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Effect of mixing procedure and steam curing on the properties of Class C fly ash-cement based Geopolymer

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Abstract
This work investigated the performance of class C fly ash-cement-based geopolymer paste with different mixing procedures and under steam curing. The geopolymer paste containing class C fly ash was mixed with cement up to 20% at 10% increments. The alkaline activator was prepared 24 hours before mixing by blending Na_2SiO_3 and NaOH 14 M with a ratio of 2:1. Two methods of mixing procedure were used in this study to mix the geopolymer pastes. Steam curing at 90 ± 5 ˚C for six hours was applied. Setting time, compressive strength and strength activity index were used in this research for performance analysis. According to the results, the setting time of geopolymer paste containing 100% C type flash ash is delayed by the use of method 2 in the mixing process. While, adding cement content speeds up both the initial and final setting times of fresh geopolymer pastes. Additional water at the early stage in the mixing process (method 2) generated excellent polymerization at longer curing periods. In contrast, the potentially agglomeration of fly ash and cement in the polymer matrix was observed after applying method 1, which is the reason for the lower strength of geopolymer paste. The strength activity index (SAI) of geopolymer paste containing cement increased at early curing periods for all mixing methods. The early-stage benefits of steam curing are achieved in geopolymer pastes containing 0% cement when mixed using techniques 1 and 2.

Keywords:
Class C Fly Ash;
Geopolymer;
Microstructures;
Steam Curing;
Strength Activity Index;

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INTRODUCTION

In recent decades, the tremendous production of Ordinary Portland Cement is facing a massive shortage of raw materials and depletion of fossil fuels, which will need a million years to renew [1]. Simultaneously, releasing an enormous amount of CO_2 emission into the atmosphere from clinker calcination of cement production will soon lead to faster global warming. In the meantime, the production of fly ash from coal-fired power plants still increases, which needs a large stockpile area for storing the fly ash. Annually, fly ash production in Indonesia reaches almost 9 million tons, and only 10-12% is used for construction projects. These numbers imply that about 90% of fly ash has not been used, possibly resulting in ecological deterioration caused by noxious components present in fly ash. Therefore, utilizing fly ash on a large scale for construction projects is a key to preventing environmental degradation and saving energy shortly [2][3].

Most researchers reported that the use of class F fly ash in geopolymer concrete obtained a higher compressive strength than class C fly ash due to the higher content of SiO_2 in class F fly ash. The most difficult problem will be the endeavor to improve class C fly ash-based geopolymer performance. Therefore, several researchers used cement and slag to improve the strength of geopolymer as reported by [4][5].

In literatures, most of the researchers study the mix design of geopolymer including water-to-geopolymer solid ratios and alkaline-toaluminosilicates' ratios, but there are few literatures discussing the mixing process of geopolymer. Rangan proposed the mixing process of geopolymer by making the alkaline liquid for 1 day before mixing with dry materials [6]. Other literature modified the mixing procedure of [6] by adding water in the middle of the mixing process [7], and the result of compressive strength was about 18.32 MPa.

The earlier research in this field shows that the curing conditions, among other factors, have a significant impact on how well geopolymer mortar performs [8, 9, 10]. In addition to the curing conditions, applying a heat curing (oven method) increased the strength of geopolymer concrete and mortar [11, 12, 13]. However, using a long duration in heat curing (oven method) decreased the strength of geopolymer mortar [13]. The amorphous geopolymer mortar's breakdown, which exposed the matrix to significant moisture loss, may be to blame for the loss of strength as the period was extended [14][15].

Regarding the literature mentioned, steam curing might prevent moisture loss which is proposed in the current study. The mixing procedure is the key to obtain the optimum results of geopolymer paste (GP) which previous researchers have not extensively discussed. Therefore, this research aims to examine the properties of class C fly ash-cement-based GP (compressive strength and setting time) under mixing methods combined with steam curing.

MATERIAL AND METHOD

Fly Ash and Cement

Fly ash used in this research, as shown in Figure 1, is a by-product from Nii Tanasa Coal-Fired Power Plant, Konawe, Southeast Sulawesi. Based on X-Ray Fluorescence (XRF) result of fly ash, as seen in Table 1, the summation of three oxides ($SiO_2+Al_2O_3+Fe_2O_3=44.51\%$) is lower than 50%. However, the lime content is more than 20%, so the fly ash used in this study is classified as class C fly ash.

Based on the literature, the use of cement as fly ash substitution increased the strength of geopolymer concrete [5]. In this study, the cement used was Ordinary Portland Cement (OPC type I). The chemical composition of fly ash and OPC I is shown in Table 1.

Figure 1. Fly ash

Table 1. Chemical compositions of binders determined by XRF [wt%]

Constituents	Fly ash	OPC I
SiO_2	19.88	19.90
CaO	24.04	64.10
Al_2O_3	12.00	5.30
Fe_2O_3	12.63	3.00
MgO	8.72	2.40
TiO_2	0.58	-
MnO_2	0.22	-
Na_2O	7.47	-
K_2O	2.23	-
P_2O_5	0.18	-
SO_3	10.26	1.90

Alkaline Activator

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were the alkaline activators employed in this study. The sodium hydroxide existed in the shape of pellets having a concentration of 99%.

In alkaline activator manufacturing, most researchers blend sodium hydroxide with water for the desired molarity and leave them for 24 hours for the cooling process before mixing with sodium silicate. However, utilizing class C fly ash in geopolymer promoted flash setting due to the high amount of CaO and Fe_2O_3 [16]. In order to prevent this phenomenon, sodium hydroxide and sodium silicate were mixed at different times to study their effect on setting time [17]. Regarding this, the authors mixed sodium hydroxide and sodium silicate in 1 hour and studied its effect on setting time. In addition, further research is still being carried out regarding the difference in mixing time between sodium hydroxide and sodium silicate and studying its effect on setting time and mechanical strength.

The NaOH was dissolved in distilled water to obtain a molarity of 14 M and left it in a room temperature for 1 hour. The sodium silicate was then mixed with NaOH 14 M with a proportion of 2:1 and kept at ambient temperature for 24 hours before blending.

Mix proportion and mixing procedure

The mixed proportion of GP was tabulated in Table 2. The samples (cylinder with 55 diameters and 100 mm height) were made in the morning at (24 ± 2) ˚C to prevent the faster polymerization at high temperatures.

Table 2. Compositions of GP (g)

Material	0%	10%	20%
Fly ash	1966.4	1740.0	1521.1
OPC 1	0	226.4	445.3
NaOH	229.4	229.4	229.4
Na_2SiO_3	458.8	458.8	458.8
Water	688.2	688.2	688.2

Two methods were used in the mixing process as described below. The part of the mixing procedure is shown in Figure 2.

Method 1

For method 1, the alkaline solution was prepared one day before mixing. Fly ash and cement were mixed in the pan at 83 rpm for 1 min, followed by the manual for 1 min. The alkaline was then inserted and mixed at 132 rpm for 1 minute, followed by the manual for 1 min. Water was poured into the mixing pan and mixed at 132 rpm for the 30s. The medium speed at 328 rpm, and also manual mixing was applied for each 1 min. In the next step, the high speed at 570 rpm was applied three different times (1.5 min, 2.5 min, and 2.5 min), which was interspersed with manual mixing for 1 min. In the last step, the fresh (GP) was inserted into the molds and wrapped with plastic to prevent excessive evaporation. In this method, water was added to the mixing process as the mixture hardened quickly during the mixing time.

Figure. 2. Mixing procedure of GP

Method 2

The alkaline activator was mixed one day prior to the sample production. Before starting the mixing process, water was dozed in the alkaline solution. The following mixing procedures were similar to method 1. The difference between this method with method 1 was that water was added to the alkaline at the early stage of the mixing process. In contrast, for method 1, water was mixed with the mixture (dry materials + alkaline) at the middle stage of the mixing process.

Steam Curing

In this study, upon extraction of the specimens from the molds (after 24 hours), a consistent temperature of 90 ± 5 °C was maintained in a hermetically sealed vessel for a duration of six hours during the thermal processing, as seen in Figure 3. After the steam curing, the samples were cooled at room temperature for 2 hours and re-wrapped with plastic. Later on, the samples were again subjected to curing at room temperature until the time of examination. Other samples were directly cured at ambient temperature after demolding and re-wrapping with plastic, which was compared the strength results to the steam-cured samples.

Setting Time

The setting time of fresh GP (including initial and final setting time) was measured according to SNI 03-6827-2002 [18]. The fresh GP was immediately formed into a ball and inserted into the conical ring mold of the Vicat apparatus. After leveling off and putting the glass plate at the base of the conical ring mold, penetration with the needle was started. The initial setting time is reached when the penetration result is greater than or equal to 25 mm, and the final setting time is reached when the needle does not penetrate the specimen.

Figure 3. Steam curing process

Compressive Strength Test and Strength Activity Index

Figure 4 shows the sample testing of the compressive strength of GP. The compressive strength at 7 and 28 days was assessed using the cylinders (55 mm diameter x 11 mm length), which were tested in triplicate. It was compressed using a device with a 150-ton force capacity. To study the effect of cement addition on the compressive strength of GPs, the strength activity index (SAI) was calculated using (1) [19]:

$$SAI = \frac{A}{B} \times 100 \qquad (1)$$

A is the compressive strength of GP containing cement, and B is the compressive strength of GP without cement content.

RESULTS AND DISCUSSION

Setting Time

The setting time test of fresh GP was only performed for method 2 because of the agglomeration of fly ash and cement in method 1, which obstructed the penetration of the needle. The result of the setting time is shown in Figure 5. In general, adding cement accelerated the initial and final setting time. An additional 10% of cement speeded up the setting time by about 85% compared to the reference mixture. At the same time, the rate of setting time at 20% cement substitution increased nearly 95% in comparison to that of the control mixture. The addition of cement content also increased the calcium oxide content (CaO), leading to acceleration of the setting time. This CaO content is the reason for this phenomenon regarding the rapid setting time of GP-containing cement. The use of 100% class C fly ash generated 315 min and 615 min of initial and final setting time, respectively, which was relatively faster than the result obtained by [20], who used class F fly ash in their research. Comparing with other literature using also class C fly ash, Wardhono et al. [4] obtained the initial and final setting time about 1.4 hours and 3 hours, respectively.

Figure. 4. Compressive strength test of GP

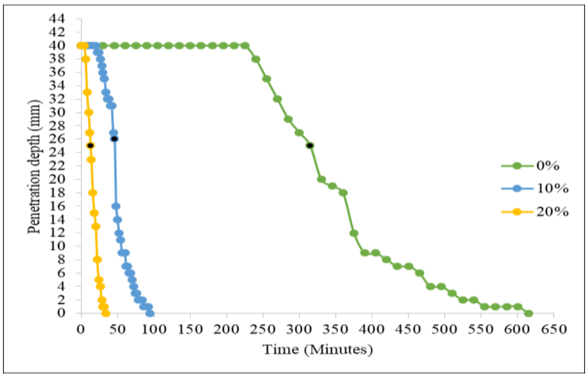

Figure 5. Setting time of fresh GPs containing cement (IST = Initial setting time; FST = Final setting time)

For the geopolymer concrete containing 100% fly ash, which is 3 times faster than the results presented. It seems that the use of method 2 in mixing process retards the setting time of GP containing 100% C type flash ash.

Compressive Strength Test and Strength Activity Index

The results of the compressive strength of GP under ambient curing are shown in Figure 6. Overall, using cement as fly ash substitution decreased the compressive strength of GP. However, method 2 slightly reduced the compressive strength of GPs containing cement compared to method 1 as observed in Figure 6(a) and (b). It seems that additional water at the early stage in the mixing process generated excellent polymerization at longer curing periods in comparison to that of method 1. The polymerization reaction is a chemical bond among Al$_2$O$_3$-SiO$_2$-Alkaline. When water was added to the alkaline solution at the early stage in the mixing process (method 2), the base solid was dissolved, and the fly ash was still bound to generate calcium-aluminate-silicate-hydrate gel (CASH gel). However, water that was mixed in the middle of the mixing process (method 1) was less beneficial to the polymerization, and it potentially created agglomeration of fly ash and cement in the polymer matrix, as depicted in Figure 8(a), causing to decrease the compressive strength as seen in Figure 6(a). Whereas, applying method 2 in the mixing process seems beneficial as no agglomeration is detected in the specimen, as shown in Figure 8(b), leading to an increase the compressive strength as shown in Figure 6(b). Comparing these results presented with the literature gives the impression that utilizing class C fly ash-based geopolymer exhibits lower compressive strength compared to class F fly ash-based geopolymer, as reported by [21, 22, 23]. The key factor of geopolymer is SiO$_2$

content which is provided more in class F fly ash. However, a higher content of CaO in class C fly ash will speed up the setting time, giving an advantage in building construction efficiency, especially for construction repair, precast construction, pavement, highways, etc. In addition, the compressive strength obtained in these results, especially for 0% and 10% of method 1 and 0%, 10%, and 20% of method 2 at 28 days of curing, meets the standard of structural application.

From Figure 7, the results of the strength activity index (SAI) of GP containing cement as fly ash substitution. Generally, the SAI of geopolymer containing cement increased at early curing periods for methods 1 and 2, except for 20% cement using method 1. The highest SAI is achieved for 10% cement at 7 days using method 2, which increased by about 42% compared to the control mixture. For the later curing periods, the SAI of geopolymer with cement decreased was lower than 100%. The effect of cement content is only for the early ages, while for the later periods is minor. The higher content of CaO in cement has a positive impact on setting time, as stated in the previous section.

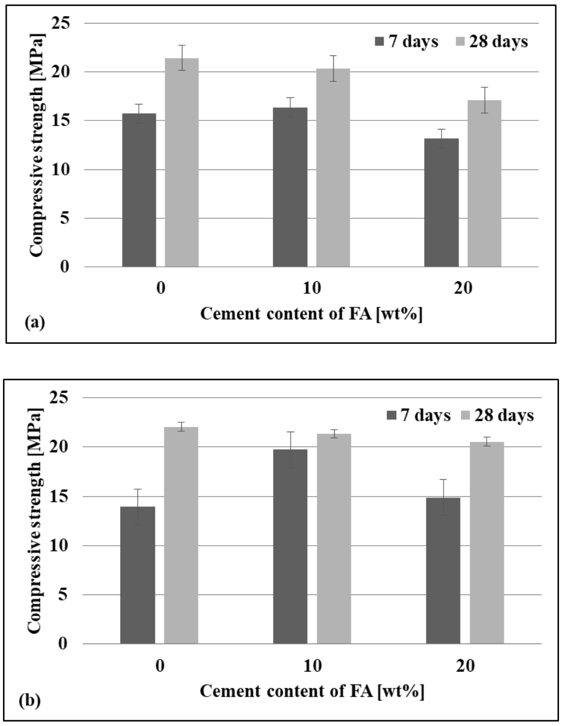

Figure 6. Compressive strength result of GP: (a) method 1; (b) method 2

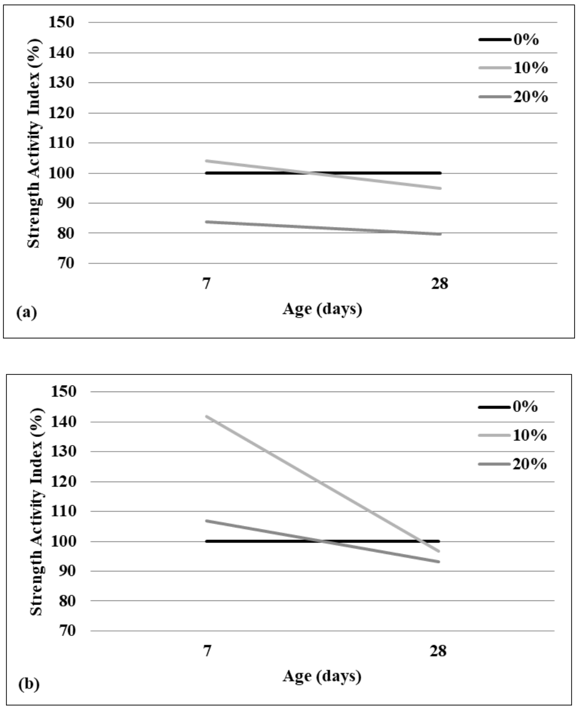

Figure 7. Strength activity index of GP: (a) method 1; (b) method 2

a. Method 1 b. Method 2

Figure 8. Specimens of GP after compressive strength test

Effect of Steam Curing

The result of the compressive strength of GPs made with method 1 and method 2 under steam curing is presented in Figure 9. In general, the compressive strength of GP made with method 2 is higher than that of GP mixed with method 1. The fact that the effect of steam curing is low for method 1 as the agglomeration of fly ash and cement already exists in polymer paste specimen as represented in Figure 8(a). Other literature found a dissolution of kaolinite due to the mixing process, resulting in a complete reaction to form geopolymer gel [24], which corresponds to the result presented. The use of steam curing increased the compressive strength of 0% (100% fly ash) at the early days. It can be observed that the strength increased by about

26% and 36% for method 1 and method 2, respectively.

In contrast, the negative effect of steam curing is noticed for GPs with 10% and 20% cement mixed by method 1, which decreased the compressive strength of GP, as seen in Figure 8. In literatures, the positive effect on the strength of geopolymer is noticed after applying heat curing oven method [11]–[13], which is similarly obtained in the result presented. Thermal processing speeds up the dispersion of silica and alumina from the starting substances, subsequently facilitating the polycondensation procedure [13].

Figure 10 shows the (SAI) of GP mixed by methods 1 and 2 under steam curing. It can be observed that the (SAI) of GPs mixed method 2 in the early days with 10% and 20% cement is higher than 0% under steam curing, as seen in Figure 10(b). The latter is not the case for the GPs with 10% and 20% cement mixed method 1, showing lower (SAI) compared to 0% after subjecting to steam curing at 7 days as seen in Figure 10(a). The SAI of GP mixed method 1 increased at 28 days to reach about 88% and 82% for 10% and 20% cement content, respectively, as shown in Figure 10(a). The decrease in SAI is noted for the GP 10% and 20% cement under steam curing at 28 days, which is 99% and 87%, respectively.

It seems that the use of cement gives a positive effect in strength enhancement at early days of curing, while for longer curing times the strength development still continues, which might be observed at 90 days of curing. In addition, GP containing greater amounts of cement exhibits a more significant impact on setting time as opposed to GP with lesser amounts of cement. To research steam curing's effects on the microstructure of GPs, scanning electron microscope (SEM) was used. In addition, back-scattered electron (BSE) imaging was also applied in this study since this method can detect more features, including pores and cracks as mentioned in the previous study [25]. 0% GP at 28 days mixed by method 2 was selected to study their microstructures by applying SEM-BSE method. Figure 11(a) appears that more cracks and small pores are detected in the microstructure of 0% GPs under ambient curing mixed by method 2. In contrast, applying steam curing to the 0% GPs incorporated by method 2 creates the GPs denser with fewer cracks and small pores as shown in Figure 11(b).

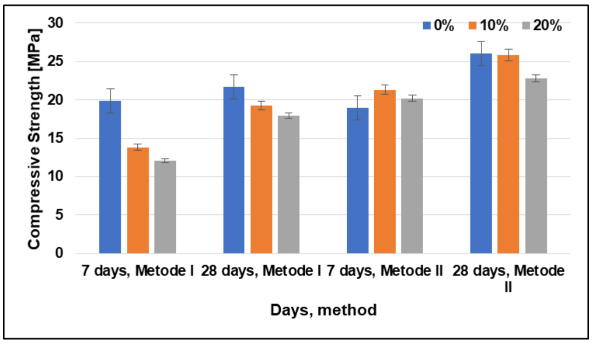

Figure 9. Compressive strength result of method 1 and method 2 of GP under steam curing

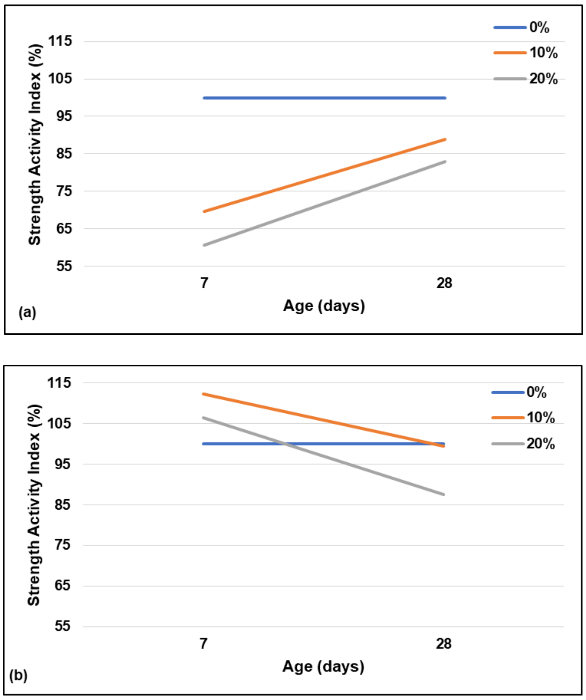

Figure 10. Strength activity index of GP under steam curing: (a) method 1; (b) method 2

Steam curing could accelerate the production of calcium-aluminate-silicate-hydrate gel (CASH gel) to fill the small pores and cracks. This is the reason for the higher strength obtained of GP under steam curing as seen in Figure 9 in comparison to that of GP under ambient curing as seen in Figure 6. This finding is in agreement with the finding of [13, 26, 27], who also found GP with fewer cracks and voids after applying steam curing.

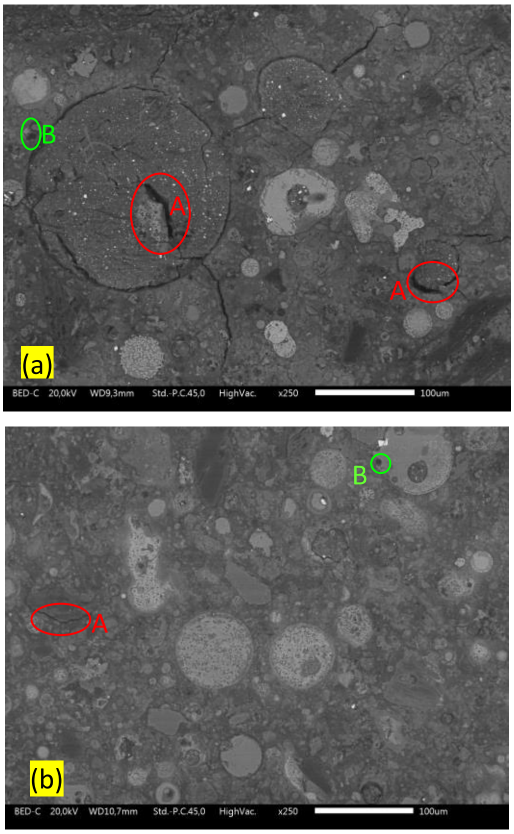

Figure 11. SEM-BSE image at 250 x magnification of 0% method 2: (a) ambient curing; (b) steam curing (A = cracks; B = small pores)

CONCLUSION

An investigation of Class C fly ash-cement-based GP is presented. The following conclusions can be made:

1. The setting time of GP containing 100% C type fly ash is retarded by the use of procedure 2 in the mixing process.
2. An addition of cement content accelerated the setting time of GP. This is caused by the higher amount of CaO content in cement, which influences the setting time.
3. The use of method 2 in the mixing process exhibits higher compressive strength of GP compared to method 1. An agglomeration of fly ash and cement, which was formed by applying method 1 decreased the compressive strength of the geopolymer.
4. The effects of cement content on the strength activity index are only for the early ages of GP.
5. Steam curing increased the compressive strength of GPs in early days of curing.
6. The use of steam curing makes the GP containing 0% cement mixed by method 2 denser with fewer cracks and small pores.

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