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Effects of Calcination Temperature of LUSI Mud on the Compressive Strength of Geopolymer Mortar

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Abstract. The abundant availability of LUSI (a short form of LUmpur SIdoarjo or Sidoarjo mud) mud of a mud volcano located in Sidoarjo, East Java, Indonesia, attracts interest of researchers to seek the possibility of utilizing it; among them is as construction material. This study focuses on the effect of calcinations temperatures of LUSI mud on the compressive strength of geopolymer mortar. Three different calcinations temperatures were investigated, i.e. 700, 800 and 900°C for five hours duration. Characterization of the mud, both the original and the calcined ones, was performed by using X-ray Diffraction (XRD) and X-ray Fluoresence (XRF) analyses. The calcined LUSI mud was then employed as precursor for making geopolymer mortar, and tested for its 7-day compressive strength. It is found that calcinations at 800°C is the optimum calcinations temperature producing the highest compressive strength.

Introduction

Since May 2006, a mud volcano in Sidoarjo, East Java, Indonesia, continues to erupt, producing mud which has covered more than 640 hectares of fertile land, industrial and housing areas, as well schools and other infrastructure facilities. The mud, nicknamed LUSI (a short form of *Lu*mpur *Si*doarjo or Sidoarjo mud) is predicted to flow out not less than 41 years with 50% chance [1].

The mud volcano has discharged approximately18*10^4 m^3 per day of mud in its peak in 2007, and it continues till present, although the volume is decreasing to about 10^4 m^3 per day. Several attempts have been done to end the eruption, however to date none was successful. Currently, the height of the cover dam is about 12 meters, although some of the mud has been channeled to a nearby river.

Attempts to utilize the mud as construction materials were mostly carried out to partially replace the use of cement in making concrete as pozzolanic material [2-4]. This is reasonable as it was found to contain SiO_2 ~ 55%, Al_2O_3 ~ 20% and Fe_2O_3 ~ 10% [4], although recently it was revealed that its main chemical composition shifted to SiO_2 ~ 30%, Al_2O_3 ~ 6% and Fe_2O_3 ~ 43% [2, 3]. The mud becomes more reactive after undergone calcinations, as its microstructure changed from crystalline into more amorphous form [3, 4]. Calcinations or sintering is a common practice to increase the pozzolanic activity of a pozzolanic material [5].

Nuruddin et al [4] found that calcinations at 600°C for one hour duration was the most effective to convert the mud to become a reactive pozzolanic material, with 10% cement replacement resulted in the highest compressive strength. The authors previous study [2] reported that by calcinations at 900°C for five hours, with particle size less than 63μm, successfully enabled the use of up to 40% LUSI mud to partially replace the use of cement in making mortar.

As most of pozzolanic materials are also potentially used as a precursor for geopolymer [6], this LUSI mud should be a strong candidate for it as well. From SEM results, it was found that LUSI is dominated by clay minerals, whereby its particle size is less than 10μm. The shape of the particle is plate-like structure [7]. However, without calcinations the mud is un-reactive due to its crystalline microstructure [8], and thus it needs heat treatment before use.

This paper focuses on the effect of calcinations temperature of LUSI mud on its microstructure and on the compressive strength of LUSI mud-based geopolymer mortar

Experimental Program

Materials. Locally available granite type river sand was used throughout the study. The specific gravity of the sand was 2.603, while the fineness modulus (FM) was 2.16. LUSI mud was collected directly from the site of mud volcano – from point 25, the closest to the center of eruption, in liquid form - in Sidoarjo, East Java, Indonesia. Sodium silicate was obtained in liquid form with Na_2O 17.14% and SiO_2 36.71%, while sodium hydroxide (NaOH) was purchased in flakes form with 98% purity. The water utilized to mix the NAOH flakes was tap water.

Calcinations of the mud. The raw mud was then dried under the sun and molded into bricks-like shape of 30x60x30mm size, and sent to oven at 100°C for 24 hours for further drying. The dry mud in bricks-like shape was then sent for calcinations in a laboratory furnace at three different temperatures, i.e. 700, 800 and 900°C for five hours. Five hour duration was followed to conform with the common practice in calcinations of ceramic tile roof in local manufacturers [2]. After the calcinations period, the calcined mud was allowed to cool down gradually in the furnace until the temperature similar to the outside or surrounding environment. The calcined mud was then sent to a rod-mill machine for grinding for about two hours, and then sieved to obtain fraction with particle size less than 63µm. Characterizations of mud were performed using XRD and XRF analyses.

Design and synthesis of geopolymer mortar. Alkaline solution used was a combination of NaOH and sodium silicate solution. The ratio of NaOH solid:sodium silicate solution was taken as 1:3 by mass. Water to the mud ratio of the geopolymer mortar was fixed at 0.4 by mass. This water is the one used to make the NaOH solution. Sand to LUSI mud ratio by mass was taken as 3:1. This composition was chosen based on the results of a preliminary investigation by the authors. The fresh geopolymer mix was then cast to be mortar cubes samples of 50x50x50mm size, and vibrated on the vibrating table for one minute to expel the air trapped in the fresh mix. Curing was performed in the oven at 100°C for 24 hours. Each compressive strength data of mortar sample was obtained as mean value of the results of three tests performed at the age of 7 days.

Results and Discussion

Mud properties. Table 1 shows the oxides composition of the original and calcined LUSI mud obtained from XRF analysis. The first row shows the chemical composition of the original LUSI mud before calcinations (designated as **BC**), while the second row shows the chemical composition after calcinations (designated as **AC**) at 900°C for five hours.

Table 1. Chemical composition of original and calcined LUSI mud as measured by XRF (% by mass)

	CaO	SiO_2	Al_2O_3	Fe_2O_3	K_2O	SrO	SO_3	MnO	ZnO	CuO	P_2O_5	TiO_2	ZrO_2
BC	7.63	28	5.7	41.42	4.50	0.00	2.1	0.64	0.13	0.27	1.5	2.90	0.33
AC	6.76	36	7.0	39.82	3.96	0.48	0.3	0.57	0.11	0.25	1.9	2.76	0.20

It was found that the most dominant oxides in the mud are SiO_2, Al_2O_3 and Fe_2O_3. The compositions are slightly different to those reported earlier [4], whereby the SiO_2 content was found to be higher, about 55%. Examination on the oxide compositions of the LUSI mud taken from five different places do not give any significant different to those presented in Table 1 [2]. The total amount of SiO_2, Al_2O_3 and Fe_2O_3 is more than 82%, and thus satisfy the requirement for pozzolanic material [9]. Table 1 also shows that after calcinations (**AC**), the percentage of SiO_2 in the mud increases significantly from 28 to 36%. The same tendency is also shown for Al_2O_3.

Figure 1 shows the XRD pattern of the LUSI mud before calcinations, while Figures 2, 3 and 4 exhibit the ones after calcinations at 700, 800 and 900°C for five hours in the laboratory furnace. From the patterns showed, calcinations convert the crystalline micro-structure of LUSI mud into more amorphous or semi-crystalline material.

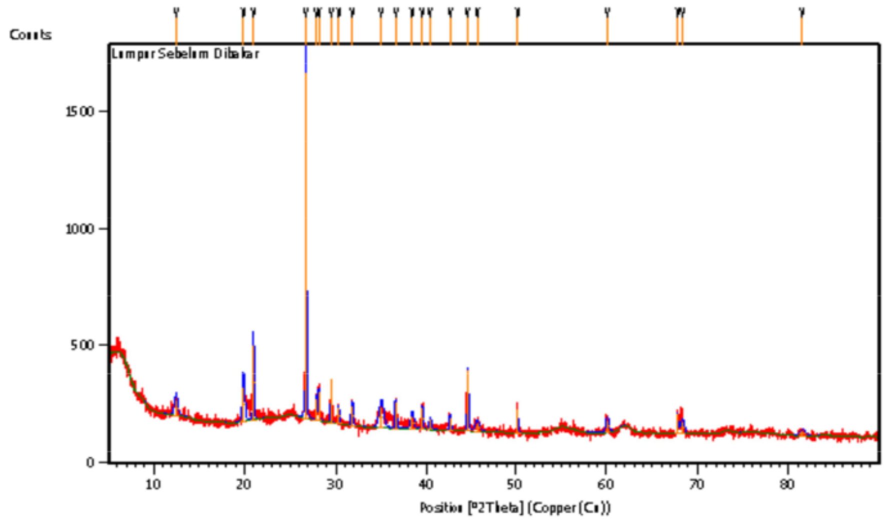

Figure 1. XRD pattern of LUSI mud before calcination

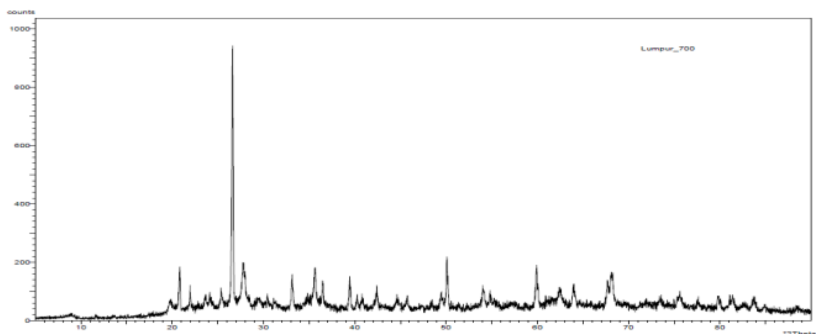

Figure 2. XRD pattern of LUSI mud after calcinations at 700°C

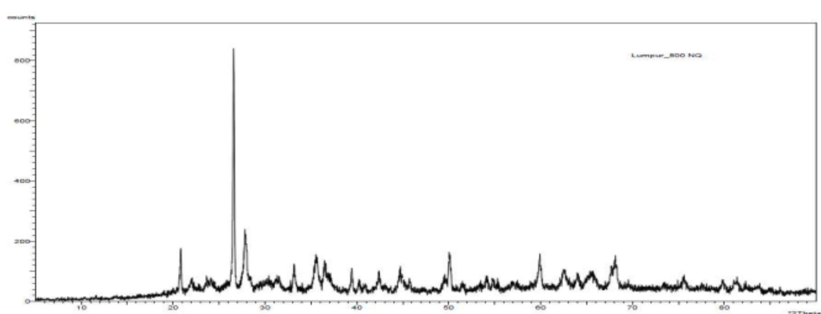

Figure 3. XRD pattern of LUSI mud after calcinations at 800°C

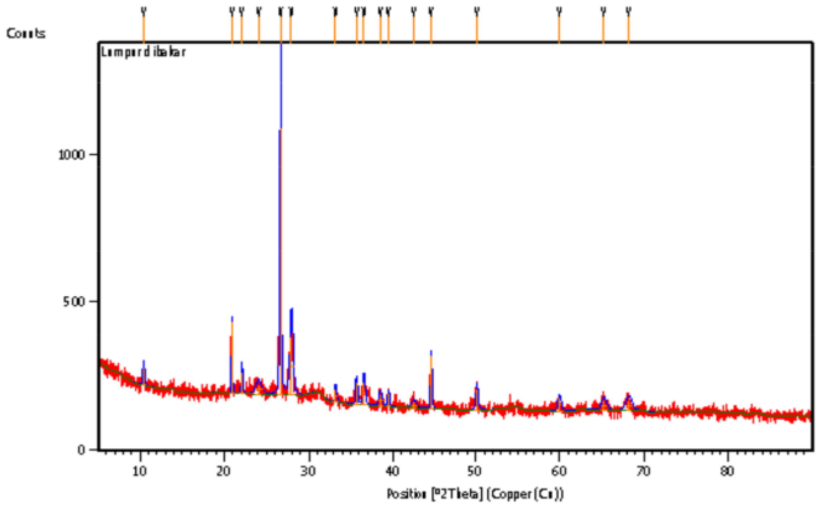

Figure 4. XRD pattern of LUSI mud after calcinations at 900°C

Comparing Figure 2 to 4, it is revealed that calcinations at 800°C seem to be the optimum temperature, as it shows the least intensity of peaks compared to those obtained after calcinations at 700°C and 900°C.

The effect of calcinations temperature to compressive strength of geopolymer mortar. Table 2 shows the compressive strength of geopolymer mortar at 7-day of age manufactured using LUSI mud calcined at three different temperatures, i.e. 700, 800 and 900°C for five hours in the laboratory furnace. The one manufactured from binder calcined at 800°C shows the highest 7-day compressive strength of 36.7MPa. The results confirmed the prediction made after observing the XRD patterns of LUSI mud calcined at different temperatures, i.e. calcinations at 800°C produces the least peaks in the XRD pattern. However, Nuruddin et al [4] found that calcinations of LUSI mud at 600°C produced the optimum pozzolanic material, while Elimbi et al [10], who worked with kaolinite clay, concluded that 700°C was the most convenient temperature for calcinations of source material for geopolymer. Table 2 also shows the unit weight of the mortar samples, which is around 2150 kg/m^3.

Table 2. Effect of calcinations temperature on the compressive strength of geopolymer mortar

	Calcination Temperature		
	700°C	**800°C**	**900°C**
fc' (MPa)	26.60	36.67	18.27
Unit weight (kg/m^3)	2114.7	2185.3	2164

Conclusion

This study investigates the effect of calcinations temperature on LUSI mud to be used as precursor for manufacturing geopolymer mortar. It is found that calcinations at the appropriate temperature converted the originally-crystalline microstructure of LUSI mud into more amorphous or semi-crystalline one. Calcination temperature of 800°C for five hours duration is the optimum calcinations temperature, whereby the intensity of peaks in the XRD pattern reduced significantly. The results is confirmed with the compressive strength of the geopolymer mortar manufactured using the calcined mud, whereby the one produced using mud calcined at 800°C shows the highest compressive strength.

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