A STUDY ON FACTORS AFFECTING GEOPOLYMERIZATION OF LOW CALCIUM FLY ASH

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ABSTRACT: This paper presents some factors affecting geopolymerization of low calcium fly ash for geopolymeric matrices. Low calcium fly ash samples were collected from two different coal-powered facilities: an Indonesian fertilizer plant and a Japanese power plant. Several series of tests were conducted using various ratios of fly ash to activator as well as ratios of activators to sodium hydroxide molarity. Each matrix consisted of a set molar ratio of three variations of Si/Al (1.5, 2, 2.5), Na₂O/SiO₂ (0.3-0.38), H₂O/SiO₂ (2.8 to 3.5), H₂O/Na₂O (9 to 10.6), and mass ratio of water/solid (0.31 to 0.45). The setting time of Japanese ash-matrices were longer than Indonesian ash. The compressive strength revealed that the Japanese and Indonesian matrices with activator ratios of 1.5 achieved 47.7 and 57.5 MPa respectively, while activator ratios of 2.5 reached 50.9 and 50.5 MPa. In addition, microstructural characterizations–XRF, XRD, SEM, EPMA-were performed. This study concludes that even ashes categorized as the same class, their mineral composition is different. Furthermore, coal combustion techniques modify ash particles, which in turn causes differences in setting time, while strength is not significantly affected.

Keywords: Fly ash, Geopolymer, Alkali activation, Characterization, Microstructure

1. INTRODUCTION

Geopolymers-based material is a sustainable and ecofriendly building material obtained from waste containing plenty of silica and aluminum; one such material is fly ash. Compared to ordinary Portland cement, geopolymers provide better properties [1,2]. Fly ash is a waste generated by coal combustion which has temperatures differ by technique, typically the following four: fluidizedbed, pulverized coal combustion 1, pulverized coal combustion 2 and coal gasification IGCC; respective operating temperatures are 850°, 1250°, 1500° and 1800° C [1]. Power generation facilities operating in Japan display higher thermal efficiency due to improved steam conditions. In contrast, Indonesia still uses power plants with lower operating temperatures [2].

Physical and chemical composition are factors which affect properties of geopolymers, in this case ash. Low CaO content in ash lengthens setting time [5,6] states that low-calcium ash-based geopolymers ordinarily have a setting time of nearby 2 hours. Previous study [3] revealed factors affecting setting time of matrices are morphology, reactivity and Ca content of ash. The micro structures of ash consist typically of amorphous (glass) rather than crystalline elements; this presents difficulty, and thus several parameters which favor and hinder geopolymerization require analyses. Disruption was discovered to be due to crystalline minerals [1]. Crystalline quartz amounts depend on cooling rates [4], while iron and quartz mineral proportions are influenced by varying coal mineral quantities. Glass fraction composition, indicated by a hump in XRD patterns [5], has also been found to vary by ash property [6]. The study regarding microstructural observations using SEM is in good agreement with phase transformations investigated by XRD [7]. Observation of ash can be performed using SEM [8], although, SEM is limited to surface morphology [9] and composition [10]. The morphology of a particle of ash is dependent on coal mineral composition, postcombustion cooling and combustion conditions [11].

Just as ash properties affect geopolymer properties [16,17], wide variations in mineral proportions of coal affect ash characterization [10,14]. Thus, several micro-analyses have been conducted to investigate ash [5,12,18]: scanning electron microscopy (SEM), X-ray Diffraction (XRD), and X-ray Fluorescence (XRF). Several studies have investigated ash reactivity [5,19], performed according to ASTM C 311 [12] to determine ash activity [13]. The aims of this research were to analyze the factors affecting two mechanical properties - compressive strength and setting time - of low calcium-fly ash-based geopolymeric matrices and to perform micro analyses of ash.

The basic roles of alkali in geopolymerization are to generate pH adequate to activate aluminosilicate materials and to charge-balance the development of aluminosilicate gel framework [14]. The mechanism is described as: (1) dissolution of raw materials in alkaline solution, (2) reorientation of the dissolved species, and (3) polycondensation to form networked gel structures [23,24].

Geopolymers have various applications in industries determined primarily by the chemical structure in terms of Si/Al atomic ratio. A low ratio of Si/Al of 1, 2 or 3 high rigidity, appropriate for cement and concrete applied to civil engineering fields [1].

2. EXPERIMENTAL CONFIGURATION

Two different of ash types, one from the Reihoku Power Plant in Japan, the other from a fertilizer plant in Indonesia and denoted JFA and IFA, were synthesized for matrix. The color of JFA was light gray, while IFA was saddle brown. Both ash types were classified as type II according to the Japanese Industrial Standard (JIS) [15] or class F based on the American standard (ASTM) [16].

For synthesized matrices, the ash was mixed with alkaline activator consisted of sodium hydroxide (NaOH) solution and sodium silicate gel (Na₂SiO₃). The NaOH solution was prepared one day before matrices mix by mixing NaOH pellets (purity 98%) with distillated water. The Na₂SiO₃ used consisted of minimum 0.02% Fe, 17-19% Na₂O, 35-38% SiO₂ and 46% H₂O. At the day of mixing, the activator, consisting of Na₂SiO₃ gel and NaOH solution, was mixed. After the solution was ready, fly ash was then added and the mixing process started.

Three groups of vicat needle tests -S1, S2, S3were performed as setting time test. The first, S1, varied ash to activator ratios. Ratio of JFA and IFA matrices synthetization with activator was 1.86; NaOH solution molarity was 8 M; and ratios of activators (Na₂SiO₃/ NaOH solution) for JFA were 1.5 and 2.5; for IFA, they were 1.5, 2 and 2.5. The second, S2, varied ash to activator ratios. JFA and IFA to activator ratios were 1.86, 1.5, and 1.22; NaOH solution molarity was 8 M; and activator ratio was 1.5. The third, S3 varied Si/Al ratios in matrix mixtures. JFA or IFA to activator ratios of Si/Al ratio in mixtures were 1.5, 2 and 2.5. These matrices were synthesized by varying Si/Al ratios. As [17] notes, Si/Al ratios determine suitability of matrices for concrete. In this study, each ash matrix was composed of 3 variations of Si/Al molar ratios of 1, 2, and 2.5 as shown in Table 1. All matrices were cast into several molds. covered with plastic, and stored at room temperature of 27°C. The setting time test was performed according to [18]. Compression strength test was performed using 50-mm diameter cylinders with a ratio of 1/2 diameter to length. Plastic sheeting was wrapped to prevent evaporation. Curing processes were conducted at a temperature of 27°C.

3. RESULT AND DISCUSSION

3.1.Fly Ash Characterized Testing Result

XRF results, as shown in Table 2, Table 1 indicate chemical compositions of JFA and IFA were similar, with the exception of CaO being 1.12% greater in IFA. It was therefore expected that both ash types would be similar in mechanical properties. XRF shows the percentage of Fe_2O_3 in IFA to be greater than that of JFA, while S exists in JFA. Both Fe_2O_3 and S are disruptors which impede the geopolymerization process [15].

XRD test identified the crystalline phase in the ash illustrated in Fig. 1. It was found that a large part of ash was amorphous, in accordance with [19]. Despite that result, both ash types contain minor portions of crystalline phases such as quartz, hematite, and mullite. Furthermore, IFA also contains magnetite, lime, anhydrite and srebrodolskite. XRD result of JFA shows that the hump was at 2 θ of 18 to 27 degree. However, IFA were without such a hump. The pattern also shows the highest intensity peak of quartz for both ash types at 2θ of 27 degree. JFA has a lower peak than IFA. For disruptor components, IFA contains magnetite and hematite, while JFA only contains hematite. The lower lime content in JFA may not support reaction, which may extend setting time [15]. These qualitative XRD results obtained for

Table 1 Ratio of matrices proportion at S3

Molar Ratio		JFA matrices			IFA matrices	
	JFA-1.5	JFA-2	JFA-2.5	IFA-1.5	IFA-2	IFA-2.5
Si/Al	1.5	2	2.5	1.5	2	2.5
SiO ₂ /Al ₂ O ₃	3	4	5	3	4	5
Na ₂ O/SiO ₂	0.3	0.3	0.3	0.3	0.3	0.38
Na ₂ O/Al ₂ O ₃	0.9	1.3	1.5	0.9	1.3	1.9
H ₂ O/SiO ₂	3	3	2.9	3	2.8	3.5
H ₂ O/Na ₂ O	9.6	9.6	9.2	10.6	9	9
*water/solid	0.36	0.38	0.39	0.31	0.35	0.45

*in mass ratio

JFA and IFA need to be supported by semiquantitative estimation of phase concentrations since the percentage of glass and crystalline content in total mass must be acquired; this is because a high percentage of reactive Si and Al in amorphous phases in ash are a primary requirement to undergo geopolymerization. Davidovits noted that according to set criteria, ash with mullite content exceeding 5% and Fe₂O₃ exceeding 10% is not suitable for geopolymers [1]. Higher amounts of glassy compound result in faster reaction. In contrast, higher content of crystalline results in a drastic decline in Al-Si decline reactivity.

Result of EPMA indicate that JFA contains several elements such as O, Na, Mg, Al, Si, P, S, K, Ca, Ti, and Fe. EPMA graph also shows that IFA shown in Fig. 2 has identical elements excepting the absence of oxide P. Fe and Ca content of IFA is far higher than JFA shown in Fig. 3. Compared to XRF pattern, EPMA showed good agreement, indicating that IFA has a higher percentage of hematite and lime (12.54% and 5.18%, respectively) than JFA (7.57% and 4.06%). The presence of Ca influences setting time and strength.

Ash microstructure form, containing generally glassy, spherical particles, is in agreement with [1]. Various shapes, sizes, and surfaces were found. SEM results revealed that the majority of JFA particles were composed of solid spheres, while IFA consisted of irregularly shaped particles as shown in Fig. 4. This is likely due to the low temperature of combustion and rapid cooling of the Indonesian coal comparing with Reihoku due to the Japanese power plant system. The irregular shapes of IFA demand more water than JFA for ash to be applied as matrices.

Reactivity test indicate that IFA mixture had higher reactivity than JFA. Reactivity of Portland cement, IFA and JFA mixtures were respectively 100%, 14% and 5%. This test showed the potential of ash to start both geopolymerization reaction and hydration simultaneously. In order to obtain reactive Si content, other tests were also conducted, resulting in reactive Si in IFA of 65% and JFA of 22% of Si total as determined by XRF. This reactivity result was used for matrix geopolymer design.

3.2. Chemical Composition of Geopolymer Matrices

In order to assist the design of low calcium fly ash based geopolymers, this study calculated molar ratio of chemical content of fly ash geopolymer matrices design: SiO_2/Al_2O_3 , SiO_2/Na_2O , Na_2O/Al_2O_3 , H_2O/SiO_2 , H_2O/Na_2O , and mass ratio of water/solid are listed in Table 1. Chemical content of matrices was derived from fly ash: SiO_2 , Al_2O_3 and Na_2O , etc.; sodium hydroxide solution: Na_2O and H_2O ; and sodium silicate gel: SiO_2 , Na_2O , and H_2O .

3.3.Setting Time of Matrices

Three setting time experiment sets were conducted using various compositions to investigate effects of fly ash and matrices on setting time. Both initial and final states were measured.



Table 2. Composition of fly ash as determined by XRF analysis (mass %)

Oxide (%)	Al ₂ O ₃	SiO ₂ ^a	S	K ₂ O	CaO ^b	TiO ₂	Fe ₂ O ₃	Na ₂ O	K ₂ O	MgO	Mn ₂ O ₃	$\begin{array}{c} SiO_2 + \\ Al_2O_3 + \\ Fe_2O_3 \end{array}$
JFA	25.35	55.19	0.98	1.09	4.06	1.98	7.57	-	-	-	-	88.11
IFA	26.05	48.47	-	1.66	5.18	0.92	12.54	0.47	1.66	2.77	0.19	87.06

^a As JIS A6201-2008, type II/ class F of fly ash contains SiO2 \ge 45 %; ^{b, c} as ASTM C618, class F of fly ash contains CaO \le 10 % and total of SiO₂+Al₂O₃+Fe₂O₃ \ge 70%



Fig. 3 EPMA of the JFA





Activators (Na2SiO3/NaOH) Mass Ratio

Fig. 5 Relationship of setting time of IFA with alkaline activators ratio at S1







Both initial and final setting times of IFA matrices with 8 M NaOH solution show decreases with an increase in activator ratio as shown in Fig. 5. These results indicate that an increased ratio of activators result in a shorter setting time, and that geopolymerization was thus faster at higher activator ratios. The final setting time of IFA matrices with activator ratios of 1.5, 2 and 2.5



Activators (Na2SiO3/NaOH) Mass Ratio

Fig. 6 JFA setting time with sodium hydroxide (8 and 14 M) and activators ratio (1.5 and 2.5) at S2



Fig. 8 Setting time with Si/Al molar ratio and water/solid mass ratio at S3

were 12.9 h, 10.9 h, and 7.3 h respectively. Contrarily, JFA did not accord to this trend, instead requiring longer setting times with an increase in activator ratios (1.5 and 2.5): 23.3h and 26.9 h, respectively, as indicated in Fig. 6. This differed from JFA with 14 M sodium hydroxide: contrary to the tendency of IFA, Fig. 6 shows an increase in activator ratio actually decreases setting times, which were 46.8 h and 28.7 h respectively. The discrepancy of these results may be due to differences in chemical content and reactivity of the two ash types. These results also show the

difficulty of the first experiment using vicat needle (S1) in discerning which factors influence setting.

NaOH solution reacting with fly ash affects geopolymeric matrices setting time. Fig. 6 shows JFA setting time under ratio of two activators of 1.5 and 2.5, and NaOH molarities of 8 and 14 M. Higher NaOH molarities resulted in longer setting times under both activator ratios. In consequence of activator ratio, the higher NaOH molarity resulted in a denser mixture. More highly concentrated matrixes resulted in slower reaction since there is not enough space for reaction and it requires more water. These circumstances lengthen setting time. Fig. 7 shows that a decrease in ash to activators mass ratio results in slower setting times. This is caused by an increase in the ash/activators mass ratio, which increases the ash amount available to absorb activators, thus resulting in stiffer matrices and decreased setting time. These three above-mentioned factors may not apply for general matrices using different fly ash and activators.

3.3.2.Effect of chemical compounds ratio: Si/Al molar ratio, water/solid mass ratio

Soluble silica is a significant factor. As a result, IFA matrices final setting times with Si/Al ratios of 1.5, 2 and 2.5 were, respectively, 34.1 h (1.5 days), 26.1 h (1 day), and 48.4 (2 days), while those for JFA were longer at about 108.4 h (4.5 days), 98.0 h (4 days), and 163.9 h (7 days). These results in accordance with are [15]: geopolymerization occurs between alkaline and Si-Al to build polymeric chains and bonding structures of Si-O-Al-O. Therefore, a particular Si/Al ratio is required to form chain and ring polymers. Fig. 8 shows that both insufficient and excessive Si/Al result in prolong setting times. In this study, a Si/Al ratio of 2 resulted in a quick setting time as a sufficient amount of Si formed more oligomers, shortening the time demanded to develop geopolymers, while a surplus Si amount in solution obstructed the dissolution reaction by silica concentration.

Both low and high water/solid mass ratios resulted in longer setting times as shown in Fig. 8, while certain middling w/s ratio resulted in reduced setting times. Interestingly, JFA required longer setting times than IFA when the w/s ratio applied to IFA was higher than that to the JFA matrices. Building upon the above results, the higher calcium content of IFA compared with JFA has an important effect on setting time, which conforms to [20].

3.4.SEM, Setting Time and Compression Strength of Matrices

Two matrices of JFA with variation of activators ratio: 1.5 and 2.5 and one of IFA: 2.5 were mixed to investigate surface morphology of the matrix. The SEM test was conducted at 21st days. As the activator ratio was increased from 1.5 to 2.5, the JFA-based geopolymer microstructure demonstrated slightly different proportions of unreacted raw materials, as can be seen in Fig. 9. It was observed that the JFA matrix with activator ratio of 1.5 was covered in unreacted raw material precursor which had formed on the crust. Some pores occurred in the surface of matrix. Nevertheless, the JFA matrix with activator ratio of 2.5 displayed a slightly greater homogeneity than that of JFA1.5. It was also found that, compared with JFA matrices, IFA matrices have a slightly lower proportion of unreacted raw materials. This was consistent with the finding of slightly higher compressive strength shown in Table 3. These results did not indicate significant differences and were in accordance with the only slight differences found in their compressive strength. Designs, which had a marked effect on strength, are shown in Table 3. The matrix with the highest compressive strength of 57.5 MPa was produced by IFA with an activator ratio of 1.5, whilst JFA achieved up to 47.7 MPa. Conversely, for an activator ratio of 2.5, JFA reached slightly higher than IFA in strength. Setting time shows the same trend: IFA matrices with an activator ratio of 1.5 had longer setting times than that with 2.5. The IFA with an activator ratio of 1.5 required 12.9 hours to set, while that with 2.5 needed only 7.3 hours. JFA matrices did not follow this pattern, displaying longer setting times with an increase in activator ratio: ratios of 1.5 and 2.5 were, respectively, 23.3h and 26.9 hours. This agrees with [21] that increasing SiO₂/Al₂O₃ molar ratio generally decreases the reaction initial rate.

Table 3 shows that for the IFA matrices, an increased molar ratio of SiO_2/Al_2O_3 (2.83, 2.97) and SiO_2/Na_2O (3.89, 4.25) resulted in a decrease in strength: 57.50 and 50.50 respectively. Conversely, an increase molar ratio of H_2O/Na_2O (9.14, 8.92) and mass ratio of water/solid (0.27, 0.26) caused a decrease in strength.



Fig. 9 SEM images of fly ash based geopolymeric matrices: (a, b) JFA matrices with activators of 1.5 and 2.5, (c) IFA matrix with activators of 2.5

Table 3. Chemical composition matrices and compression strength values

							Set	°Strength
^{a.} Design	SiO ₂ /Al ₂ O ₃	SiO ₂ /Na ₂ O	Na ₂ O/Al ₂ O ₃	H_2O/SiO_2	H ₂ O/Na ₂ O	^b w/s	Time	
	All r	atios are mola	r (MR) except v	v/s, which is i	n mass ratio		hour	MPa
IFA 1.5	2.83	3.89	0.73	2.35	9.14	0.27	12.9	57.50
IFA 2.5	2.97	4.25	0.70	2.10	8.92	0.26	7.3	50.50
JFA 1.5	3.20	4.28	0.75	2.14	9.14	0.27	23.3	47.70
JFA 2.5	3.35	4.66	0.72	1.91	8.92	0.26	26.9	50.90

^a·Matrices from JFA and IFA with ratio of ash to activators of 1.86, 8 M sodium hydroxide, and variation in activators ratio; ^b· w/s: water to solid mass ratio; ^c· strength: compressive strength

Regarding [1], to produce geopolymer with a user-friendly category activator, the SiO₂/Na₂O molar ratio must exceed 1.45, which was considered in this design. Furthermore, effects of SiO₂/Na₂O molar ratio on compressive strength appear insignificant, which is in accordance with [22]. In addition, shape of particle is found to influence the setting time [23]. The discrepancy between matrices of both ash types are in accordance with Provis at [21], who stated that the chemistry of geopolymeric aluminosilicate materials is merely commencement to be understood. With the further development of this understanding that each ash shows differences on the setting time and mechanical properties of ashbased geopolymers, it comes the ability to design geopolymers composition is limited to particular applications.

4. CONCLUSION

This study showed evidence that though ashes may contain similar chemical compounds and be classified as the same type, differences in combustion temperatures and conditions affect the ash, causing large differences on the setting time of matrices. The main factors of ash which can favor reaction are large amounts of Si-Al, morphology containing irregularly shaped particles, lime, and amorphous phase; conversely, factors which can hinder reaction are large amounts of crystalline phase, Fe₂O₃, and S. Moreover, chemical molar ratios reveal that the proportion of SiO₂/Al₂O₃, SiO₂/Na₂O H₂O/Na₂O and H₂O/Na₂O impact compressive strength of matrices.

Neither the experiment grouping matrices based on determining variations of activator-fly ash nor that determining chemical compound ratios was sufficient to reach definite conclusions due to discrepancies in results. Therefore, the design of the geopolymers were unsuitable to general applications. Geopolymerization much needs to be further developed understood to design geopolymers that is not limited to specific raw material applications, even for common applications.

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