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THE EFFECT OF SEASHELL WASTE ON SETTING AND STRENGTH PROPERTIES OF CLASS C FLY ASH GEOPOLYMER CONCRETE CURED AT AMBIENT TEMPERATURE

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Abstract

The main issue of fly ash-based geopolymer concrete is the need for heat curing treatment. The use of a material with a high calcium content is assumed to be able to resolve this problem. This paper reports the effect of seashell waste on setting time and strength properties of Class C fly ash geopolymer concrete cured at ambient temperature. Geopolymer concrete was prepared by using high calcium Class C fly ash activated by activator solution. A proportion of seashell waste was added with fly ash to accelerate the curing at ambient temperature instead of using heat curing. The setting time and strength properties were calculated by Vicat, slump, and compressive strength tests. The result shows that the highest compressive strength was achieved by geopolymer concrete with 10% seashell waste addition and exhibited a comparable strength to that normal concrete. The 10% seashell waste addition also increased the setting time, which affected the strength of geopolymer concrete. It can be suggested that the seashell waste inclusion with Class C fly ash improves the performance of geopolymer cured at ambient temperature.

Keywords: Ambient temperature, Class C fly ash, Geopolymer concrete, Seashell waste, Setting time, Strength.

1. Introduction

The increase of the world population is followed by an increase in the construction industry. This has resulted in increased demand for construction materials, especially in the use of Portland cement (PC). However, the increasing demand for PC has brought to environmental problems related to CO_2 gas emissions, which led to global warming issues. The production of 1 ton PC also produces approximately 0.7-1 ton of CO_2 gas with PC production alone contributes about 6% of CO_2 emissions [1-4].

The use of fly ash, a by-product material of coal combustion process in power plant, is an alternative to resolve this problem [5]. Recent researches show that fly ash can be used as 100% cement replacement material by activating it with alkali activator [6-8]. However, the main issue of Class F fly ash is the need for high temperatures to achieve its structural integrity due to the low Ca content in fly ash [9, 10]. Generally, Class F fly ash-based geopolymer concretes perform slower strength development and longer setting time at ambient temperature. It requires high-temperature treatment to achieve its structural integrity [11]. According to Temuujin et al. [12], this temperature issue can be overcome by adding calcium compound as it can produce calcium silicate hydrate (C-S-H), which can be cured at ambient temperature. In addition, according to Guo et al. [13], the use of fly ash types with high Ca content as the main material of fly ash-based geopolymer also a solution to cope with the high-temperature problem. The authors found that the main geopolymeric gel and C-S-H gel co-exist and bond some remaining fly ash spheres. Further, Wardhono et al. [14] found that the addition of 50% slag with high Ca content on Class F fly ash/alkali-activated slag mortar blends could provide a solution for high-temperature problem during the curing process of Class F fly ash geopolymer.

The use of seashell waste as a material addition is also an alternative to solve the temperature issue in fly ash-based geopolymer material due to high calcium content. The amount of seashell is available abundantly along with Indonesia coastal areas. Based on the Ministry of Marine and Fisheries Indonesia report in 2007 [15], the total production of a seashell was approximately 65,266 tons. This activity produces thousands of tons of empty seashell (seashell by-product) to be discharged, as these are considered as waste. Further, in a study conducted by Othman et al. [16], the ground cockle seashell has approximately 97% by weight of $CaCO_{3,}$ which is suitable for additional material in geopolymer specimen.

This paper reports an experimental study to investigate the effect of seashell waste on strength properties and setting time of high calcium Class C fly ash geopolymer concrete activated by activator solution. A proportion of seashell waste was added with Class C fly ash to accelerate the curing at ambient temperature instead of using a heat curing treatment. The strength and setting time properties were determined by compressive strength, Vicat and slump tests.

2. Materials and Mix Proportions

2.1. Fly ash

The primary material for geopolymer concrete was fly ash with high Ca and Fe content with a specific gravity of 2.58. It was obtained from Paiton coal-fired power plant, which is located in Indonesia. The chemical composition of fly ash was determined by XRF test on PANalytical Minipal 4. The chemical breakdown of fly ash is listed in Table 1.

The fly ash contains CaO of 12.84% (> 10%), the sum of SiO_2, Al_2O_3, and Fe_2O_3 of 81.87% (> 50%). It is categorized as Class C fly ash in accordance with ASTM C618 [17]. The high ferrite content in fly ash was affected by the source of coal. Further, the fineness of fly ash was determined by the sieve no. 200 (0.074 mm mesh).

Table 1. Chemical breakdown of Fly Ash (FA).

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	SO_3
Fly ash	18.13	4.77	58.97	12.84	0.69	0.92

2.2. Seashell powder

The seashell powder was obtained from seashell wastes in Indonesia. It had a high CaO content. The seashell was prepared by grinding and pulverizing it until passed a 0.149 mm mesh (sieve no.100). The chemical composition of a seashell is displayed in Table 2.

Table 2. Chemical breakdown of seashell powder.

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	SO_3
Seashell powder	2.37	0.00	2.61	91.86	0.27	0.00

2.3. Activator solution

A mixture of sodium silicate and 10 M NaOH was used as geopolymer alkaline activators. The sodium silicate was obtained from commercially available sources with the ratio of SiO_2 to Na_2O was 3.30. The 10 M NaOH was prepared by dissolving 400 gram NaOH pellets with 1-litre de-ionized water. A ratio of NaOH to sodium silicate of 0.67 was applied according to previous research [18].

2.4. Mix proportions

Table 3 refers to the detail of Class C fly ash geopolymer concrete (CFGC) mix proportions. Four CFGC and one PC concrete mix designs were investigated, designated CFGC1, CFGC2, CFGC3, CFGC4, and Control. The mix design or CFGC was developed from previous research [15].

Table 3. Mixture proportions of CFGC specimens.

Mixture	PC	Fly Ash	Sea shell	Aggregate		Sodium Silicate	NaOH	Water
				Fine	Coarse			
Control	1.0	-	-	1.63	3.02	-	-	0.40
CFGC1	-	1.0	0.0	1.63	3.02	0.24	0.16	-
CFGC2	-	0.9	0.1	1.63	3.02	0.24	0.16	-
CFGC3	-	0.7	0.3	1.63	3.02	0.24	0.16	-
CFGC4	-	0.5	0.5	1.63	3.02	0.24	0.16	-

The total aggregate (fine and coarse) in geopolymer concrete was kept to 76% by volume for all mixes, while the total weight of binder (fly ash, seashell, and activator solution) was maintained to 24%. The fineness modulus of fine and coarse aggregates was 2.29 and 4.98, respectively. Due to a solid content on activator solution, a water to solid (w/s) ratio of 0.23 was used rather than water to binder ratio (w/b). The water quantity in the CFGC mix was determined from the total sum of water content in the sodium silicate and NaOH. The quantity of solid was calculated as the mass of fly ash and the solid content of activator solution according to previous research [19].

3. Experimental Procedure

3.1. Specimen preparation

The mixing of CFGC specimens was carried out using a concrete mixer. The mixture was poured into a 100 mm × 200 mm cylindrical molds and vibrated. The CFGC specimens were left at room temperature for 24 hours. The specimens were then demolded and cured at ambient or room temperature at 25 ^{o}C - 30 ^{o}C with humidity of 40% - 50% prior testing due to the high Ca content of the fly ash raw material.

3.2. Specimen testing

The compressive strength of CFGC specimens were determined in accordance with ASTM C39 [20]. The CFGC specimens were tested at the age of 3, 7, 14, 21 and 28 days after casting. Three CFGC cylinders were tested for each data point. The compressive strength test was considered completed until the collapse of CFGC specimens.

The setting time performance of CFGC specimens was carried out by depth penetration test (Vicat test) in accordance with ASTM C191 [21]. This test was done to investigate the effect of seashell addition on the setting time of CFGC paste. Slump test value using Abrams Cone was also carried out to identify the workability performance of CFGC specimens. The test was completed in accordance with ASTM C143 [22].

4. Results and Discussion

4.1. Setting time and workability

The depth penetration test result on the effect of seashell addition on setting time of CFGC are given in Fig. 1. The test was conducted in accordance with ASTM Standard [21] and carried out at ambient temperature. The results demonstrate that the setting time of normal concrete (control specimen) was 6 hours at 40 mm depth penetration, while CFGC setting time was 3 hours 30 minutes (CFGC1 with 0% seashell).

The addition of seashell slightly affected the setting time of CFGC1 specimen. CFGC2 with 10% seashell addition exhibited a 0.5 hour faster setting time than CFGC1 with the setting time of 3 hours. It was attributed to the high Ca content in seashell raw material. The angular shape of seashell waste compared to the spherical shape of Class C fly ash particles is also one factor that shorten the setting time rate of fly ash/seashell geopolymer paste to set. Similar finding was also found by Deb et al. [18] by using slag material as addition material. According to the authors, this is mainly because of the accelerated reaction of the calcium and the angular shape of the slag as compared to the spherical shape of fly ash particles. The addition of slag increased the rate of setting time of geopolymer specimen at ambient temperature [15].

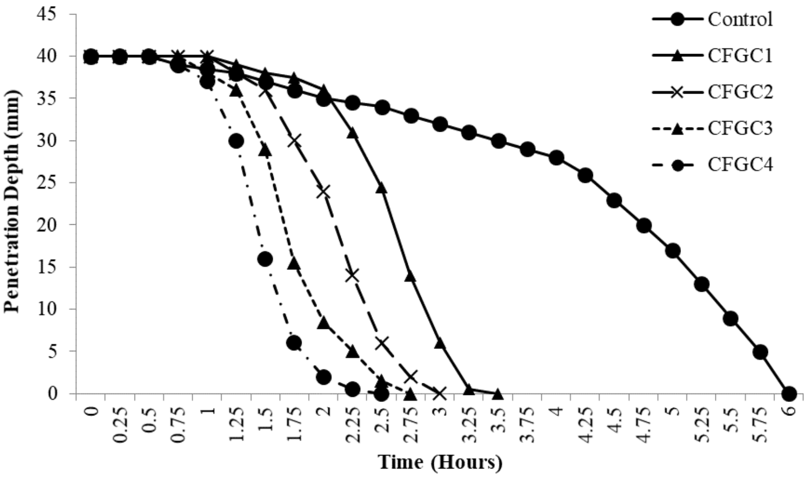

Fig. 1. Depth penetration test (Vicat) of CFGC specimens.

Figure 1 shows that CFGC has a slower early setting time, which occurred at 1st hours compared to that PC setting time at 0.5th hours. This might be attributed to the high Ca compound of seashell addition in CFGC specimens. According to Feng et al. [23] and Mindess et al. [24], the final reaction products of high calcium material, such as slag, are similar to that cement hydration products with the main difference is on the reaction rate at early age. According to the authors, the addition of high calcium material will cause two reactions model in geopolymer, i.e., alkali-activation and fly ash geopolymer reactions. This result a slower setting time of CFGC specimens at early age compared to PC, as shown in Fig. 1 from 0 hours to 1 hours. However, the high Ca content from the Class C fly ash and seashell addition results a rapid setting time, which results a faster setting time of CFGC specimens to that PC after the first reaction occurred. Moreover, further seashell inclusion significantly increased the setting time rate of fly ash-based geopolymer. The setting time of CFGC3 (30% seashell) was 2 hours 45 minutes and getting faster to 2.5 hours for CFGC4 at 50% seashell addition.

Figure 2 shows the slump test value of CFGC specimens measured by slump test using Abrams Cone in accordance with ASTM C143 [22]. The slump test result shows that the standard slump test was not appropriate to measure the workability of CFGC specimens. The liquid characteristic of CFGC1 (0% seashell addition) results in a collapse with the slump value of 21.50 cm. According to Atis et al. [25] and Bouzoubaa et al. [26], the spherical shape of fly ash particles and the lubricant effect of sodium silicate play the important role towards the high flowability and high slump results for the CFGC specimens.

Despite CFGC exhibits a high value of slump test, the workability of CFGC were affected by the addition of seashell material. The addition of 10% seashell (CFGC2) lowers the slump test value of CFGC specimens from 21.50 cm to 19.50 cm.

Further seashell addition significantly decreased the slump test value by approximately 22%. CFGC3 (30% seashell addition) showed a decrease in slump test value from 19.50 cm to 15.33 cm, and continued to decrease to 11.67 cm in the addition of 50% seashell as shown by CFGC4 specimen.

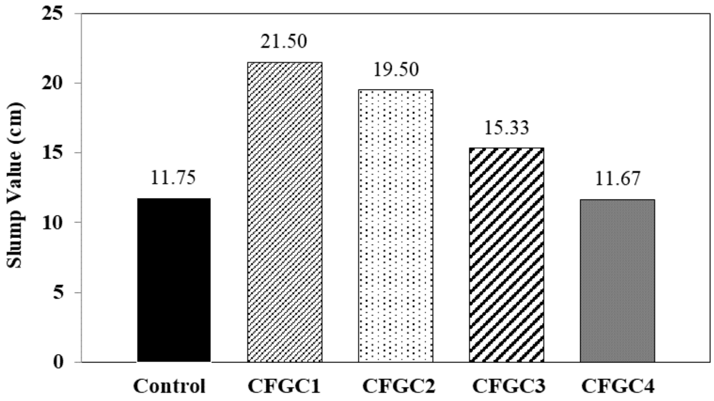

Fig. 2. Slump test value of CFGC specimens.

4.2. Strength development

The results of compressive strength test reported for the CFGC for all mixes are shown in Table 4 and Fig. 3. The CFGC specimens exhibit a low strength at early age (3 days) after casting. All mixes show a compressive strength value under 10 MPa at early age, except for CFGC2 with the highest compressive strength of 10.25 MPa. The CFGC1 with 0% seashell addition demonstrates the worst performance. It failed to be tested due to the soft structural integrity of the CFGC specimen. However, all mixes exhibit a significant improvement of compressive strength up to 28 days.

Table 4. Compressive strength of CFGC specimens.

Mixture	Compressive strength (MPa)				
	3 days	7 days	14 days	21 days	28 days
Control	6.44	9.10	13.56	17.86	21.54
CFGC1	Failed	10.79	15.85	20.26	21.85
CFGC2	10.25	12.67	16.62	21.18	23.91
CFGC3	7.68	10.48	14.33	19.88	20.94
CFGC4	5.71	9.45	13.35	18.70	19.51

The highest initial strength was achieved by CFGC2 (10% seashell addition) with a strength of 10.25 MPa at 3 days (42.87% final strength at 28 days). It demonstrates a significant increase from 7 (strength of 12.67 MPa) to 28 days with the final strength of 23.91 MPa.

This might attribute to Ca content in seashell material in accordance with the previous finding in setting time and workability. CFGC2 also demonstrates a slightly higher compressive strength (23.91 MPa) compared to that PC control (21.54 MPa) at 28 days. In addition, despite CFGC1 failed to achieve its structural integrity at an early age, it shows a comparable strength to that PC control at final age. In general, all mixes display a significant increase with time from 7 to 28 days.

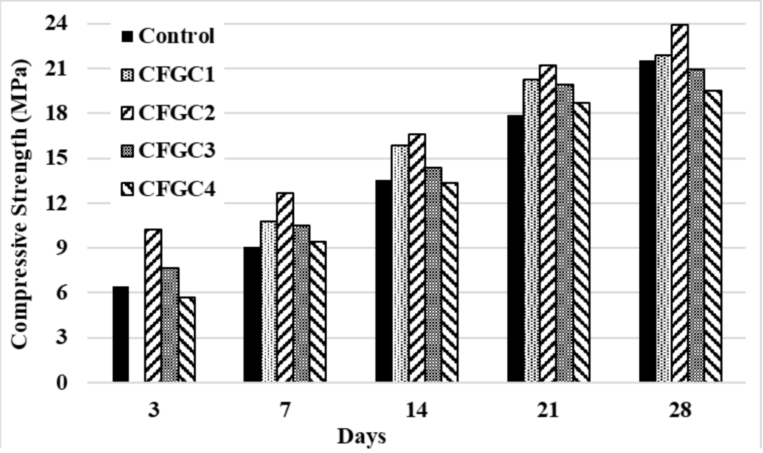

Fig. 3. Compressive strength test results of CFGC specimens.

The development of CFGC strength due to the seashell inclusion are shown in Fig. 3. All of the CFGC mixtures show a strength increased throughout 28 days. The highest increase in strength was achieved by CFGC2 with 10% seashell inclusion with an increase of 11.24 MPa from 12.67 MPa (7 days) to 23.91 MPa at 28 days. The strength development also observed in CFGC3 (30% seashell) and CFGC4 (50% seashell) with an increase of 10.46 MPa and 10.06 MPa from 7 to 28 days, respectively. According to Diaz et al. [27], the high strength of fly ash-based geopolymer is attributed to the Ca content. The high Ca content forms the formation of C-S-H compounds, which affects the compressive strength and the setting rate of fly ash-based geopolymer and increase the strength of geopolymer specimens. Further, according to Temujiin et al. [12], the addition of Ca compounds improves the dissolution reaction of fly ash in alkaline. This reaction results in precipitation C-S-H or C-S-A-H phases and increase the strength properties of geopolymer.

However, the addition of seashell of more than 10% significantly reduce the strength of CFGC. Figure 4 exhibits the effect of a seashell on the final strength of CFGC at 28 days. It shows a significant reduction of strength in all ages of CFGC3 and CFGC4 specimens. A strength reduction of 12.4% has been demonstrated at CFGC3 specimen from 23.91 MPa (CFGC1 with 10% seashell) to 20.94 MPa at 30% seashell addition. A further decrease in strength was also observed along with the seashell addition. The addition of 50% seashell (CFGC4) significantly decreased the strength performance of CFGC specimens from 23.91 MPa (CFGC1 with 10% seashell) to 19.51 MPa with a total reduction of 18.4% at the age of 28 days. The strength reduction in high Ca inclusion might attribute to the particle size of seashell material (passed a 0.149 mm mesh, sieve no. 100), which was bigger than fly ash particles size (passed a 0.075 mm mesh, sieve no. 200). It causes an uneven distribution of Ca material. It reduces the ability to fill the gap between the aggregates, thus increasing the pore network connectivity between the aggregates and the geopolymer paste. According to Sinsiri et al. [28], the strength of fly ash geopolymer is influenced by the fineness of its constituent material, with the highest fineness of material filling the crack between the aggregates. High Ca inclusion will significantly increase this crack, which leads to the strength reduction on CFGC3 and CFGC4 with seashell addition of 30% and 50%, respectively.

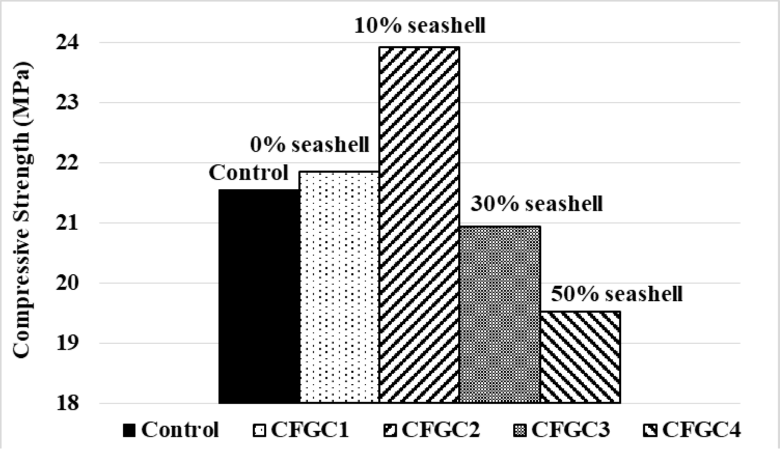

Fig. 4. Compressive strength test of CFGC specimens at 28 days.

In addition, the addition of seashell on fly ash with high Ca content (Class C fly ash) tend to result in unreacted Ca compound. The polymerization reaction between Si and Al used most of the Si compound in geopolymer specimens to form the geopolymer matrix [29]. The inclusion of seashell (with more than 90% Ca compound) couple with the high Ca compound in Class C fly ash produce unreacted Ca. This unreacted Ca goes through a hydration reaction to form calcium hydroxide compound, which might lead to a concrete expansion and create a crack in geopolymer specimen. Thus, the more seashell inclusion in Class C fly ash/seashell-based geopolymer specimen will provide more Ca compound, which significantly reduces the strong performance as shown in CFGC3 (30% seashell) and CFGC4 (50% seashell). Similar performance was found in previous research using slag inclusion [14]. According to the authors, the inclusion of slag significantly affects the strength development of high Ca fly ash/slag-based geopolymer specimen. However, high slag inclusion has been observed to have strength gain before showing a strength reduction with time. This would suggest that the inclusion of a material with a high Ca compound in appropriate proportions, such as slag or seashell, contribute to the strength gain with time. Furthermore, the use of seashell can provide a solution to the heat curing treatment of geopolymer specimen to achieve its structural integrity at ambient temperature.

5. Conclusions

The depth penetration test (Vicat), slump test value and compressive strength test for CFGC specimens were investigated experimentally for 28 days. The main conclusions that can be drawn from this research are given below.

- The setting time rate of high Ca fly ash geopolymer was significantly affected by seashell inclusion indicated by an increase in the setting time rate of CFGC specimens. The setting time rate accelerated from 3.5 hours (0% seashell) to 2.5 hours at 50% seashell inclusion.
- The addition of seashell to high Ca fly ash/seashell geopolymer significantly improved the workability of CFGC specimens. It was indicated by lowering the slump test value along with the increase of seashell addition.

- The inclusion of seashell on high Ca fly ash geopolymer concrete can resolve the requirement of heat curing of geopolymer concrete. The Class C fly ash/seashell geopolymer concrete can be produced at ambient temperature.
- The highest strength was achieved by the CFGC2 with 10% seashell addition and exhibited a higher strength value to that PC concrete at 28 days. However, the addition of more than 30% seashell tends to reduce the strength development throughout 28 days.
- The fly ash/seashell geopolymer specimen can be produced at ambient temperature due to the high Ca compound in seashell material. Thus, it can overcome the heat curing treatment issue on fly ash geopolymer concrete production.

Nomenclatures

Al	Aluminate
Al_2O_3	Aluminium oxide
Ca	Calcium
$CaCO_3$	Calcium carbonate
CaO	Calcium oxide
CO_2	Carbon dioxide
Fe	Iron
Fe_2O_3	Iron (III) oxide
K_2O	Potassium oxide
M	Molar
NaOH	Sodium hydroxide
Na_2O	Sodium oxide
Si	Silicate
SiO_2	Silicate oxide
SO_3	Sulfur trioxide

Abbreviations

ASTM	American Society for Testing Materials
CFGC	Class C Fly Ash Geopolymer Concrete
C-S-A-H	Calcium Silicate Aluminate Hydrate
C-S-H	Calcium Silicate Hydrate
FA	Fly Ash
PC	Portland Cement
w/b	Water to Binder Ratio
w/s	Water to Solid Ratio
XRF	X-Ray Fluorescence

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