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Effect of Adding Acid Solution on

Setting Time and Compressive Strength of High Calcium **Fly Ash Based Geopolymer** 21

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anas.evelin

@gmail.com d **)djwantoro.h@petra.ac.id** Abstract. **Fly ash** 7

with high calcium oxide content when used as the base material in geopolymer concrete could cause flash setting or rapid hardening. However, it might increase the compressive strength of geopolymer concrete. This rapid hardening could cause problems if the geopolymer concrete is used on a large scale casting that requires a long setting time.

CaO content can **be** indicated **by pH** values **of the fly ash,** 1

while higher pH is correlated with the rapid

setting time of fly ash-based geopolymer. This study investigates **the** addition **of** 26

acid solution to reduce

the initial pH of the fly ash 13

and to prolong the setting time of **the** mixture. **The** 5

acids used in this study are

hydrochloric acid (HCl), **sulfuric acid** (H_2SO_4), **nitric acid** (HNO_3) **and acetic acid** 3

(CH_3COOH).

It was found that the addition of acid solution in **fly ash was** 6

able to decrease the initial pH of

fly ash, however, the initial setting time of 32

geopolymer was not reduced. It was even faster than that of the control mixture. The acid type causes various influence, depending on the fly ash properties. In addition, the use of acid solution in

fly ash reduces **the compressive strength of geopolymer** mortar. It **is** concluded that **the addition of** 2

acid solution cannot prolong the rapid hardening

of high calcium fly ash geopolymer, and 40

it causes

adverse effect on the compressive strength. INTRODUCTION **The** 41

effort to replace cement as construction material had been done by introducing fly ash as the binder with geopolymer reaction.

The use of fly ash as a base **material** 5

is very beneficial, because

fly ash is by-product of the combustion 35

products and the resulting CO_2 emissions are 80% lower than Portland cement [1]. The geopolymer reaction commences when the low calcium or high calcium fly ash, a material rich in silica element [2-6], mixed

with an alkaline solution of sodium hydroxide (NaOH) **and sodium silicate.** 20

There was some difficulty when

using high calcium fly ash in **the** geopolymer reaction because **of** the **flash setting** 2

or very rapid hardening occurrence on the mixture. Flash setting is a condition where the process of hardening or setting time occurs very rapidly, only a few minutes after the addition of alkaline solution. This poses a big problem when the use of geopolymer concrete on a large scale and long time setting is necessary [7]. Researchers previously said if fly ash has high levels of calcium and high concentration of NaOH solution used could cause flash setting [8-10]. The occurrence flash setting is suspected due

to the higher content of calcium **(CaO) in the fly ash.** 22

In general, fly ash is an alkaline material,

high calcium fly ash has a 44

pH about 10-12, while low calcium type had pH about 8-9. Davidovits [11] indicates fly ash with high pH levels higher than 11 would have a high probability of rapid hardening. The authors' previous studies have also found that higher pH fly ash value which correlated to the higher calcium content in fly ash that leads to flash setting of the mixture [12]. Attempts to overcome the rapid setting of geopolymer had been done using superplasticizer [13,14], and sucrose or sugar [15]. The use of superplasticizer and sucrose can increase the time setting time, however, the additives decrease the final compressive strength of the mixture. Subsequent research found that the addition of borax can lengthen the setting time to some extent but still not enough for mass casting [16]. The effort to

prolong the setting time of the geopolymer concrete **using high calcium fly ash** still continues. **The** use **of** 2

hydrochloric acid compounds for fly ash treatment can reduce the calcium level in fly ash was shown by Lieberman et al. [17]. This then raises the possibility that the use of acid to prevent flash setting as well as increase the

setting time of high calcium fly ash by lowering **the** 33

pH level. Tinnea & Young [18] also found that the addition of citrate acid can slow down the hydration process preventing excessive hardening of conventional concrete. The acid is potentially used as a retarder to slow the hydration process on cement. In subsequent research, the use of citric acid as a hydration process retarder on Portland cement also developed [19]. Other researchers also confirmed that the citric acid could slow the setting time on conventional concrete at very early age [20]. Thus, the addition of various acid solutions to fly ash can cause a decrease in pH value and reduce the free CaO levels in fly ash so

that the setting time can be extended. This research evaluates **the** effect **of** 23

several acid solutions addition in the high calcium

fly ash to its **setting time and compressive strength** properties **of** the **geopolymer** 4

mortar. The acids used in this study are

hydrochloric acid (HCl), **sulfuric acid** (H_2SO_4), **nitric acid** (HNO_3) **and acetic acid** 3

(CH_3COOH). EXPERIMENTAL METHODS Materials Fly ash used is high

calcium fly ash obtained from Paiton **Power Plant,** Probolinggo, **East Java.** 2

This study used two fly ash samples (F1 & F2) taken at different times. The sand used is from Lumajang quarry, East Java, and the gradation is controlled to conform to ASTM C778 [21]. The acids used in the study are 10.5 M hydrochloric acid (HCl), 16.9 M sulfuric acid (H_2SO_4), 16

.3 M nitric acid (HNO_3) and 17.2 **M** acetic **acid** 39

(CH_3COOH). The alkali solution

used in this study was sodium hydroxide (NaOH) and sodium silicate Grade 52. **The** 15

XRF result and the sieve analysis results are shown

in Table 1. From the XRF **test results can be** seen **that the** 17

fly ash is categorized as type C based on ASTM C311 [22], with the content of SiO_2 + Al_2O_3 + Fe_2O_3 more than 50% and CaO levels above 10%.

TABLE 1. Chemical and physical properties of the **fly ash.** 24

No. Parameter Unit F1 F2 1 SiO_2 % wt 2 Al_2O_3 % wt 3 Fe_2O_3 % wt 4 CaO % wt 5 MgO % wt 6 Na_2O % wt 7 SO_3 % wt 8 K_2O % wt 9 TiO_2 % wt 10 P_2O_5 % wt 11 MnO_2 % wt 12 L O I % wt 13 %Passing sieve #325 % wt 35.46 16.91 15.43 16.98 7.23 2.83 1.72 1.32 0.75 0.26 0.18 0.4 88 Mixture and Testing 34.29 16.62 15.38 18.18 7.52 2.97 1.63 1.35 0.73 0.25 0.17 0.36 92 The composition of mix design for making the geopolymer mortar mixture are as follows; sand

to fly ash ratio of 2, free **water to fly ash ratio** 25

of 0.25, the NaOH molarity is calculated to be 8 M for the given free water mass and

ratio of sodium silicate solution to sodium hydroxide solid **was 2.5. All ratio** 14

is calculated in weight. Curing of the geopolymer mortar specimens are done oven at 60C

for 24 hours and then rest **at room temperature until testing** 18

time. The acid solution

was added at 1. 67% **of** the **fly ash weight.** 36

The addition dosage was adjusted from Kusbiantoro et al [15]. The same amount of acid solution was added to differentiate its influence on the geopolymer properties with different concentration. The measurement of pH was done in two methods, ASTM D 5239 [23] and using Davidovits [11]. According to the ASTM method, pH testing is done by mixing 20 grams of fly ash into 80 grams of distilled water, stirring until evenly and allowed to stand approximately 15 minutes until the fly ash settles, while according to Davidovits, the pH testing is done by mixing 10 grams of fly ash into 100 grams of distilled water, stirred and allowed to settle. The test was done with digital pH meter with an accuracy of 0.1 decimal with the condition that digital pH meter should only be exposed to water and should not be exposed to

fly ash deposit. **The initial setting time** 45

was conducted on geopolymer paste, excluding the sand in the mixture, using Vicat needle test based on ASTM C 191 [24]. The

compressive strength test was **conducted at 7, 14 and 28 days** on **the** 16

mortar specimens. Three replication of the

compressive strength test was conducted for 46

each age. RESULTS AND DISCUSSIONS Changes of pH with Addition of Acid Solution Table 2 shows that the addition of the acid can cause the decrease of

pH of the fly ash and distilled **water** solution. **Both** F1 **and** 11

F2 fly ash have pH higher than 11, measured by two methods. F2 have higher pH that F1 shown by measurement from ASTM method, which correlates with the CaO content from XRF measurement. After the addition of 1.67% of acid solution into the fly ash powder, there was a reduction of pH at a different point depending on the acid type. Davidovits [11] mentioned

that the higher **pH** value **of fly ash** 47

could be an indication about the CaO content, causing the flash setting to occur. The authors suspect that the CaO content of fly ash could be decreasing due to the acidic solution binds to the free CaO in fly ash, shown by the reduced pH. However, the uniformity of fly ash and acid solution is still in doubt due to the low volume of acid solution. TABLE 2. pH of the fly ash before and after acid solution treatment. Fly ash Acid solution Solution (%w FA) ASTM 5239 (20/80) Davidovits method (10/100) F1 Control - 11.3 11.2 Control Hydrochloric acid (HCl) F2 Sulfuric acid (H_2SO_4) Nitric acid (HNO_3) Acetic acid (CH_3COOH) - 11.6 11.2 9.4 10.2 1.67 7.3 8.0 9.2 9.9 9.3 9.4

Setting Time of the Geopolymer Paste The initial setting time 9

is tested using Vicat needle on the geopolymer paste. Figure 1 shows that there is a significant difference in

initial setting time for F1 **and** F2. **The** initial **setting time of the** 10

F2 was about 8 minutes faster than the F1, could be because of the

CaO content in F2 **fly ash is** 2% higher **than the** 31

F1 fly ash. The pH of the F2 is also higher than the F1, as shown in Table 2.

With the addition of acid solution **in the** dry **fly ash, the** 27

initial setting time was considerably faster for F1, while the F2 only shown a relative small change. This could be because of the already short initial setting time of the F2. The similar effect of rapid hardening is also shown by Kusbiantoro et al [15] when citric acid was used. He suspected the reaction is due to the formation of C_3A which can cause quick hardening. In conventional concrete, C_3A or tricalcium aluminate reactions result in an acceleration of hydration resulting calcium hydroaluminate ($3CaO\text{-}Al_2O_3\text{-}Ca(OH)_2\text{-}nH_2O$ or hydroxy-AFm), which makes concrete paste harden faster [25]. The addition of acid into the dry F1 fly ash accelerate its initial setting time about 40-60% when compared to the control. While for F2 samples, the addition of acid accelerates about 8% compared to the control specimen. The addition of several type of acids namely

hydrochloric acid, sulfuric acid, nitric acid, and acetic acid 3

at 1.67% of fly ash weight, did not produce a longer initial setting time of the geopolymer. This result is similar to the previous studies with the addition of citric acid to the geopolymer paste [15]. And the acceleration of setting time was not aligned with the previous studies on the use of acid in the normal concrete, where the citric acid could act as a hydration process retarder on cement [19] and conventional concrete [18]. Initial setting time (Mins) 18 16 14 12 10 8 6 4 2 Initial setting time (Mins) 18 16 14 12 10 8 6 4 2 0 0 F1 F1HCl $F1H_2SO_4$ $F1HNO_3$ $F1CH_3$ COOH (a) F2 F2HCl $F2H_2SO_4$ $F2HNO_3$ $F2CH_3$ COOH (b) FIGURE 1.

Initial setting time of fly ash reacted **with** 1

the different acid solution,

for (a) F1 **fly ash and (b)** F2 **fly ash.** 1

Compressive Strength of Geopolymer Mortar **The** increase **in compressive strength** 12

with age is

shown in Fig. 2, the compressive strength **of** the F2 **fly ash is** higher than **the** 19

F1 fly ash also correlated with the slightly higher CaO

content in the F2 **fly ash. For geopolymer** 42

mortar with various acid added initially in the fly ash, the addition of 1.67% of acid solution into the F1 fly ash decreases the result of the compressive strength compared to the control geopolymer mortar. Hydrochloric acid had the highest reduction of strength at 7 days testing compared to other acids, the reduction was about 40% compared with the control mortar. There was an increase of the strength at the later age, showing that reduction could be reversed. This

result is similar to research on **the use of** citric acid **in** 6

conventional concrete that causes the reduction of

compressive strength of concrete **at** very **early age** 6

[20]. For F2 fly ash, the highest reduction of strength happened on the specimens with acetic acid addition. The specimen did not increase in strength with age on contrary with the specimens with hydrochloric acid. Specimen with the addition of hydrochloric acid and nitric acid still show some

increase in the compressive **strength up to 28 days,** 37

showing that there could be other reaction occurring at the later age beside the initial geopolymer reaction. This

reduction in compressive strength remains **with the** 8

geopolymer mortars at 14 days and 28 days for sulfuric and acetic acid when compared to the control mixture for both fly ashes. It is shown then the addition of acid solution on the initial paste, without other treatment, resulting in detrimental condition to the physical properties of the

geopolymer mortar. Compressive strength (MPa) 60 7 days **Compressive strength (MPa)** 11

60

7 days 50 14 days 50 14 days 38

40 28 days 40 28 days 30 30 20 20 10 10 0 0 F1 F1HCl F1H_2SO_4 F1HNO_3 F1CH_3 F2 F2HCl F2H_2SO_4 F2HNO_3 F2CH_3 COOH COOH (a) (b) FIGURE 2. The compressive strength

of geopolymer mortar for (a) F1 **fly ash and (b)** F2 **fly ash.** 1

Effect of Initial pH

of Fly Ash on **Setting Time and Compressive Strength** 4

In previous study [26, 27], it was believed that pH could influence the

initial setting time and the **compressive strength of geopolymer paste.** 4

Higher pH values lead to very fast setting times because of the high

CaO content in fly ash. Figure 3 shows **the** results **of** 5

initial pH testing of fly ash after the various acid addition measured by Davidovits' method to the initial setting time and the

compressive strength. Based on the **results, it is** shown **that** 30

the addition of sulfuric acid reduced the initial pH greater than other acid solutions. However, the initial setting time that occurs in the paste is much faster than the addition of other acids. The initial setting time was faster for lower pH when the pH is changed using acid solution. The

results also show **that** at **the compressive strength** value was lowered **when the** 12

acid solution was added to the mixture with a minor influence from the pH. The type of acid have more influence on the reduction of the strength, hence adding the acid solution in fly ash would reduce the compressive strength potential of the mixture. Initial setting time (Mins) 10 28d Comp. Strength (MPa) 60 9.8 9.6 9.4 9.2 9 8.8 8.6 8.4 8.2 8 HNO_3 CH_3COOH H_2SO_4 HCl F2 50 40 30 20 10 0 H_2SO_4 HCl F2 HNO_3 CH_3COOH 7.0 8.0 9.0 10.0 11.0 12.0 7.0 8.0 9.0 10.0 11.0 12.0 Initial pH of fly ash (a) Initial pH of fly ash (b)

FIGURE 3. Correlation **of initial** pH **of** the **fly ash with (a)** 1

initial setting time and (b) **28 days' compressive strength.** 34

CONCLUSIONS

Based on the results of analysis and discussion **in this study, the** 28

conclusions can be drawn as follows: 1. The addition **of** 29

acid solution to fly ash decreases its initial pH so that it is probable that the free CaO bound to the acid solution. The pH reduced at a different rate depending on the acid type and its molarity. 2. The

decrease in initial pH of fly ash did not result **in** 13

an increase of the initial setting time but the opposite occurred. This showed that there was no connection between initial pH and initial setting time when an acid addition is used. The addition of acid solution caused acceleration of

the initial setting time of the geopolymer paste 9

consistently showed by sulfuric acid (H_2SO_4) mixture. Initial setting time occurs quickly by various degree depending on the acid solution and fly ash material. 3. The addition of the acid solution causes the

compressive strength of the geopolymer to 8

be lower when

compared to the control. The addition of 8

acetic acid (CH_3COOH) produced the lowest compressive strength compared with other mixture. Moreover, the decrease of compressive strength did not change with time, in contrast to the mortar geopolymer added with hydrochloric acid (HCl) and nitric acid (HNO_3), which have slight increase in strength as the age increases. 4. There is a correlation of initial pH with the

setting time of the geopolymer concrete **with** the initial pH **of fly ash,** 10

with lower pH giving the faster setting time. The compressive strength also

influenced by the acid **addition** however **the effect** 43

is more correlated to the acid type than the initial pH. REFERENCES 1. P. Duxson, J. L. Provis, G. C. Lukey, and J. S. J. van Deventer, "The Role of Inorganic Polymer Technology in the Development of 'Green Concrete,'" Cem. Concr. Res., vol. 37, no. 12, pp. 1590–1597, 2007. 2. H. Yildirim, M. Sümer, V. Akyüncü, and E. Gürbüz, "Comparison on efficiency factors of F and C types of fly ashes," Constr. Build. Mater., vol. 25, no. 6, pp. 2939–2947, 2011. 3. D. Hardjito, S. E. Wallah, D. M. J. Sumajouw, and B. V Rangan, "Factors Influencing the Compressive Strength of Fly Ash-Based Geopolymer Concrete," Civ. Eng. Dimens., vol. 6, no. 2, pp. 88–93, 2004. 4. A. M. Mustafa Al Bakri, H. Kamarudin, M. Bin Hussain, I. Khairul Nizar, Y. Zarina, and A. R. Rafiza, "The Effect of Curing Temperature on Physical and Chemical Properties of Geopolymers," Phys. Procedia, vol. 22, pp. 286–291, 2011. 5. N. Lloyd and B. V. Rangan, "Geopolymer Concrete with Fly Ash," Second Int. Conf. Sustain. Constr. Mater. Technol., vol. 3, pp. 1493–1504, 2010. 6. D. Hardjito and Fung, S.S., "Fly Ash-based Geopolymer Mortar Incorporating Bottom Ash," Modern Applied Science, vol. 4, no. 1, pp. 44-52, 2010. 7. Antoni, S.W. Wijaya, and D. Hardjito. "Factors Affecting the Setting Time of Fly Ash-Based Geopolymer" Materials Science Forum, vol. 841, 2016. 8. M. T. Junaid, O. Kayali, A. Khennane, and J. Black, "A Mix Design Procedure for Low Calcium Alkali Activated Fly Ash-Based Concretes," Constr.

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