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Sulfate Resistance of Fly Ash-Based Geopolymer Mortar

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Abstract. This paper presents sulfuric acid attack of fly ash-based geopolymer mortar. Precursor used in this study was fly ash, and activator used was NaOH and Na₂SiO₃. The ratio of activator/precursor, ratio of Na₂SiO₃/NaOH, and ratio of fine aggregate/precursor is 0.42, 2.00, and 2.00, respectively. The molar concentration of NaOH which was used were 8, 10, 12, 14, and 16 M. This study used cube specimen with 5 cm x 5 cm x 5 cm. The results showed that the higher the molar concentration of NaOH, the lower the weight loss. Maximum percentage of weight loss is 3.54% occurred for the specimen with molar concentration of NaOH 8 M. The compressive strength for all specimens decreased due to the longer duration of immersion in sulfuric acid solution. However, this percentage of decreasing for compressive strength will be as lower as increasing the molar concentration of NaOH used. The maximum percentage of decreasing is 35.49% for specimen with NaOH 8 M with 90 days of immersion.

INTRODUCTION

The Geopolymer material resulted from the process of geosynthesis between alumino-silicate and alkali silicate which formed polymer of tetrahedral skeleton of SiO₂ dan Al₂O₃. This material is also known as inorganic polymers, due to its chemical composition similar to the natural zeolite materials, but its microstructure is amorphous [1-3]. Inorganic polymer material also can be yielded from process of synthesis by mixing materials which act as precursor and activator. Material of precursor, should contain alumina and silicate in high concentration. Example of this materials, e.g fly ash, rice husk ash, blast furnace slag, metakaolin, etc. And for material as activator, generally use of the combination solution of Na₂SiO₃ or K₂SiO₃ and NaOH or KOH. The reaction process between these materials as explained above yield polymer paste which had building behaviour as same as cement paste. The material using polymer paste as a binder known as geopolymer concrete. The molar concentration of NaOH will effect the mechanical properties of geopolymer mortar. As higher as the molar concentration of NaOH used, as higher as the compressive strength of the geopolymer mortar. As higher as the ratio of Na₂SiO₃/NaOH, also will increased the value of compressive strength [4-6]. Other factors which will effect the compressive strength e.g method and temperature of curing, curing time, water/precursor ratio, and fine aggregates/precursor ratio [7-9]. This paper will summarize the geopolymer mortar durability due to the usage of fly ash, the variation of molar concentration of NaOH, the variation of Na₂SiO₃/NaOH ratio, and variation of activator/precursor ratio.

MATERIAL AND EXPERIMENTAL STUDY

The material used in this research were e.g fine aggregate from Tanjung Raja, fly ash from waste of PLTU Tanjung Enim, sodium hydroxide (NaOH) which as flake form with 98% purity, and natrium silicate (Na₂SiO₃) which as gel form with 58% purity, the water used was aquades, and Na₂SO₄ solution with concentration of 10%. The results of X-Ray Fluorence (XRF) testing fly ash is shown in Table 1. According to ASTM C 618-03, minimum oxides of SiO₂ + Al₂O₃ + Fe₂O₃ is 50% for type C and 70% for type F and N. Then fly ash used in this research was categorized as type F where the oxides of SiO₂ + Al₂O₃ + Fe₂O₃ was 88.820%, and the value of LoI was 2.610% and the maximum LoI value is 6%.

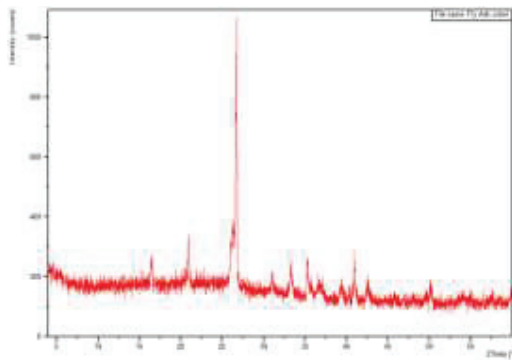
Figure 1a showed the result of X-Ray diffraction (XRD) test of fly ash. From this figure can be explained that the fly ash used was more dominant of amorf structure than crystalline structure, thus the fly ash is more reactive.

The photo of scanning electron microscope (SEM) test for fly ash was shown in Figure 1b. The photo showed that fly ash particles had spherical form. The spherical form enabled the geopolymer mortar more workable.

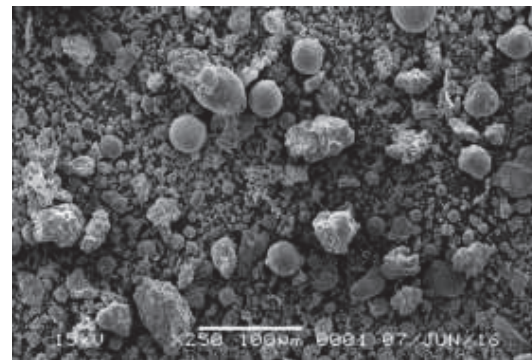
In this experiment, various of molar concentration of NaOH were used e.g 8, 10, 12, 14, and 16 M. Whereas the ratio used for NaOH and Na₂SiO₃, for Na₂SiO₃ and precursor, and for fine aggregate and precursor were 2.00, 0.42, and 2.00 respectively. Then, the fresh geopolymer mortar were casted using 50 mm x 50 mm x 50 mm cube mold, and kept in rest period for two days. After demoulding, specimens were cured by heat curing at 90°C in a oven for 24 hours. The last step wrapped all the specimens with plastic sheet until 28 days. The conduct the durability test, the specimen were soaked in the solution of natrium sulfate (Na₂SO₄) with the molar concentration of 10% for period of 7, 28, 56, and 90 days.

TABLE 1. The chemical composition of fly ash

Oxides	Quantity (%)	Oxides	Quantity (%)
SiO ₂	53.49	PbO	0.004
Al ₂ O ₃	29.35	ZrO ₂	0.053
Fe ₂ O ₃	5.98	CuO	0.005
CaO	3.33	P ₂ O ₅	0.424
Na ₂ O	0.68	BaO	0.047
K ₂ O	0.51	V ₂ O ₅	0.021
MgO	1.35	Cr ₂ O ₃	0.004
SO ₃	0.51	ZnO	0.015
LOI	2.61		



(a)



(b)

FIGURE 1. Characteristic of fly ash (a) The result of XRD test of fly ash (b) The SEM of fly ash

RESULTS AND DISCUSSION

Slump Flow

The slump flow test was conducted according to ASTM C124. The result of the test was shown by the graph on Figure 2. From this graph, can be explained that as higher as the molar concentration of NaOH used, as lower as the value of slump flow. The value of slump flow for NaOH 8 M, 10 M, 12 M, 14 M, and 16 M were 16.45 cm, 15.15 cm, 15.25 cm, 13.60 cm, and 11.20 cm, respectively.

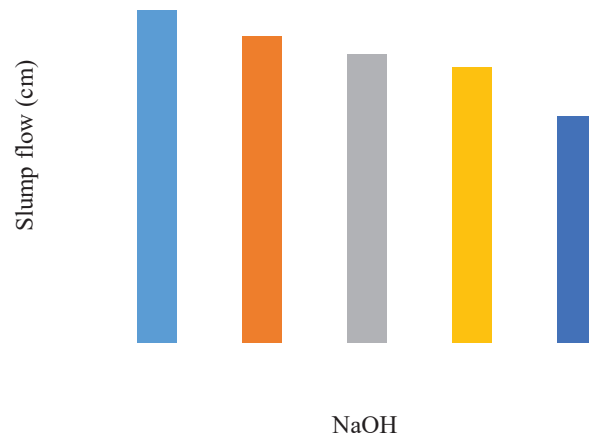


FIGURE 2. The results of slump flow

Setting Time

Setting time test was done according to ASTM C191. The Figure 3 showed the result both of initial and final setting time of the mixture. It can be explained that as higher as the molar concentration of NaOH, as faster as the initial and final setting time. This is due to the faster of polymerization process between NaOH solution with fly ash.

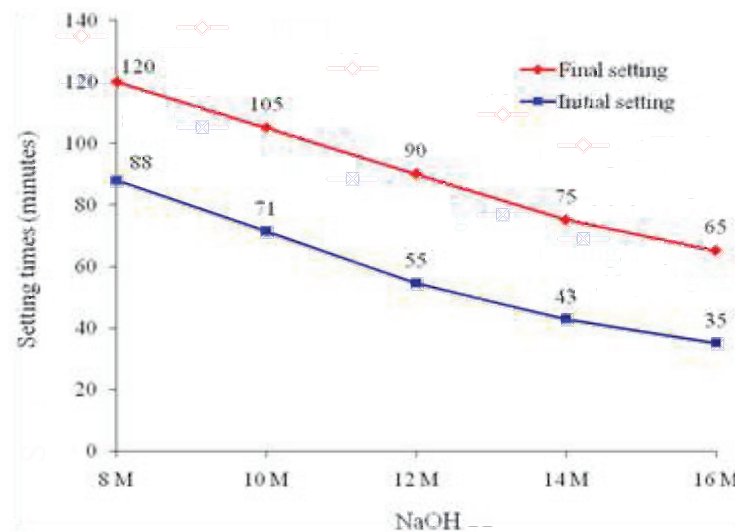


FIGURE 3. The results of slump flow

Compressive Strength

The compressive strength of geopolymer concrete for various of molar concentration of NaOH for 28 days without expose to sulfuric acid solution was shown in Figure 4. From this study, the highest compressive strength was given by specimen with NaOH 16 M e.g 55.60 MPa.

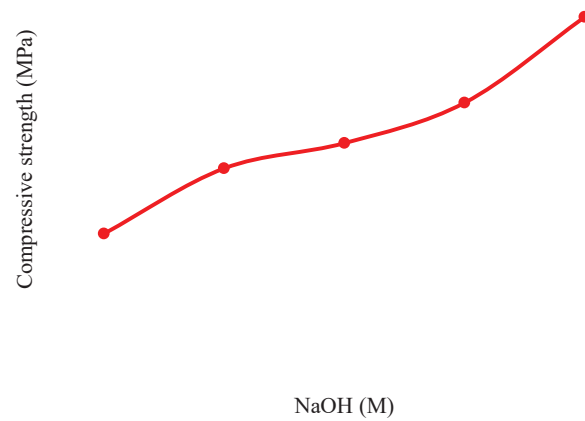


FIGURE 4. Result of compressive strength test

Weight Loss

For weight loss test specimens were immersed in sulfuric acid solution for 7, 28, 56, and 90 days. The percentage of specimens weight loss for various of molar concentration of NaOH for 7, 28, 56, and 90 days of immersion in sulfuric acid solution can be seen in Table 2. Figure 5 showed the percentage weight loss vs time of immersion. From Table 2 can be seen that the percentage of weight loss of each specimen increased simultaneously with time of immersion in sulfuric acid solution. Yet, as higher as the molar concentration of NaOH used in the mixture, as lower as the percentage of weight loss. Then, from Figure 6, can be explained that the rate of decreasing from weight loss much steeper simultaneously with decreasing of NaOH molarity used in the geopolymer mortar.

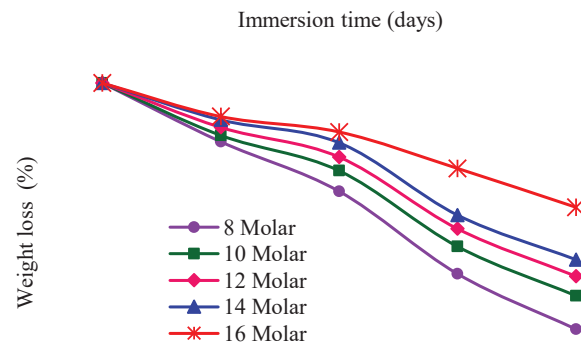


FIGURE 5. Weight loss

TABLE 2. Percentage of weight loss

NaOH concentration (M)	Weight loss (%)			
	Time of immersion (days)			
	7	28	56	90
8	0.84	1.56	2.74	3.54
10	0.75	1.26	2.53	3.06
12	0.64	1.07	2.10	2.70
14	0.55	0.86	1.90	2.54
16	0.48	0.71	1.23	1.79

Residual Compressive Strength

The result test of compressive strength for each specimens after soaking in the sulfuric acid solution for 7, 28, 56, and 90 days and each percentage of decreasing calculating from 28 days compressive strength was shown in Table 3. From the data in Table 3, it was shown that the compressive strength of each specimen decreased simultaneously with the time of soaking in sulfuric acid solution. Yet, as higher as percentage of decreasing of compressive strength for various days of soaking. The degradation on the compressive strength could be due to the depolymerization of alumina silicate and alkali silicate polymer in sulfuric acid solution and formation of ettringite and gypsum. The behaviour of high water absorption from ettringite and gypsum to increase internal stress to form microcracks. And then degrade the compressive strength of geopolymer mortar. Figure 6a and 6b showed the compressive strength curve and the percentage of decreasing from compressive strength, respectively, for various of soaking time in sulfuric acid solution. From Figure 6a can be seen that all the curves experiment similar sloping of compressive strength decreasing curves, as showed in Figure 6b, being steeper for lower NaOH molarity used in the mixture.

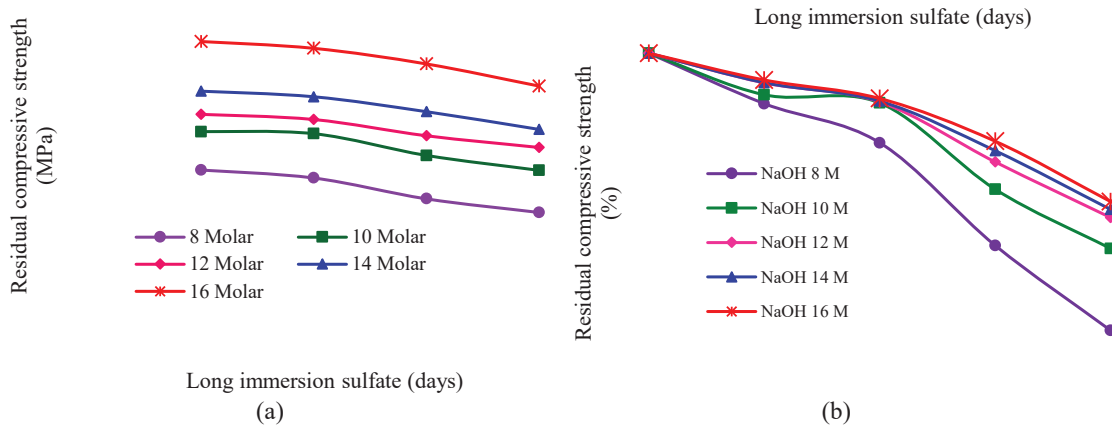


FIGURE 6. Compressive strength after immerse in sulfate

TABLE 3. Compressive strength after 7, 28, 56, and 90 days soaking in sulfuric acid solution

NaOH (M)	Compressive strength									
	Before soaking		After soaking							
			7 day		28 day		56 day		90 day	
	28 days		MPa	(%)	MPa	(%)	MPa	(%)	MPa	(%)
8	30.7		28.7	6.45	27.2	11.46	23.2	24.61	20.5	35.49
10	38.2		36.2	5.31	35.8	6.36	31.6	17.40	28.7	24.99
12	41.1		39.6	3.79	38.6	6.23	35.4	13.91	33.1	21.03
14	45.8		44.0	3.76	43.0	6.12	40.1	12.46	36.6	19.97
16	55.6		53.7	3.42	52.4	5.79	49.3	11.26	45.0	18.99

Microstructures

Photo of SEM test were shown in Figure 7 and 8 for geopolymer mortar of various NaOH molarity used both for without soaking and 90 days of soaking in sulfuric acid solution, respectively. From Figure 7 for geopolymer mortar without soaking in sulfuric acid solution, as higher as the molarity of NaOH used, as denser as the microstructure. The denser microstructure could yielded higher compressive strength as already discussed above. From visualization of Figure 8 can be shown the effect of soaking in sulfuric acid solution for 90 days on geopolymer mortar as higher as the molarity of NaOH used, as denser as the microstructure. Thus yielded higher compressive strength as discussed above. From the microstructure point of view, sulfuric acid solution would

increased the porosity of microstructure. And also caused some microcracks due to the higher water absorption behavior as discussed above.

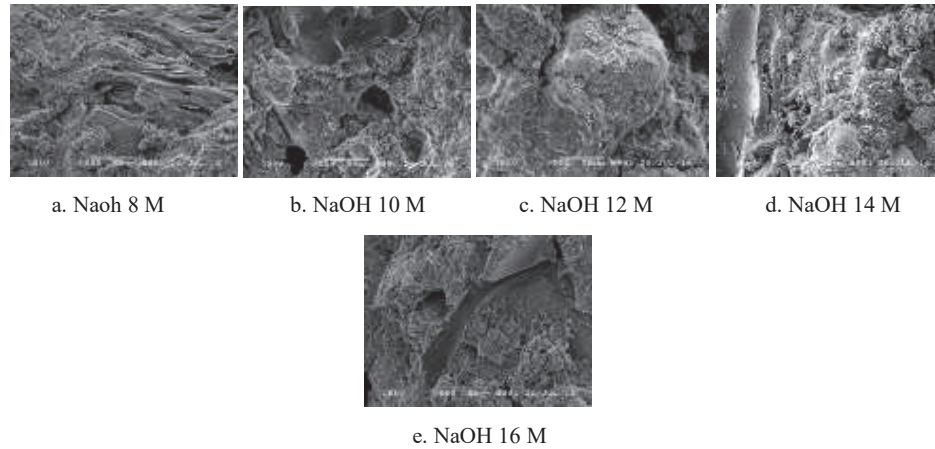


FIGURE 7. Microstructures for geopolymer mortar without soaking in sulfate

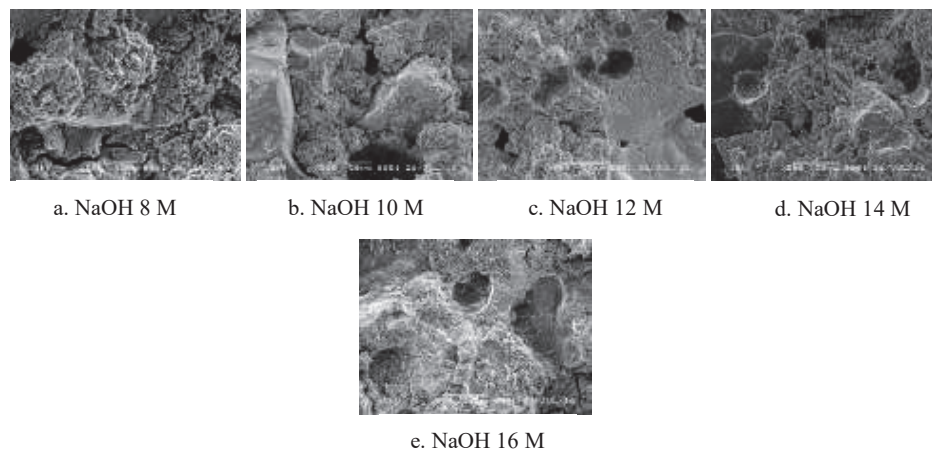


FIGURE 8. Microstructures for geopolymer mortar after 90 days soaking in sulfate

CONCLUSIONS

Based on this study, can be concluded as follow:

1. The maximum compressive strength is 55.60 MPa at 28 days was given by geopolymer mortar with molarity concentration of NaOH 16 M.
2. Mass gradation of geopolymer mortar would increased simultaneously along with the time of soaking in sulfuric acid solution. The higher the molarity of NaOH used, the lesser the mass degradation.
3. The higher the molarity of NaOH used for geopolymer mortar, the lesser the effect of sulfuric acid solution on its microstructure.
4. Sulfuric acid solution would increased the porosity of geopolymer mortar.

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