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A. ANTONI[1*], I.J. YUGIARTO[1], R.J.S. HADI[1], A. KUNCORO[1], D. HARDJITO[1]

INFLUENCE OF CFBC FLY ASH CHEMICAL COMPOSITION AND MECHANICAL ACTIVATION ON THE PROPERTIES OF GEOPOLYMER

Circulating fluidized bed combustion (CFBC) fly ash has the potential as a precursor to making geopolymer concrete because of its rich silica and alumina content. However, there is a problem in utilizing CFBC fly ash caused by its chemical and physical properties that differ from the widely used pulverized coal combustion (PCC) fly ash. CFBC fly ash has a higher water requirement than PCC fly ash due to its angular particle shape, and higher sulfur and lime contained also caused a different reaction in the geopolymer system. Mechanical activation by milling the CFBC fly ash could decrease the water requirement in the mixture and make good quality CFBC fly sh-based geopolymer concrete. Three CFBC fly ash samples from different power plants in Indonesia with different chemical compositions were used in this research. The first had a low lime and no sulfur content, the second had high lime and no sulfur content, and the third had high lime and sulfur content. The milling process using a ball mill for two hours decreased the water requirement, as shown in the lower normal consistency of the fly ash. The reactivity also was increased, shown by the faster initial setting time. Besides its higher reactivity, lower water requirement increased the compressive strength of geopolymer mortar produced. This study also showed that the existence of calcium and sulfur content in the CFBC fly ash could cause unexpected results shown by the change of initial setting time, water requirement, and compressive strength.

Keywords: Fly ash; CFBC; mechanical activation; geopolymer; chemical composition

1. Introduction

Geopolymer mortar is produced from the reaction of source material rich in silica and alumina with an alkaline activator [1]. Using geopolymer as a binder can reduce the use of Portland cement and its carbon dioxide emission in the manufacturing process. The reduction of Portland cement consumption would reduce the global warming effect and climate change in the long term [2]. Fly ash, a precursor in making geopolymer concrete, has a rich alumina and silica content and can produce good-quality geopolymer concrete [3]. Fly ash, a waste material from burning coal in power plants, has been studied extensively for its viability in geopolymer systems. Considering the low cost and availability of fly ash, the effort to utilize fly ash as Portland cement replacement needs to be increased.

Based on the combustion system, pulverized coal combustion (PCC) fly ash is more commonly used and studied, as it would provide better workability and reactivity. However, recently circulating fluidized bed combustion (CFBC) powerplant has gained traction due to its lower burning temperature and cleaner combustion because the ash goes through the repeated circulating process. The CFBC burning process also has a higher efficiency when compared to the PCC burner, and more CFBC burner is being utilized in many parts of the world [4,5]. The CFBC fly ash is rarely used as a cement replacement material because it has an irregular shape and large surface area that requires higher water content in the mixture [6,7]. Nevertheless, CFBC fly ash still has the potential to be used as a material in construction as a cement substitute because CFBC fly ash has a pozzolanic activity that is good and has the self-cementitious characteristic [8]. The CFBC fly ash is rarely used as a geopolymer precursor due to its physical properties and chemical content [9,10]. The CFBC fly ash often has a high calcium oxide and sulfur content when the coal source contains sulfur [11,12]. The chemical content is due to the CFBC burning process introducing calcium carbonate into the combustion chamber to bind the sulfur compound released when burning the coal.

The CFBC fly ash could be mechanically activated by milling to improve the physical properties and reduce water requirements in the fresh mixture. The mechanical activation also increases the chemical reactivity of the material. Using a ball milling process decreased the water requirement on CFBC fly

[1] PETRA CHRISTIAN UNIVERSITY, DEPARTMENT OF CIVIL ENGINEERING, JL. SIWALANKERTO 121-131, SURABAYA 60236, INDONESIA

* Corresponding author: antoni@petra.ac.id

ash, increased its specific gravity and compactness, and increased the compressive strength [13,14]. The milling process on fly ash can produce higher reactivity and compressive strength due to the particle becoming finer and more homogeneous [15]. However, the previous study mainly focused on the mechanical activation of the CFBC fly ash. At the same time, the current study also investigated the influence of chemical compositions on the geopolymerisation reactions. The effectivity of the milling depends on the method of milling and duration, and the improvement rate also depends on the chemical composition of the fly ash itself.

This study evaluates the CFBC fly ash as a precursor for geopolymer mortar from the chemical composition and after mechanical activation using ball milling. This research also continues our previous study on low-sulfur CFBC fly ash [16]. Three CFBC fly ash samples were obtained from three power plants in Indonesia, and the fly ash obtained had different chemical compositions in their calcium and sulfur content. Mechanical activation by ball milling was done to change the water requirement of the fly ash in the mixture. Changes in fresh and hardened properties were evaluated from its change of normal consistency, specific gravity, setting time, and compressive strength.

2. Experimental method

2.1. Materials

This research used three CFBC fly ash sources from small CFBC powerplants from Ngoro, East Java, the island of Belitung, and the region of Gorontalo in Indonesia coded N, B, and G. The CFBC powerplants have a capacity of 2×15 MW, 2×16.5 MW, and 2×25 MW, respectively. Fig. 1 shows the fly ash samples obtained. Fly ash N had the lightest color, whereas fly ash B was the darkest, and fly ash G was brownish-red.

These three fly ash samples were tested for their pH, chemical composition, phase by X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) test, and shapes by Scanning Electron Microscope (SEM). The physical properties of normal consistency, specific gravity, and particle size analysis were also done on the fly ash samples before and after the ball milling process. The geopolymer mortar was made alkaline activator in the form of several concentrations of an aqueous solution of NaOH and the sodium silicate in the liquid form obtained from the local chemical supplier in Surabaya, Indonesia. The sodium silicate's aqueous content was measured at 50% in the laboratory oven. Graded silica sand was used as filler in making the geopolymer mortar, and the fineness modulus of the silica sand used was controlled at 2.05.

2.2. Methods and Tests

Mechanical activation on the fly ash was done by the ball milling process. The ball mill used was a steel cylinder with a diameter of 35 cm and length of 42 cm, and it rotates at a constant speed of 49 rotations/minute. The milling material inside used cylindrical shape (cylpebs) steel media with a diameter and height of 35 mm. The ratio of fly ash to cylpebs was 1:10 by mass. The duration of the milling process was set at two hours in this study, with a fly ash sample of 2.5 kg for each milling process.

The geopolymer mortar was made by directly mixing the CFBC fly ash, sand, and alkaline activator. The water-to-binder ratio was determined using the flow table test with a target flow diameter of 15±1 cm for the same consistency between mixes. Due to the higher water requirement of CFBC fly ash, the water-to-binder ratio was found at 0.72 for the three untreated fly ash samples. This value also corresponds to the higher normal consistency of the CFBC fly ash measured from the Vicat's normal consistency test of 0.67 to 0.70. After the milling process, there was a reduction in the normal consistency value, and subsequently, the water-to-binder ratio for the milled fly ash samples was reduced to 0.44 and 0.61.

Each untreated (U) and milled (M) fly ash sample was made into a geopolymer mortar with a NaOH solution concentration of 6M, 8M, and 10M, with the ratio of sodium silicate liquid to sodium hydroxide solution (SS/NaOH) of 2.0, 2.5, and 3.0, by mass. The mass ratio of sand to fly ash was 2 for all mixtures. Table 1 shows the mix proportion of three 5 cm cubes of geopolymer mortar with a water-to-binder ratio (w/b) of 0.72, 0.61, and 0.44 that was used to make the geopolymer mortar in this study based on the workability target.

The geopolymer mortar was cast into 5 cm cubes for compressive strength testing. An oven-curing process of 60°C for 24 hours was done for all specimens sealed in their mold to accelerate the geopolymerization process [1,17]. The compressive strength tests were done at 7 days, 28 days, and 56 days with three replications. The initial setting time test was measured on

(a) (b) (c)

Fig. 1. The physical condition of the untreated CFBC fly ash. (a) fly ash N, (b) fly ash B, (c) fly ash G

TABLE 1

Mix design for mortar geopolymer based on its water requirement

				w/b = 0.72			w/b = 0.61			w/b = 0.44		
NaOH concentration	**Sodium Silicate (*l*): NaOH (*l*)**	**Fly ash (g)**	**Sand (g)**	**NaOH (*s*) (g)**	**Water (g)**	**Sodium Silicate (*l*) (g)**	**NaOH (*s*) (g)**	**Water (g)**	**Sodium Silicate (*l*) (g)**	**NaOH (*s*) (g)**	**Water (g)**	**Sodium Silicate (*l*) (g)**
6M	2.0	300	600	23.1	96.4	239.2	19.6	81.7	202.6	14.1	58.9	146.1
	2.5			20.3	84.7	262.6	17.2	71.8	222.5	12.4	51.8	160.5
	3.0			18.1	75.5	281.0	15.4	64.0	238.0	11.1	46.2	171.7
8M	2.0	300	600	29.8	93.1	245.8	25.2	78.9	208.2	18.2	56.9	150.2
	2.5			26.1	81.5	268.9	22.1	69.1	227.9	15.9	49.8	164.4
	3.0			23.2	72.5	287.0	19.7	61.4	243.2	14.2	44.3	175.4
10M	2.0	300	600	36.0	90.0	252.0	30.5	76.3	213.5	22.0	55.0	154.0
	2.5			31.4	78.5	275.0	26.6	66.5	232.9	19.2	48.0	168.0
	3.0			28.0	69.7	292.6	23.6	59.0	247.9	27.0	42.6	178.8

the geopolymer paste using the Vicat needle test with the same mixture without adding sand [18].

3. Results and discussions

3.1. Fly Ash characteristics

The result of the XRF test of the three fly ash samples is shown in TABLE 2. Based on ASTM C618-19 [19], the fly ash classified in the F class has SiO_2+Al_2O_3+Fe_2O_3 of more than 50%, CaO less than 18%, and SO_3 is less than 5%. Therefore, fly ash N is categorized as class F, fly ash B as class C, and fly ash G with a sulfur content higher than 5% cannot be included in the classification. The result of the XRD test is shown in Fig. 2. These three samples of fly ash had multiple peaks in common. One of them was on the highest position peak at 26.6°, which was Quartz (Q). 20.8° peak and 50.1° also showed the existence of Quartz, whereas at 35.6°, fly ash N and G showed the existence of Magnetite (MA). Moreover, fly ash G contained Mullite (M) at 25.5°.

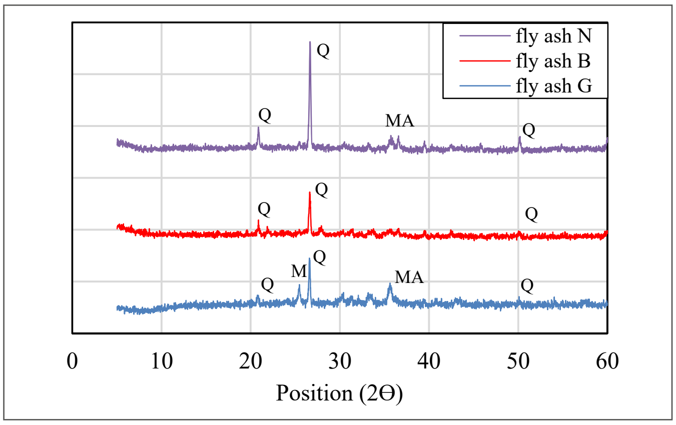


Fig. 2. Result of the XRD Test

Fig. 3 shows the SEM micrographs of the three untreated CFBC fly ash used in this study. The fly ash particles mainly

TABLE 2

Result of XRF composition and pH of the fly ash

Fly ash	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	MnO	K_2O	BaO	TiO_2	HgO	pH
N	40.25	15.46	31.97	**6.25**	—	0.31	1.97	0.30	1.84	—	8.0
B	35.59	16.80	23.82	**18.79**	—	0.87	0.44	0.44	2.28	0.68	10.5
G	18.33	7.91	47.17	**16.82**	**5.21**	0.51	0.93	0.56	1.04	—	11.0

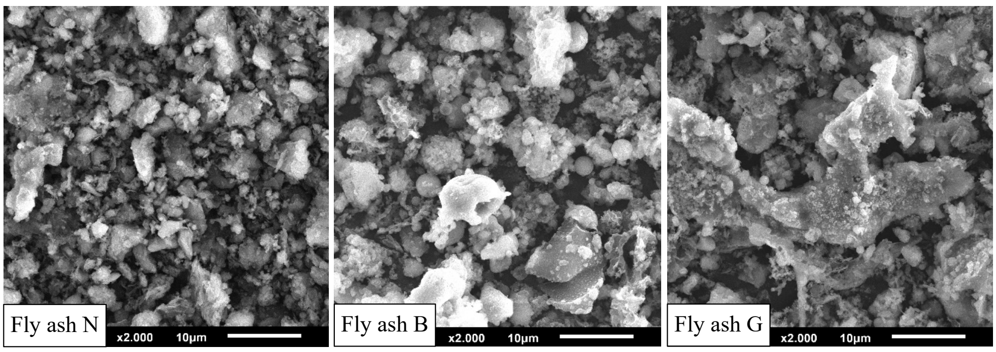


Fig. 3. SEM micrographs of the untreated fly ash

have irregular shapes with considerable variation in their size. A lower temperature in the CFBC burner than that of the PCC burner resulted in only a partial fusion of the solid materials. It caused the ash material to form irregular shapes and required higher water content for comparable workability. Ball shape particles were observed on the three fly ash samples, but only in a small fraction.

3.2. Mechanical activation

The mechanical activation by milling the fly ash in a ball mill could reduce the water requirement of the fly ash shown by the normal consistency test. The three fly ash samples had a reduction of about 0.7 to about 0.3 when tested for normal consistency, as shown in TABLE 4. Fu et al. also reported a reduction of normal consistency after milling [13]. Although the milling process reduced the normal consistency, it was not as low as the PCC fly ash, which usually has normal consistency of about 0.2. The milling process could reduce the particle size and break down large pores or voids in the fly ash particles. This pore reduction in the fly ash is also supported by the increase of specific gravity for each fly ash [20].

The particle size distribution summary of the fly ash is also shown in TABLE 3. The milling process was shown to reduce the particle size of the fly ash B and G. However, fly ash N was shown to have a slight increase in its diameter, and the result could be due to the agglomeration of the fly ash particle due to the occurrence of surface ionic charge in the milling process. The normal consistency of fly ash N was reduced significantly, showing that the particle size measured by particle size analysis (PSA) cannot directly be used to judge the physical properties of the fly ash.

The water-to-binder ratio in making geopolymer mortar needs to be determined by considering the normal consistency of the CFBC fly ash. The water content needs to be selected to achieve the workable target mix since no admixture was used in making the geopolymer mortar. The w/b was selected at 0.72 for the untreated fly ash and w/b of 0.44 for the milled fly ash after several trial mixes. However, the fly ash G required higher water content to achieve the desired workability in the geopolymer mortar mixture, hence w/b of 0.61 was used. This higher water demand could be attributed to the sulfur compound in the fly ash G.

3.3. Initial setting time

The initial setting time test was done on the paste sample using the Vicat needle. The same w/b was used to make the geopolymer paste, as shown in TABLE 1, without adding sand. All untreated CFBC fly ash used w/b of 0.72, whereas for milled fly ash N and B, w/b of 0.44 was used, and milled fly ash G used w/b of 0.61. The initial setting time test is shown in Fig. 4. The different maximum values of the vertical axis showing the setting time for each CFBC fly ash must be noted. From the initial

TABLE 3

Physical properties of the before and after the milling process

Material	Condition	Normal Consistency	Specific Gravity	d(10) (µm)	d(50) (µm)	d(90) (µm)	Mean (µm)
N	Untreated	0.67	2.08	2.36	34.76	94.73	41.28
	Milled	0.28	2.30	2.02	36.39	111.05	45.08
B	Untreated	0.70	1.86	3.66	37.09	113.58	48.17
	Milled	0.33	2.11	0.81	19.64	46.43	21.39
G	Untreated	0.70	2.39	2.76	25.33	62.92	30.85
	Milled	0.35	2.55	1.14	24.75	48.43	24.17

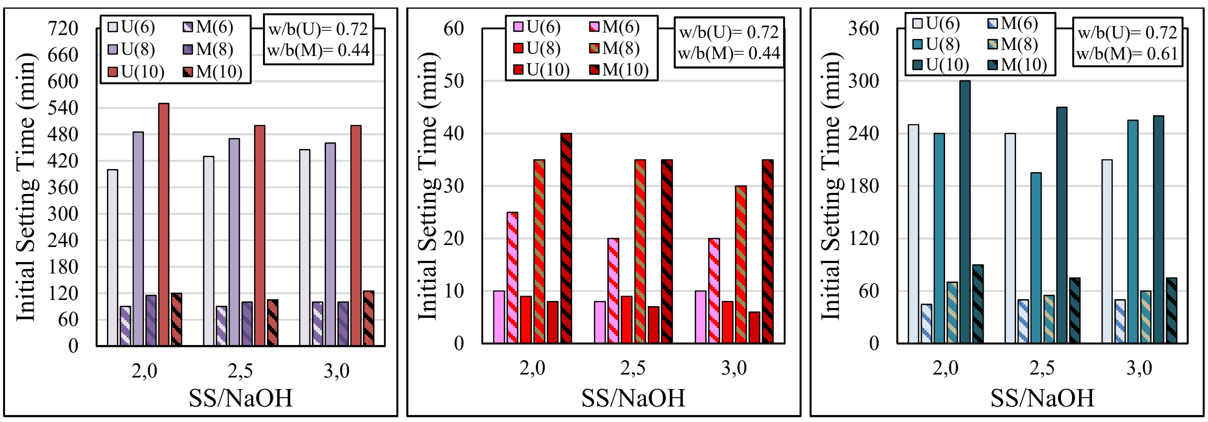


Fig. 4. Result of the initial setting time test for (a) fly ash N, (b) fly ash B, and (c) fly ash G

setting time test, there was a significant trend of initial setting time obtained mainly due to the variation of the chemical content of the fly ash consistent with previous findings [20].

Fly ash N, with its low calcium content, had the longest initial setting time in the geopolymer mixture. The untreated fly ash N had an initial setting time of 400 to 550 minutes with a noticeable trend based on the variation of NaOH concentration and ratio of sodium silicate to the NaOH solution. The milled fly ash N had a faster initial setting time of 90 to 125 minutes, with the same trend based on the alkaline activator concentration and ratio. The reduction of initial setting time was found to have a beneficial effect because of the increased reactivity in the fly ash from the milling process. However, the reduction of the water-to-binder ratio in the mixture also influenced the faster setting time.

Fly ash B with high calcium content was shown to have a fast initial setting time. The untreated fly ash B had an initial setting of 6 to 8 minutes. The fly ash B was similar to the high calcium PCC fly ash with a very fast initial setting time that can be considered a flash setting condition [21,22]. The milled fly ash B was shown to have a slightly longer initial setting time, contrary to the expected higher reactivity from the milling process. The longer setting time could be due to the reduction of reactivity of the finer calcium oxide particle exposed to carbon dioxide while in storage since there was a lag time from milling until the preparation of geopolymer paste. Further study is needed to confirm this effect. The influence of the alkaline activator concentration and its ratio remained consistent with the fly ash N.

The initial setting time of fly ash G had a similar trend with fly ash N but with a faster setting time. The alkaline activator concentration and its ratio have the same trend as the fly ash N, but with some mixed results in the NaOH concentration of 8 M. The faster setting time could be due to the fly ash G having a higher content of calcium oxide than that of fly ash N. However, the availability of sulfur oxide in the fly ash could cause complications in the mixing process and require higher water content for the milled fly ash G; thus higher w/b of 0.61 was used. The faster setting time of the fly ash G also did not correlate with the increased quality of the geopolymer mortar produced, as shown by its strength development.

3.4. Compressive strength

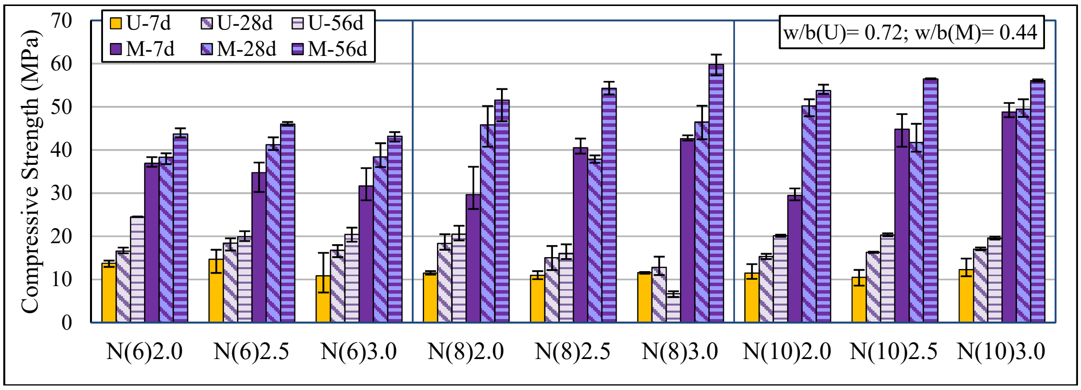
The compressive strength test was done for untreated and milled fly ash N, B, and G samples for 7, 28, and 56 days. The code (6), (8), and (10) denote the molar concentration of NaOH solution, whereas 2.0, 2.5, and 3.0 are the ratio of sodium silicate and NaOH solutions. Fig. 5 shows the compressive strength obtained for each mixture with three replications for each test. The value of w/b used to make the geopolymer mortar was also included in the figure since there were variations in the water demand for the target workability. This change of w/b would affect the compressive strength obtained, and the trend with a variation of the alkaline activator concentration and its ratio was observed.

Fig. 5(a) shows the compressive strength result of the untreated and milled fly ash N. The highest compressive strength for the untreated fly ash N was observed on the N(6)2.0 with 24.51 MPa at 56 days, whereas the milled fly ash N sample obtained 59.79 MPa at 56 days for a mixture of N(8)3.0. There was a significant increase in compressive strength for all of the milled fly ash, showing that the mechanically activated CFBC fly ash can still be used as a geopolymer precursor if we can reduce the water demand in the mixture. The geopolymer system is incompatible with the plasticizer used in the Portland cement mixture. Thus the reduction of water demand in the geopolymer solely depends on the water demand of the binder used. With the changes in alkaline activator concentration and its ratio, there were changes in the compressive strength obtained. However, for the range of alkaline activator ratio used in this study, the compressive strength of the geopolymer mortar was found to be sufficiently similar.

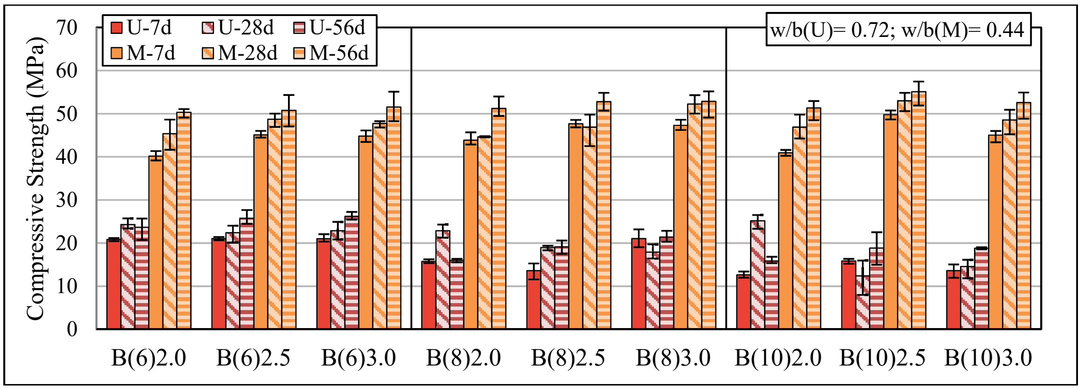
The same compressive strength trend was also observed for untreated and milled fly ash B, shown in Fig. 5(b). The untreated fly ash B had the highest compressive strength on mixture B(6)3.0 with a strength of 26.24 MPa at 56 days, and the milled fly ash had the increased compressive strength of 55.11 MPa at 56 days on B(10)2.5 mixture. The compressive strength could increase about two-fold to around 50 MPa when the milled fly ash B was used. The reduction of w/b undoubtedly influenced the increase in strength, but it could also be due to the increased reactivity of the fly ash. Higher calcium oxide content in fly ash B was not observed to influence the compressive strength obtained.

For the fly ash G (Fig. 5(c)), it was found that the mechanical activation was not effective in reducing the water demand in the geopolymer mixture nor in increasing the compressive strength of the mortar. The highest compressive strength obtained in the milled fly ash G with a mixture of G(10)2.0 was 23.09 MPa, similar to the untreated fly ash with compressive strength of around 20 MPa. The influence of sulfur compound in the CFBC fly ash for the geopolymer system was found to have a detrimental effect which could be due to higher water demand in the fresh mixture and also incompatibility of reaction in the geopolymer system. This finding has a very significant consequence when using CFBC fly ash as a precursor in the geopolymer system, as the majority of the CFBC powerplant produces fly ash with higher sulfur oxide content due to the design of the combustion system.

The mechanical activation of the CFBC fly ash by ball milling could reduce the water demand for good workability and increase the reactivity of the fly ash. However, the geopolymer reaction is still influenced by the chemical content of the fly ash. The initial setting time and compressive strength of the CFBC fly ash-based geopolymer were greatly influenced by its chemical content. Sulfur compounds in the fly ash have a significant incompatibility with the alkaline activator and cause difficulty in fresh geopolymer mortar, limiting the geopolymerization reaction.



(a)



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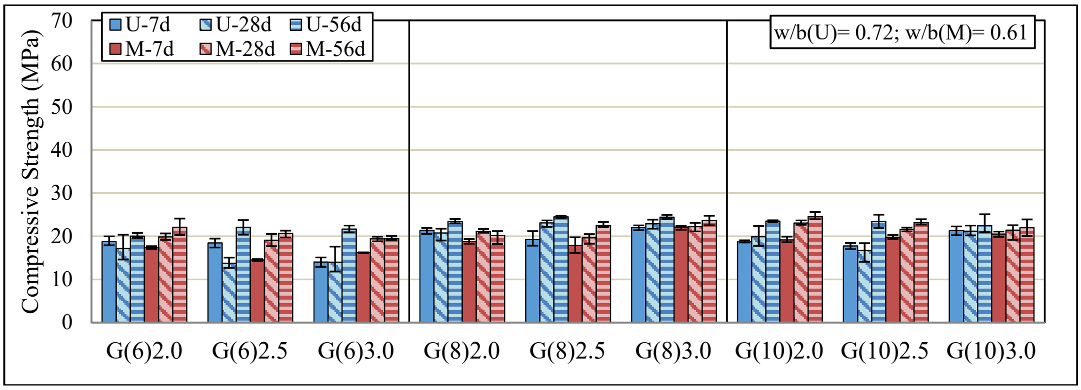

(c)

Fig. 5. The compressive strength of geopolymer mortar samples made from (a) fly ash N, (b) fly ash B, and (c) fly ash G

4. Conclusions

- The characteristic of CFBC fly ash is different from PCC fly ash in terms of chemical composition and physical properties. The CFBC fly ash potentially have a higher sulfur and calcium content due to the design of the CFBC system to bind sulfur in the coal with the use of calcium carbonate. The physical particle of the CFBC fly ash has an irregular shape and requires higher water content for the same target workability to be used as a base material of geopolymer mortar.
- Mechanical activation by ball milling on CFBC fly ash reduces the particle size and break pore or void in the fly ash reducing the water requirement, which was shown with the decrease in the normal consistency value. With the reduction of the water requirement, higher geopolymer strength can be obtained with a lower water-to-binder ratio in the mixture.
- The mechanical activation of the CFBC fly ash also changes the reactivity of the fly ash and its initial setting time. However, the chemical composition of the fly ash has a more significant influence on the setting time process. High calcium CFBC fly ash has a very fast initial setting time, while low calcium CFBC fly ash tends to have a longer setting time, similar to PCC fly ash. The sulfur content on the CFBC fly ash could be incompatible with the alkaline activator geopolymer system but was not previously observed due to the low sulfur content in PCC fly ash.

- The alkaline activator composition has some influence on the geopolymer mortar produced. However, within the current study, the geopolymer mortar can be produced by varying sodium hydroxide concentration from 6 M to 10 M, and the ratio of sodium silicate to sodium hydroxide solution of 2.0 to 3.0. The compressive strength was more influenced by its water-to-binder ratio.
- The existence of sulfur compound in CFBC fly ash affects the geopolymer reaction adversely. This negative effect could be due to the higher water demand of the sulfur compound in the fly ash or the incompatibility of the sulfur compound with the alkaline activator. Further investigation is needed to clarify the incompatibility reaction of the sulfur compound in the geopolymer system.

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REFERENCES

[1] N.A. Lloyd, B.V. Rangan, Geopolymer Concrete with Fly Ash (2010).
[2] K.S. Devi, V.V. Lakshmi, A. Alakanandana, Impacts of Cement Industry on Environment-An Overview, (2017).
[3] M.I.A. Aleem, P.D. Arumairaj, Int. J. Eng. Sci. Technol. **1**, 118-122, (2012).
[4] M. Zahedi, F. Rajabipour, ACI Mater. J. **116**, 163-172 (2019).
[5] P. Chindaprasirt, U. Rattanasak, C. Jaturapitakkul, Cem. Concr. Compos. **33**, 55-60 (2011).
[6] T.L. Robl, K.C. Mahboub, W. Stevens, Second International Conference on Sustainable Construction Materials and Technologies. (2010).
[7] S. Siddique, H. Kim, J.G. Jang, Constr. Build. Mater. **289**, 123150 (2021).
[8] T. Wu, M. Chi, R. Huang, Constr. Build. Mater. **66**, 172-180 (2014).
[9] L. Huo, J. Li, Z. Lu, W, Zhang, C. Hu, J. Wuhan Univ. Technol. **34** (10), 14-18 (2012).
[10] H. Xu, Q. Li, L. Shen, M. Zhang, J. Zhai, J. Waste Manag. **30** (1), 57-62 (2010).
[11] C. Baek, J. Seo, M. Choi, J. Cho, J. Ahn, K. Cho, Sustainability **10** (12), 4854 (2018).
[12] W.G. Lee, J.E. Kim, S.H. Jeon, M.S. Song, J. Korean Ceram. Soc. **54**, 380 (2017).
[13] X. Fu, Q. Li, J. Zhai, G. Sheng, F. Li, Cem. Concr. Compos. **30**, 220 (2008).
[14] R. Szabó, I. Gombkötő, M. Svéda, G. Mucsi, Arch. Metall. Mater. **62**, 2B, 1257-1261 (2017).
DOI: https://doi.org/10.1515/amm-2017-0188
[15] X.G. Li, Q. Bin Chen, K.Z. Huang, B.G. Ma, B. Wu, Constr. Build. Mater. **36**, 182 (2012).
[16] G. Mucsi, S. Kumar, B. Csoke, R. Kumar, Z. Molnár, Á. Rácz, F. Mádai, Á. Debreczeni, Int. J. Miner. **143**, 50 (2015).
[17] A. Antoni, S.D. Shenjaya, M. Lupita, S. Santosa, D. Wiyono, D. Hardjito, Civ. Eng. Dimens. **22** (2), 96-100 (2020).
[18] A. Antoni, S.W. Wijaya, D. Hardjito, Mater. Sci. Forum **841**, 90-97 (2016).
[19] ASTM C618, Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete (2019).
[20] A. Antoni, J. Satria, A. Sugiarto, D. Hardjito, MATEC Web Conf. **97**, 01026 (2017).
[21] A. Antoni, A.A.T. Purwantoro, W.S.P.D. Suyanto, D. Hardjito, Iran. J. Sci. Technol. – Trans. Civ. **44** (1), 535-543 (2020).
[22] R. Mohamed, R.A. Razak, M.M.A.B. Abdullah, L.A. Sofri, I.H.Aziz, N.F. Shahedan, Arch. Metall. Mater. **67**, 2, 563-567 (2022). DOI: https://doi.org/10.24425/amm.2022.137791