Influence of Heat Treatment Temperature, Particle Fineness and Replacement Ratio of Sidoarjo Mud as Pozzolanic Material

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ABSTRACT: The potential of treated Sidoarjo Mud as pozzolanic material has been investigated previously with positive results by applying heat treatment by burning it at high temperature. This study focusses on the influence of heating temperature, particle fineness or nominal size and cement replacement ratio of treated Sidoarjo mud in mortar. The treated Sidoarjo mud was characterized by a series of test of Thermal gravimetric Analysis (TGA), X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF). The heating temperature variation applied were at 500, 600, 700, and 800°C for two hours, and oven dried but untreated sample for comparison. The particle sizes were varied by sieving passing size 600-150, 150-75, and <75µm, while the percentage of cement replacement ratio was of 10, 20, and 30% by mass. The results show that the burned mud at 600°C, fineness <75µm, and replacement ratio 10% produce highest compressive strength. Heating the mud at 500-800°C would make it become pozzolanic whereas the unburned mud is not eligible to be used as pozzolanic material

1. INTRODUCTION

Sidoarjo mud volcano that erupted in May 2006 in Porong, Sidoarjo, Indonesia had caused significant environmental and social damage to the surrounding area. To date it spews more than 10.000 m^3 of mud material daily and nothing can be done with this enormous amount of material except just containing it by coverdam and flowing the excess materials to the sea through Porong River. It is estimated that the outflow of mud material will continue for 25-30 years [1].

Sidoarjo mud is measured to be containing 53.08% Silica and 18.27% Alumina [2] has a potential to be used as a pozzolanic material, but initial studies have shown that the untreated mud cannot be used as cement replacement or addition in concrete because it unreactive, high water absorption and also detrimental to the concrete mix [3]. In 2010, Nurudin et al [4] showed that by burning the mud at a temperature of 600°C for 2 h and ground the particles to fineness of <100µm, it can be used as a pozzolanic material. Cement replacement of 10% mud yielded the best compressive strength.

In this paper, we would like to investigate more on the optimum heating temperature, the effect of particle size on the compressive strength and pozzolanic activity and also the replacement ratio of the treated Sidoarjo Mud. The variation of heating temperature need to be investigated to see the effect of different temperature on the pozzolanic activity of the mud. Different particle size and its effect on hardened concrete also need to be studied, The research is expected to provide solutions to overcome the Sidoarjo mud flow and its material abundance so it can be economically used as cement replacement in making concrete.

2. EXPERIMENTAL STUDY

2.1 Materials

Cement material used was Portland Pozzolan Cement (PPC) made by local manufacturer, as Ordinary Portland Cement (OPC) was not available on the market anymore. Sand was obtained from local quarry in Mojokerto with fineness modulus (FM) of 1.514 and specific gravity of 2.785.

Mud slurry were obtained at location P70A from Sidoarjo mudflow area, and placed in electrical oven at 110°C for 24 hours to obtain dried mud. The dried mud was then heat treated to a specified temperature in laboratory furnace (Figure 1). After heating the mud were finely ground.

The results of X-ray fluorescence (XRF) analysis conducted on the dried mud and after heating sample is shown in Table 1. For comparison, the XRF analysis of previous research was included also. It was shown that there was a significance different in oxide component measured from the Sidoarjo mud compared to the previous research. The silicon oxide and aluminate oxide that was higher in percentage was now reduced, and iron oxide was now the largest component measured up to 41.4%, while in the previous research it was only about 5-10%. This could mean that there are changes on the mud material composition from different layer of the earth crust. According to ASTM C618 [5], the terms of a Pozzolan material that is the total content of compounds SiO_2, Al_2O_3, Fe_2O_3, and minimum 70%, mass loss due to burning a maximum of 10%. SO_3 content of a maximum of 4%, with this definition, the Sidoarjo mud still could be classified as pozzolanic materials.

Figure 1 Oven dried mud and heating in laboratory furnace

Table 1 XRF analysis result of Sidoarjo Mud

Oxide	year 2006 [2] (%)	year 2009 [4] (%)	dried mud (%)	500°C (%)
SiO_2	53.08	54.9	28	33.4
Al_2O_3	18.27	20.44	5.7	6
Fe_2O_3	5.6	10.64	41.4	37.1
CaO	2.07	4.9	7.63	7
K_2O	1.44	2.35	4.5	4.08
Cl		1.14	3.7	2.5
TiO_2	0.57	1.16	2.9	2.5
SO_3	2.96	1.31	2.1	2.6
P_2O_5		1.06	1.5	1.6
MgO	2.89	1.76	1	2
Others		0.34	1.55	1.2

2.2 Specimens and Testing Conducted

Pozzolanic effect of the heat treated Sidoarjo mud was conducted on concrete mortar cube of 5×5×5 cm^3. Constant binder to sand ratio by weight was kept at 1:2.5, and the water binder ratio was 0.5 by weight.

Compressive strength test was conducted on age 7, 28 and 56 days with three specimens for each variable. Porosity as measured by water absorption test was conducted on the specimens at 28 days. Curing of the mortar specimen was conducted by immersing in water and taken out one day before the compressive testing.

Strength Activity Index (SAI) [6] is the ratio of compressive strength of concrete containing Pozzolan material with standard concrete containing no Pozzolan material. SAI was measured for all variable to determine the reactivity of the pozzolanic materials after heat treatment. The SAI of a minimum of 75% was considered to be sufficient, considering the abundance material of the Sidoarjo mud need to be used as much as possible.

The experimental study was conducted in three stages by varying heating temperature, mud particle size and cement replacement ratio. Heating temperature of 500, 600, 700 and 800°C. The particle sizes were separated by passing mesh #30 (600µm), #100 (150µm) and #200 (75µm). Cement replacement ratio conducted was 10%, 20% and 30%. Specimen with no mud and 20% cement replacement of oven dried but unburned mud were also made as references.

3. RESULTS AND DISCUSSIONS

3.1 Thermal Gravimetric Analysis

Thermal Gravimetric Analysis (TGA) was performed on solid mud that have not been heat treated. The results of the analysis (Figure 2) shows that the mud mass decreased with increasing temperature given. There are two peak points, which are at 45.92°C and 467.5°C, which means at both temperature there was the greatest decrease of mud mass. Initial peak point were caused by the loss of moisture (H_2O) in the mud, while the second peak point indicates the beginning of the change in crystal form in the mud to become amorphous phase. After the second peak, there was no more significant mass reduction up until temperature of 1400°C. From this result, it can be determined that the heating temperature should be conducted at temperature higher than 467.5°C.

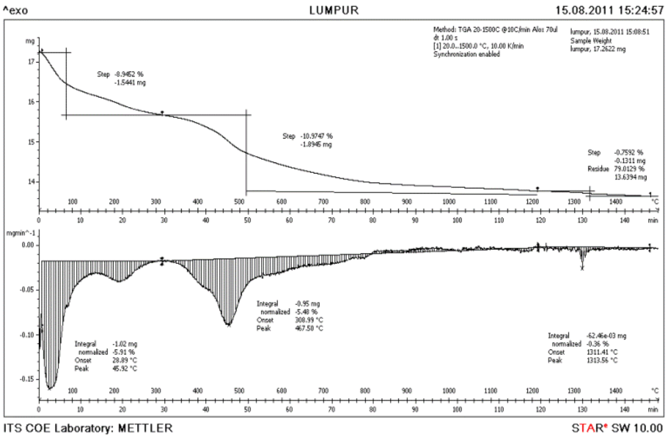

Figure 2 Thermal Gravimetric Analysis on oven dried Sidoarjo Mud

3.2 X-ray Diffraction of Heat Treated Sidoarjo Mud

X-Ray Diffraction (XRD) analysis is used to determine the form of the structure of the material is amorphous or crystalline. Increasing number of peak points on the graph shows the XRD crystalline structure while lesser peak show the more amorf form. Each reflection angle in the XRD result correspond to the crystal of the specific oxide.

Figure 3 showed the XRD analysis for Sidoarjo mud before heat treated and after heating at 500°C and 800°C. It was shown that after heat treatment of 500°C, there were reduction of the crystal peak at several compound, showing that the mud has becoming more amorf and hence more reactive. After 800°C heat treatment, there were also reduction of the number of the peak, but there was also newer peak forming.

Therefore it can be concluded that the higher the heating temperature, would cause a change in the crystalline form of a compound to be more amorphous and if the temperature were increased even more, at a certain temperature it could become crystals of an another compound.

The visual appearance of the Sidoarjo mud after heat treatment is shown in Figure 4. The color of the heat treated mud was changing from light reddish brown in to dark brown at higher temperature region. This could mean that at higher temperature there could be some part that was burnt, and turned into black crystal.

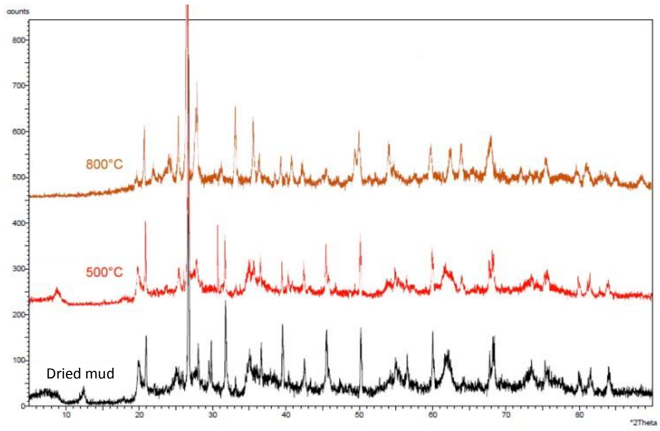

Figure 3 XRD result of the heat treated mud and non-treated

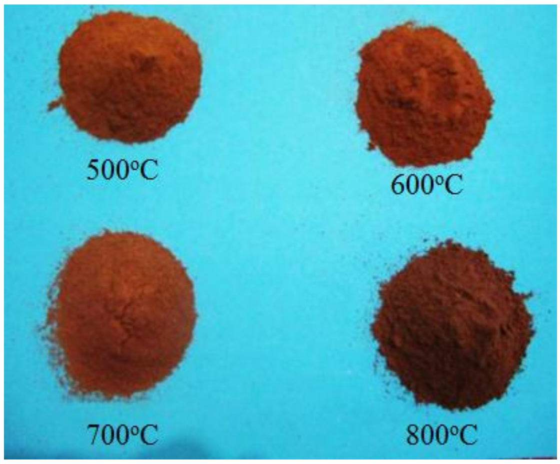

Figure 4 Different mud color from different heated temperature

3.3 Heat Treatment Temperature

Mortar specimen to investigate the heat treatment temperature influence were made by setting the variable of cement replacement ratio at 20% and particle size passing sieve #100 with particle size smaller than 150µm. Table 2 shows the results of the compressive strength test at 7, 28 and 56 days and its calculated Strength Activity Index (SAI) for each heating temperature. The compressive strength results are also plotted in Figure 5.

General trend obtained are that the cement replacement by Sidoarjo mud causes a decrease in compressive strength of mortar at all ages of testing. At 7 days age, there were some reduction of strength with increasing of heating temperature. But at later age, it was shown that there are significant increase of the compressive strength. The results showed that there are some pozzolanic activity of the heat treated specimen compared to the untreated specimen. The pozzolanic reactivity seem to be happening on all of the heat treated Sidoarjo mud regardless the heating temperature.

From the result of SAI calculation, it was shown that at 7 days age, the SAI of the heat treated mud were all lower that 75%. This could be due to slower reactivity of the heat treated mud. At 28 days and 56 days age, all of the heat treated Sidoarjo mud have SAI higher than 75%, showing it could be used as Pozzolan material.

Table 2 Compressive strength and SAI for different heating temperature

Age	**7 days**		**28 days**		**56 days**	
Temperature (°C)	**Strength (MPa)**	**SAI (%)**	**Strength (MPa)**	**SAI (%)**	**Strength (MPa)**	**SAI (%)**
no mud	26,71	100	36,88	100	38,80	100
dried mud	14,23	53,28	19,61	53,17	22,23	57,31
500	18,53	69,39	29,87	80,98	30,54	78,73
600	14,50	54,28	32,27	87,48	34,78	89,64
700	12,57	47,07	29,73	80,61	30,60	78,87
800	16,33	61,12	32,24	87,42	34,16	88,05

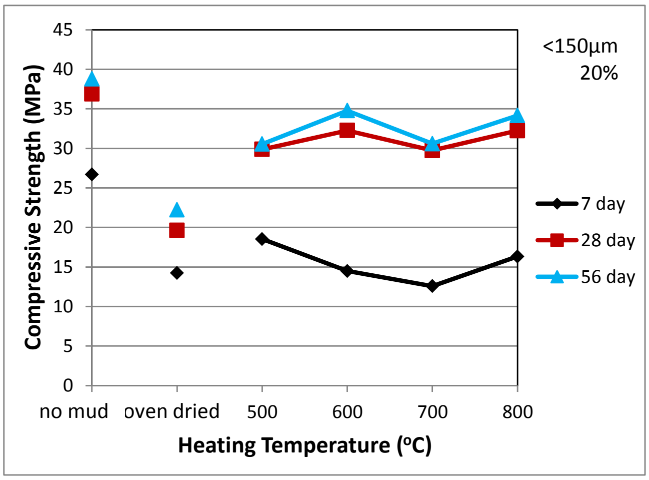

Figure 5 Compressive strength of mortar made from different temperature of heat treatment

The result of specimen using only oven dried mud also showing that it is necessary to use heat treatment on the Sidoarjo mud, if it is going to be used as pozzolanic material in concrete. Unprocessed mud would only reducing the quality of concrete and no economic value.

3.4 Particle Fineness of the Heat Treated Mud

For the particle fineness variation, the specimen were set using the previous result of the heating variable that were most effective in 7 days age which is 500°C. the mud replacement content were still fixed at 20% by weight of cement. Variation of particle fineness of the heat treated mud are 600-150µm (passing sieve #30), 150-75µm (passing sieve #100), and <75µm (passing sieve #200), that will be designated as mortar M30, mortar M100, and M200. The compressive strength results and the SAI calculation are shown in Table 3, and its compressive strength are plotted in Figure 6.

Table 3 Compressive strength and SAI for different particle size

Age	7 days		28 days		56 days	
Fineness Passing mesh	**Strength (MPa)**	**SAI (%)**	**Strength (MPa)**	**SAI (%)**	**Strength (MPa)**	**SAI (%)**
no mud	26,71	100	36,88	100	38,80	100
M30(600µm)	16,00	59,90	18,55	50,28	20,78	53,57
M100(150µm)	20,67	77,37	27,69	75,06	29,21	75,28
M200(75µm)	24,00	89,85	29,20	79,17	31,78	81,92

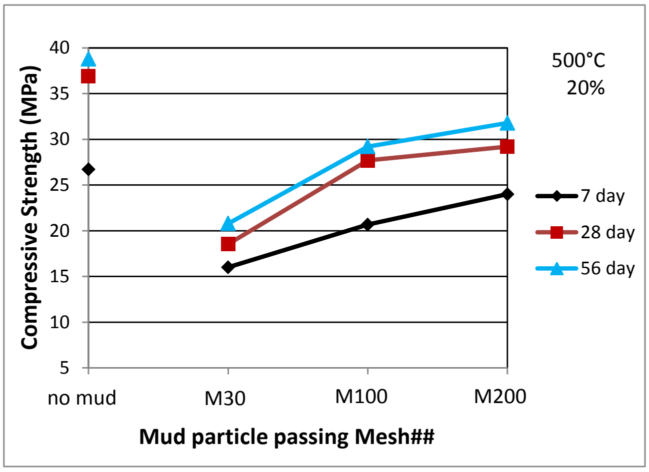

Figure 6 Results of mortar compressive strength by varying particle size of the heated mud

The results showed that the finer particle size of the mud would have higher compressive strength. This is due to the reactivity of the mud particle is increasing with the reduction of its size. Mortar M30 was shown to have the lowest strength and also lowest strength increase rate, because the mud particle were to coarse to be able to have pozzolanic reaction. The mud particle in Mortar M100 and M200 have sufficient particle fineness in order to cause pozzolanic reaction and increasing strength at age 28 and 56 days. SAI of the M200 also showed that at initial age, there were also some reactivity of the mud because of higher SAI at 7 days of 89.85%.

The finer the particle size would contribute to the increase of the compressive strength. At size smaller than 150µm, it was sufficient to cause pozzolanic activity in the mortar. For economic reason, it was not necessary to grind the particle to very fine size.

3.5 Cement Replacement Ratio

Similar to the previous variable, mortar specimen for different replacement ratio used heating temperature considered to be most effective in testing the temperature variation in the age of 7 days which is 500°C. Particle size used are passing sieve #100 but retained in #200, which is 150-75µm. Variation of the replacement of mud mortar are 10%, 20%, and 30% by weight, and its designated as mortar K10, K20, and K30. Table 4 showed results of compressive strength and SAI with different cement replacement ratio, and the compressive strength of the mortar with different cement replacement ratio are also shown in Figure 7.

The result showed that with the increase of replacement ratio there was reduction of strength, this result is common in the pozzolanic material. At 7 days strength K10 could achieve 27.05 MPa, which is slightly higher than the control specimen. Lowest compressive strength was found on K30 specimen at 24.09 MPa at 56 days.

The Strength Activity Index calculated showed that cement replacement of 10% and 20% still have SAI higher than 75%. While for 30% replacement ratio it could reach only at 62.1%. Higher replacement ratio would need higher reactivity material, which could be achieve by grinding the heat treated mud at finer particle size.

Table 4 Compressive strength and SAI for different cement replacement ratio

Age	7 days		28 days		56 days	
Cement replacement (%w)	**Strength (MPa)**	**SAI (%)**	**Strength (MPa)**	**SAI (%)**	**Strength (MPa)**	**SAI (%)**
no mud	26,71	100	36,88	100	38,80	100
K10	27,05	101,26	33,38	90,49	34,80	89,70
K20	20,67	77,37	27,69	75,06	29,21	75,28
K30	18,93	70,88	21,76	58,99	24,09	62,10

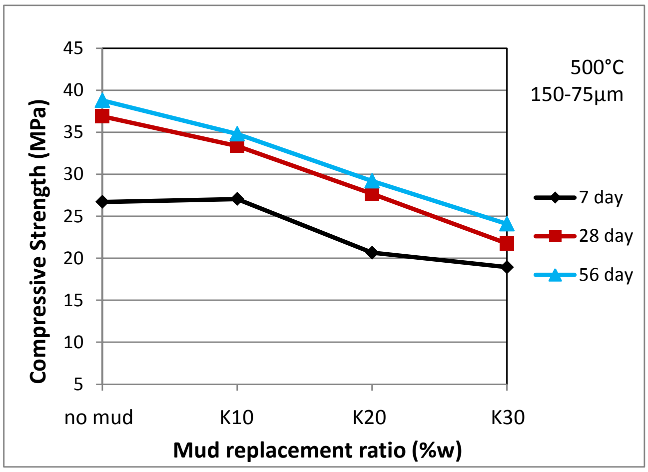

Figure 7 Compressive strength of mortar with different cement replacement ratio by heat treated Sidoarjo mud

3.6 Large Pore Porosity

Large pore porosity testing was conducted on mortar specimens of all variations. This test aims to determine the content of large pores in the mortar, by immersing mortar specimen in water for 27 days, measured the surface dried weight and then placed in the oven for 24 hours to dry the moisture content. Porosity of the large pores is measure by the weight of water evaporated in the oven. The greater the weight difference indicates that the more porous mortar. The result of the large pore porosity are shown in Figure 8, Figure 9 and Figure 10.

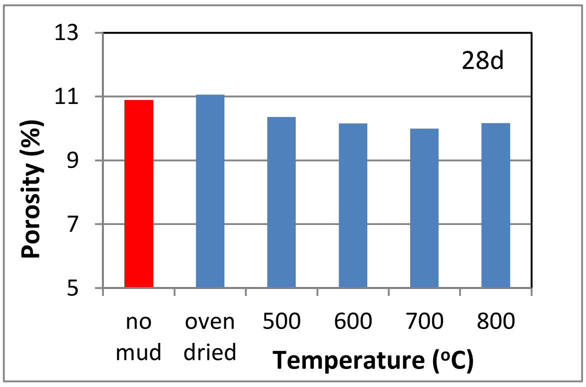

Figure 8 Large pore porosity of mortar for temperature variable

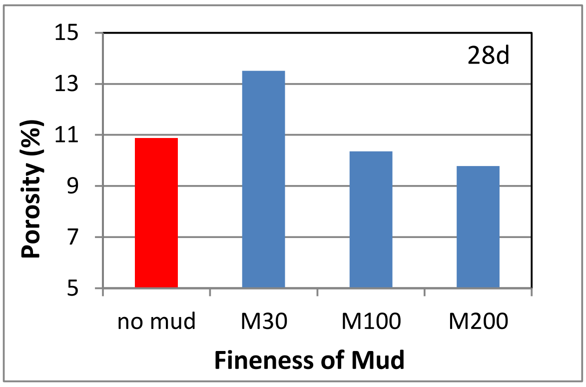

Figure 9 Large pore porosity of mortar for different particle fineness

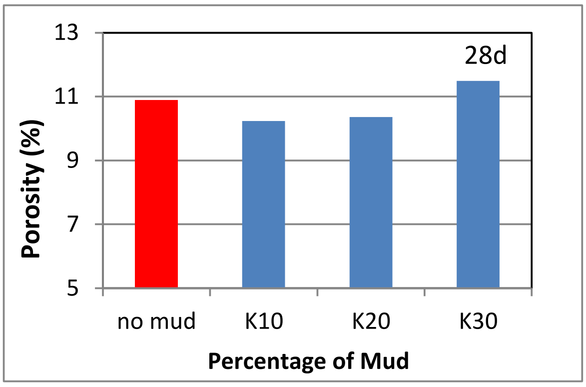

Figure 10 Large pore porosity of mortar with different cement replacement ratio

Figure 8 shows large pore porosity of mortar with variable of heat treatment temperature. the general trend obtained are the replacement of cement by mud causes a decrease in porosity. The reduction of the porosity showed that the mortar is more compact although this did not linearly correlated with its strength. For unburned mud, the porosity was larger due to more water absorption of the untreated mud, and reduction in the mortar workability.

The variation of particle size showed to be significantly influencing the large pore porosity as shown in Figure 9. Mortar with a particle fineness of 600-150µm (M30) has greater porosity than the control mortar (no mud). This is because of the particle size in M30 are quite rough and making the mortar specimen lacking finer particle. Porosity of the mortar with particle of 150-75µm and less than 75µm (showed a smaller porosity than the control mortar (no mud). From this result it was found that the smaller the particle size of mud, the porosity of the mortar would become lesser. This could be said also as microfiller effect. The mud particles fill gaps that can not be filled by cement and sand, thus the porosity ware reduced.

Figure 10 showed the porosity of specimens with different cement replacement ratio. At 10% and 20% cement replacement ratio, the porosity was lower than the control specimen while at 30%, the porosity increased higher than the control specimen. This result could be due to lower percentage of cement content and reduced hydration product.

4. CONCLUSIONS

From the experimental study conducted, the following conclusions were made:

1. Heating temperature that produces the highest compressive strength is 600°C for 2 hours, in this study. From the resulting compressive strength at temperatures of 500-800°C combustion still qualify pozzolanic Strength Activity Index (SAI) of 75%. Thus limit the combustion temperature of the mud that is 500-800°C. No extreme temperature control is required at the combustion stage meaning it could used more traditional burning method.
2. Particle fineness of Sidoarjo mud that produces the highest compressive strength is <75µm size. But the difference in compressive strength generated by the fineness of 150-75µm size also close to <75µm size. Therefore the particle size of smaller than 150µm is sufficient.
3. The greater the replacement of cement by the Sidoarjo mud produces a lower compressive strength. Mud content of 10% produces a maximum compressive strength, however from the SAI standard, 20% cement replaced by Sidoarjo mud can still be used as a pozzolanic material.
4. Sidoarjo mud that was not heat treated by high temperature heating can not be used as an pozzolanic material. Sidoarjo mud can only be utilized after heating process of 500-800°C. The heating changes the crystallinity of the particle into more amorf one hence more reactive.
5. Sidoarjo mud used from year 2006, 2009 and in this study (2011) has different oxide content. In this study, it was measured that the Sidoarjo mud contains less SiO_2 (28%), but high enough Fe_2O_3 (41.42%). This difference could be due to different layers of the earth crust that was the source of the Sidoarjo mud. Therefore in future studies, testing the oxide compounds are very important practice before the mud is used.

5. REFERENCES

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