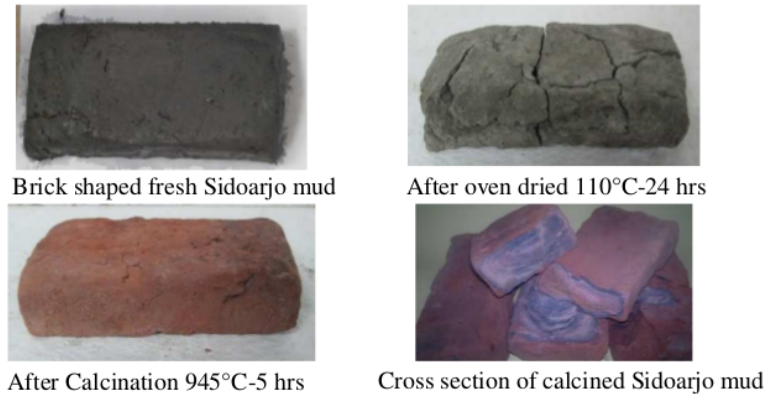


Fig 1: Physical Changes of Sidoarjo Mud before and after Heat Treatment

Table 1: XRF Analysis Results Sidoarjo Mud Sample (% by mass)

Oxide	Content (%)	Oxide	Content (%)
SiO ₂	56.75	SO ₃	0.96
Al ₂ O ₃	23.31	TiO ₂	0.38
Fe ₂ O ₃	7.37	MnO	0.14
CaO	2.13	Cr ₂ O ₃	0.01
K ₂ O	1.04	Na ₂ O	2.7
MgO	2.95	LOI	1.2

Milling. The calcined brick of Sidoarjo mud was ground in the cylindrical steel grinding machine with rod steel bar as the grinding tools [5]. Milling was conducted for 2, 4 and 8 hours, respectively, to investigate the effect of fineness of the calcined Sidoarjo mud to its reactivity as geopolymer precursor in making geopolymer mortars. To ensure the fineness is under 63 μm , the powder was sieved using #200 sieve. The particle size analysis results show that with longer grinding time the specific surface area is increased, although not in linear trend (Table 2). As the longer milling time applied, there could be some agglomeration happened on the mud particles. Even though the reactivity can be increased, but it was not detected by the measurement.

Table 2: Particle Size Analysis of Calcined Sidoarjo Mud

Grinding Time (hours)	Specific Surface Area (m ² /g)	d(0,1) (μm)	d(0,5) (μm)	d(0,9) (μm)
2 hours	1.30	1.832	15.180	475.604
4 hours	2.34	0.937	7.991	114.402
8 hours	2.58	0.768	6.632	342.661

Geopolymer mixtures and specimens. Sodium Hydroxide flake and Sodium Silicate solution were used as the alkaline activator. The purity of Sodium Hydroxide flake was up to 99%, while the composition of Sodium Silicate were 17.14% Na₂O, 36.71% SiO₂, and the ratio of SiO₂ and

Na_2O was 2.14. Water to binder ratio, by mass, was kept constant at 0.3, ratio of sand to mud was 2:1 by mass, and NaOH solid to Sodium Silicate solution ratio was set at 1:2, based on previous experiment. Molarity of NaOH solution was 10M. The Sidoarjo mud geopolymer was cast into mortar cube sample of $5 \times 5 \times 5 \text{ cm}^3$ size and vibrated on vibrating table to remove the entrapped air. Curing was performed in laboratory oven at 100°C for 24 hours. Each compressive strength data was average of 3 samples measured at 7 days testing.

Testing. The workability of fresh geopolymer mortar was measured by the flow table apparatus, while the compressive strength was measured by testing the mortar cube sample of $5 \times 5 \times 5 \text{ cm}^3$ at 7 days. Acid resistance was measured by strength changes, by mass, after immersion in 10% sulphate acid [6], starting from day 3. Chloride penetration was measured by non-steady state migration test based on NT Build 492 [7]. Shrinkage of the specimen was determined using dial gauge set on cylindrical specimen of diameter 100 mm and height 200 mm.

Results and Discussion

Influence of particle size. The variation of particle size of calcined Sidoarjo mud is indicated by the difference in milling time duration from two to eight hours. The effect of the particle size on the fresh and hardened geopolymer properties is shown in Fig. 2. Milling time of 8 hours resulted in a more workable mixture, which is consistent with the finding from the pozzolanic activity of Sidoarjo mud [5]. Most probably, finer particle is dissolved by the alkaline activator at higher rate due to having more soluble content. Compressive strength of the geopolymer mortar is showing the same trend as the one of high volume Sidoarjo mud mortar. Finer particle of the calcined Sidoarjo mud reacts faster, resulted in higher strength. Strength increase of 55% was obtained by increasing the milling time of the mud from 2 to 8 hours.

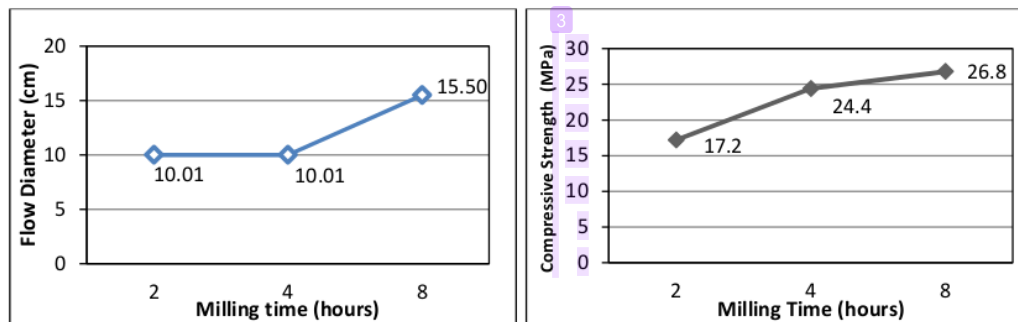


Fig. 2: Flow of the fresh geopolymer mortar and 7 days compressive strength by varying the milling time

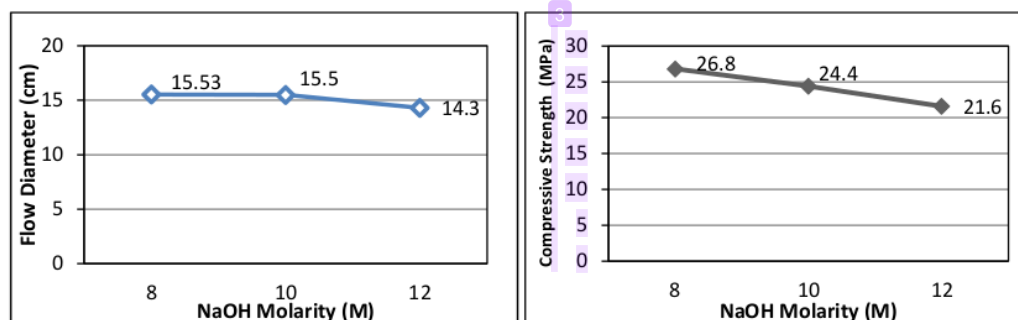


Fig. 3: Flow of the fresh geopolymer mortar and 7 days compressive strength by varying Sodium Hydroxide Molarity

Molarity of alkaline activator. Fig. 3 shows the influence of the sodium hydroxide molarity on the fresh and hardened geopolymer mortar properties. The behavior of the fresh mortar did not vary significantly with the changes of the NaOH molarity. The figure also shows that there is slight reduction on the compressive strength of the geopolymer mortar with the increase of the NaOH molarity.

Sulphate Acid Resistance. Ten percent – by mass - of Sulfuric acid solution was used to accelerate the testing duration to measure the resistance of Sidoarjo mud-based Geopolymer to sulfuric acid attack. 8 hours grinding time of Sidoarjo mud was used. The mortar was soaked from day-3 until day-56. It was found that there was almost no change in compressive strength with the increase of time of exposure (Fig. 4). The strength loss up to 56 days is considered good when comparing to the fly ash geopolymer [6].

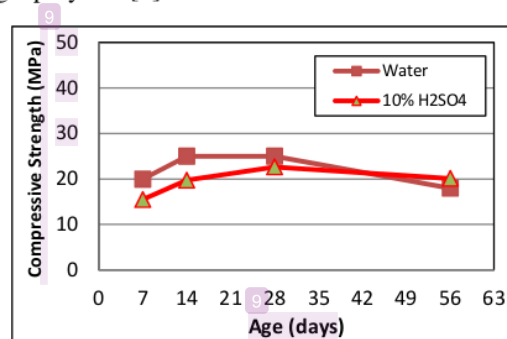


Fig. 4: Compressive strength of the Sidoarjo mud geopolymer submerged in water and sulphate acid.

Chloride Ion Penetration Resistance. Chloride Ion Penetration Resistance was measured by Rapid Chloride Migration Test [7] to evaluate the permeability of the geopolymer mortar. Calcined Sidoarjo mud with 8 hours grinding time was used. The results show that the non-steady state migration coefficient (D_{nssm}) values obtained was $6.437 \times 10^{-12} \text{ m}^2/\text{s}$ (Fig. 5). This result is comparable to the previous study on geopolymer mortar made from fly ash, with a value D_{nssm} $4.849 \times 10^{-12} \text{ m}^2/\text{s}$, and also to the High Volume Fly Ash Concrete with 60% fly ash content and water to binder ratio of 0.3 that has D_{nssm} value of $14.345 \times 10^{-12} \text{ m}^2/\text{s}$ [8]. The smaller migration coefficient indicates higher resistance to penetration of chloride ions.

Shrinkage. Fig. 6 shows the measurement of the shrinkage of the geopolymer mortar measured by using a dial gauge daily until 28 days. The shrinkage is shown to occur in a faster rate during the earlier age of the mortar, especially until day 16, with the value of 675 micro strain. After that there is no significant increase of strain. The shrinkage of the Sidoarjo mud-based geopolymer mortar is larger than one of the fly ash-based geopolymer as reported by Wallah [9]. The shrinkage of the fly ash geopolymer was measured at 100 micro strain in early age at about 20 days, and afterward did not increase again.

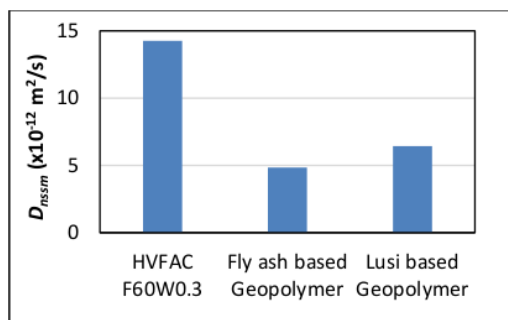


Fig.5: Diffusion Coefficient of the Geopolymer

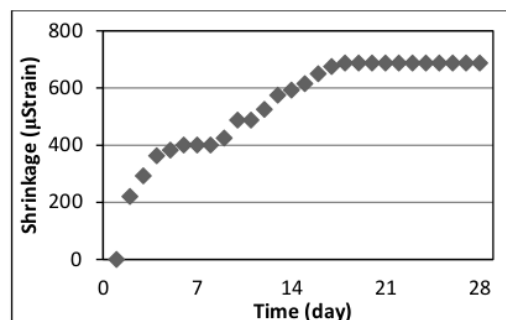


Fig. 6: Shrinkage of the Geopolymer

Conclusion

This study investigates the effect of the particle fineness of the calcined Sidoarjo mud to be used as a geopolymer precursor. Based on the results and discussion, there are several conclusions that can be drawn, i.e.:

- a. The finer particle of the calcined Sidoarjo mud has better reactivity, thus resulted in higher compressive strength of geopolymer mortar. Milling time of 8 hours with SSA of 2.58 m²/g in the rod mill resulted in an increase in compressive strength about 55%, compared to the one of 2 hours milling time with SSA of 1.30 m²/g.
- b. The variation of sodium hydroxide molarity in between 8 – 12 M does not show significant difference in the compressive strength, indicating that the use of alkaline activator can be reduced.
- c. Durability properties of the Sidoarjo mud geopolymer measured by sulphate acid resistance and chloride penetration shows that it has comparable properties to fly ash-based geopolymer.
- d. Shrinkage measurement of the Sidoarjo mud geopolymer shows significantly larger value compared to fly ash geopolymer. However, long term shrinkage was not observed in this study.

Acknowledgements

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