

## Research Article

# A Study on the Influence of Sodium Silicate Concentration and $\text{SiO}_2 : \text{Na}_2\text{O}$ Ratio on the Properties of Low-Calcium Fly Ash-Based Alkali-Activated Materials Cured at Ambient Condition

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Low-calcium Fly ash-based alkali-activated materials (AAM) have some extraordinary properties such as high fire resistance and low shrinkage. However, they have lower strength and high setting time when curing at ambient temperature conditions. Therefore, this research aimed to improve the strength and setting time of low-calcium fly ash-based AAM curing at ambient temperature conditions. The effect of the changes in concentration and modulus of sodium silicate and curing condition of the materials were studied on the properties of the AAM. Fly ash type II, 32% sodium metasilicate pentahydrate, 32 and 56% sodium disilicate solution, 8M sodium hydroxide, and standardized were used. Using 32% sodium disilicate solution significantly improved the flowability of mortar. Moreover, it increased the compressive strength and remarkably decreased the setting time of AAM at ambient temperature curing. The decrease in the concentration of sodium disilicate has a significant influence on the reactivity of fly ash.

## 1. Introduction

Portland cement (PC) concrete has some remarkable properties such as the ability to cast in different shapes and sizes, high compressive strength, and reasonable cost. Moreover, its reinforcement by steel makes it an extraordinary construction material. According to these facts, Portland cement concrete is the most commonly used construction material in the world. Annual global production of ordinary Portland cement (OPC) reaches 4 billion tonnes [1]. But according to the statistics, OPC is responsible for the emission of 8%  $\text{CO}_2$  of the globe [2]. Therefore, they are not environmentally friendly materials and one of the major causes of global warming, which is a serious issue nowadays.

When aluminum silicate materials such as fly ash and slag react with an alkali source, they produce a material that has binding properties [3, 4]. These kinds of materials are called Geopolymer or alkali-activated materials (AAM).

Compared to OPC, AAM is a more environmentally friendly material because they emit a much lower amount of  $\text{CO}_2$  to the atmosphere [5, 6]. Moreover, they contain some by-products as their primary constituents, such as fly ash and slags [7].

Besides being environmentally friendly materials, AAM have some other advantages, such as obtaining high early strength and superior mechanical properties [8, 9]. They have better resistance to fire, acid attack, and alkali-silica reaction [10–12].

Sodium hydroxide (NaOH) and sodium silicate solution (water glass) are the two prominent alkaline materials for the making of AAM [13]. According to the molar ratio ( $\text{MR} = \text{SiO}_2 : \text{Na}_2\text{O}$ ), sodium silicate has several types such as sodium metasilicate ( $\text{MR} = 1$ ) and sodium disilicate ( $\text{MR} = 2$ ) [14]. Conventionally, water glass is used at a concentration of around 50% [9, 15–18], which makes the solution to be viscous and sticky, and this causes AAM to have a workability problem. Sodium silicate solution not only provides an

alkaline environment to dissolve the aluminosilicate precursors, but they are a source of silica and contribute significantly to developing mechanical strength [19].

Low calcium fly ash-based AAM has some extraordinary advantages. They possess geopolymeric characteristics such as low shrinkage and high fire resistance [20], but compared to the slag-based AAM, they have lower strength [18, 21] and longer setting time [16] when cured at ambient temperature conditions. The high setting time of the material is considered one of the major problems. This means that they cannot set or harden well at ambient temperature curing conditions. They sometimes need more than 40 hours to achieve their final setting [16].

Incorporating the slag with fly ash improves the reactivity of the aluminosilicate precursors that result in a decrease in the hardening or setting time of the samples at ambient temperature and modifies some other properties of concrete such as compressive strength, flexural strength, and density of AAM [17]. As the slag is very active and increases the reactivity and hydration of the materials [22], this causes the materials to emit a higher amount of heat during the reaction process [23]. Therefore, the drying shrinkage of alkali-activated materials with slag is much higher than the other types [17, 18].

This research aims to solve the problem related to low-calcium fly ash-based AAM. Therefore, the influence of the concentration and molar ratio of sodium silicate is studied on the setting time, flowability, and compressive strength of AAM.

In the previous experiment [24], while characterizing the one-part mixing method of AAM, granular sodium metasilicate was used with a concentration of 32%. A quick setting of the mortar consisting of 100% low-calcium fly ash was observed. Hence, the role of concentration and types of sodium silicate on the setting time of the AAM is considered for this experiment.

## 2. Methodology

**2.1. Materials.** Fly ash type II was used as an aluminosilicate precursor, the composition and other properties are illustrated in Table 1. Sodium silicate and sodium hydroxide were used as alkali activators (AA). The concentration of NaOH was 8 molar. Sodium silicate: NaOH ratio was set to 1.5 for the paste and mortar samples. According to the  $\text{SiO}_2 : \text{Na}_2\text{O}$  modulus, two types of sodium silicate were used: Sodium metasilicate pentahydrate ( $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ ), which has the modulus of 1, and sodium disilicate ( $\text{Na}_2\text{O} \cdot 2\text{SiO}_2$ ) with the  $\text{SiO}_2 : \text{Na}_2\text{O}$  ratio of around 2. The sodium metasilicate was in the granular solid-state, while sodium disilicate was used in form of solution gel. The granular sodium metasilicate consisted of 28%  $\text{SiO}_2$ , 28%  $\text{Na}_2\text{O}$ , and 54%  $\text{H}_2\text{O}$ , and sodium disilicate aqueous solution was composed of 36%  $\text{SiO}_2$ , 18%  $\text{Na}_2\text{O}$ , and 46%  $\text{H}_2\text{O}$ . Standardized sand conforming JIS R 5201 was used as a fine aggregate for making the mortars.

**2.2. Mix Proportion and Samples Detail.** In this research, 3 types of paste for the setting time test, 3 types of mortar for flowability test, and 5 types of cylindrical sample for compressive strength test were prepared. A sample (GAA) that had

almost the same mix proportion with GA was taken from [25]. Table 2 and Table 3 show the chemical composition and other characteristics of the pastes and mortars, respectively. AA solution was composed of 8M NaOH and sodium silicate with different types and concentrations.

To investigate the effect of the sodium silicate on the setting time, flowability, and compressive strength of the AAM, 3 patterns of sodium silicate solution were utilized. Sodium metasilicate solution with a concentration of 32% and sodium disilicate solution with concentrations of 32% and 54% were prepared. For preparation of AA solution containing sodium disilicate each NaOH and sodium disilicate solution were prepared separately and then mixed and stored. Granular sodium metasilicate pentahydrate was dissolved in NaOH solution to make the AA solution by a water bath shaker according to [24]. As the AA solution containing sodium metasilicate is sensitive in keeping it at a lower or ambient temperature and crystallization can easily occur inside it [24]. Therefore, for better characterization, all types of alkaline solution in this experiment was kept and used at a higher temperature of around 40 °C.

For the characterization of the mortar curing condition, ambient temperature curing and heat curing were used. At ambient temperature conditions, they were cured at around 20°C, but for the others which were heat cured, the samples were put inside incubators at 45°C for 24 hours, and then they were put at the ambient temperature condition until the period of their compressive strength test.

**2.3. Preparation of Paste and Setting Time Test.** Preparation of the paste and setting time test were done according to the JIS R 5201 [26]. For the mixing, a paddle mixer was used. First, fly ash and AA solution were mixed for 1 minute. Then, the mixing was stopped for 90 seconds followed by a 1-minute mixing. Next, they were poured into molds. The pouring procedure was required to be completed within a minute. Then, initial and final setting times of the pastes were tested.

**2.4. Mortar Preparation, Flow, and Compressive Strength Test.** The fly ash was first mixed with the alkaline solution for 1 minute, followed by the addition and mixing of standardized sand for 30 seconds each at low and high speed. Then, the mixing procedure was stopped for 90 seconds, followed by a high-speed mixing for 1 minute.

Then, the mortars were poured into cylindrical molds (50 mm diameter × 100 mm height). All the fresh mortars except samples containing 54% sodium disilicate had enough consistency that did not require a vibrator for the compaction. Samples with a 54% concentration of sodium disilicate were compacted by a vibrator. Then, the samples were sealed with plastic and cured.

Flow test was conducted twice for each mortar, and the procedure was done according to the JIS R 5201 [26]. Cylindrical molds with a diameter of 50 mm and a height of 100 mm were used to study 7, 28, and 56 days' compressive strength. The compressive strength test was also conducted according to JIS R 5201 [26].

TABLE 1: Specific gravity (S.G.), the chemical composition of materials in weight percentage (wt%) by XRF, and chemical analysis of standardized sand.

Materials	Chemical composition (% weight)							SG(g/cc)
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Na <sub>2</sub> O	H <sub>2</sub> O	K <sub>2</sub> O	
Fly ash	55.19	25.35	7.57	4.06				2.33
Standardized sand	98.4	0.41	0.36	0.16	0.01	<0.2	0.01	2.64

TABLE 2: Mix proportion of and detail of each type of paste.

No	Samples	Fly ash (g)	SiO <sub>2</sub> /Na <sub>2</sub> O	32% sodium metasilicate+8M NaOH AA solution (g)	32% sodium disilicate+8M NaOH AA solution (g)	54% sodium disilicate +8M NaOH AA solution (g)	AA solution/fly ash
1	PA	400	2	0	0	166	0.42
3	P1A	400	1	140	0	0	0.35
5	P2A	400	2	0	140	0	0.35

TABLE 3: Mix proportion of and detail of each mortar type.

No	Fresh mortar origin	Cylindrical samples	Fly ash (kg/m <sup>3</sup> )	Sand (kg/m <sup>3</sup> )	SiO <sub>2</sub> : Na <sub>2</sub> O	32% sodium metasilicate+8M NaOH AA Solution (kg/m <sup>3</sup> )	32% sodium disilicate+8M NaOH AA Solution (kg/m <sup>3</sup> )	54 % sodium disilicate +8M NaOH AA Solution (kg/m <sup>3</sup> )	AA solution/fly ash
1	GA	GAO	517.6	1536.2	2	0	0	278.7	0.54
2	G1A	G1AA	517.6	1536.2	1	278.7	0	0	0.54
3		G1AO	517.6	1536.2	1	278.7	0	0	0.54
4	G2A	G2AA	517.6	1536.2	2	0	278.7	0	0.54
5		G2AO	517.6	1536.2	2	0	278.7	0	0.54

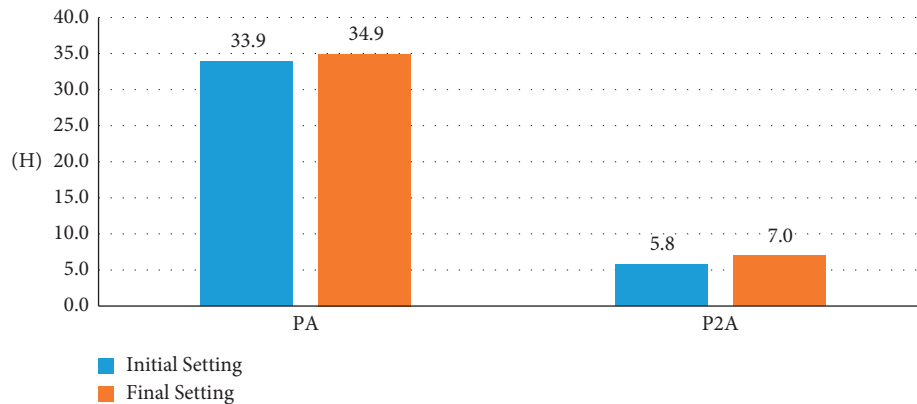


FIGURE 1: Influence of the change in concentration of sodium silicate solution on the setting time of the pastes.

### 3. Result

**3.1. Influence of the Sodium Silicate Concentration.** In this experiment, sodium disilicate solution was used in two concentrations, 32 and 54%. In PA and GA, the concentration of the water glass was 54%, while in P2A and G2A, it was 32%. Conventionally, the fly ash-based AAM is produced by a water glass with a concentration of around 50% and cured at a higher temperature inside an oven or incubator [9, 15, 25].

**3.1.1. Setting Time.** Figure 1 shows the setting time of AAM with different concentrations of sodium silicate. PA, which was made by a conventional method, had 54% water glass in its

composition. The initial and final setting times of the paste were around 35 hours, which were notably high and the result confirmed the previous research [16]. This type of AAM is not applicable on-site. The slow reactivity of low-calcium fly ash in an alkaline environment is the factor behind the delayed setting time. Therefore, they need to be cured at higher temperatures or partially replaced with slag to reduce the setting time, but using slag increase the shrinkage of the material. Interestingly, when the concentration of the water glass was reduced to 32%, there was a significant development in the initial and final setting time of the paste. The initial and final setting times of the P2A were 5.8 and 7 hours, respectively. The increase in the amount of water might improve the reactivity of the fly ash and cause the paste to set quickly.

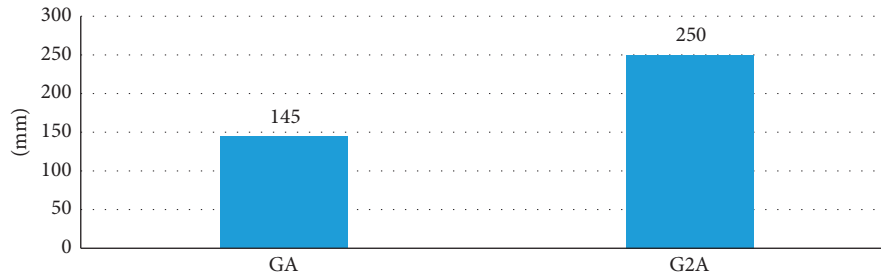


FIGURE 2: Influence of the change in concentration of sodium silicate on the flowability of AAM.

**3.1.2. Flowability.** Figure 2 shows the effect of the sodium silicate concentration on the flow properties of AAM. GA, a mortar that was made of 54% sodium silicate, had a flowability of 145 mm, which was much lower than the 32% sodium silicate concentrated AAM. G2A had a flow value of 250 mm. As mentioned in the introduction section, conventional alkali-activated mortar or concrete has a higher viscosity. This viscosity is caused by the presence of  $\text{SiO}_2$  in the sodium silicate solution. In this research, increasing the water amount in the solution reduced the viscosity and improved the flowability.

**3.1.3. Compressive Strength.** Figure 3 shows the influence of the water glass concentration on the 28-day compressive strength of the mortars. GAA and G2AA were made by 54 and 32% sodium disilicate solution, respectively. Both of them were cured at ambient temperature conditions. In this study, GAA, which had almost the same composition as GA of this experiment, was taken from another research [25]. The result shows that G2AA with 25.8 MPa significantly had higher compressive strength than the GAA. The compressive strength of the GAA was 8 MPa.

Using sodium silicate with the lower concentration decreased the setting time and increased the compressive strength of the AAM in ambient temperature conditions. This indicates that the reactivity of the fly ash was increased with an increase in the amount of water and a decrease in the PH value of the alkaline solution.

### 3.2. Influence of the $\text{SiO}_2 : \text{Na}_2\text{O}$ Ratio

**3.2.1. Setting Time.** Figure 4 shows the influence of the sodium silicate molar ratio ( $\text{SiO}_2 : \text{Na}_2\text{O}$ ) on the initial and final setting times of P1A and P2A samples which were made by sodium metasilicate and sodium disilicate solution, the concentration of both types of sodium silicate was 32%. P1A had an initial setting time of 6.3 hours and a final setting time of 8.4 hours. Both the initial and final setting times of P1A were slightly higher than P2A, which had initial and final setting times of 5.8 and 7 hours, respectively. This indicates that the  $\text{SiO}_2 : \text{Na}_2\text{O}$  ratio of sodium silicate solution with the same concentration does not have any significant influence on the setting properties of low-calcium fly ash-based AAM. The final setting time of both kinds of material was slightly lower than OPC paste. OPC paste has a final setting time of around 10 hours [27]. This means that in terms of the setting

time, both kinds of materials are reasonable for the cast-in-situ condition.

**3.2.2. Flowability.** Same as the setting time test, the change in the  $\text{SiO}_2 : \text{Na}_2\text{O}$  ratio did not have any significant effect on the workability and flow value of both types of AAM. Figure 5 shows that both types, G1A and G2A, had good flow value of 244 mm and 250 mm, respectively.

**3.2.3. Compressive Strength.** Figure 6 shows the influence of the  $\text{SiO}_2 : \text{Na}_2\text{O}$  on the compressive strength of both types of AAM. G1AA had a compressive strength of 4.7 MPa, 6.4 MPa, and 23.2 MPa at 7, 28 and 56 days, respectively. At the same time, G2AA's 7, 28 and 56 days' compressive strength were 4 MPa, 25 MPa, and 46 MPa, respectively. This indicated that both types had almost the same compressive strength at 7 days, but for 28 and 56 days, the compressive strength of the G2AA increased by 403 and 719%, respectively. This revealed that the increase in  $\text{SiO}_2$  content of sodium silicate solution improved the compressive strength remarkably at ambient temperature curing.

This result indicates that with using 32% sodium disilicate, remarkable 28 and 56-day compressive strength can be achieved, which is appropriate for use in the structural field. However, the 7-day compressive was much lower, but that may be increased by a slight increase in the concentration of sodium disilicate or a decrease in the solution; fly ash ratio as the flow value of the mortar was high enough for the reduction.

**3.3. Effect of the Curing Condition on Compressive Strength.** Figure 7 illustrates the effect of heat and ambient curing conditions on the compressive strength of AAM made by different types and concentrations of sodium silicate. The heat curing had a significant influence on the 28-day compressive strength of AAM made by 54% sodium disilicate. Because of this, the conventional type of low-calcium fly ash AAM is cured at a higher temperature. At ambient curing conditions, GAA had a compressive strength of 8 MPa at 28 days, and with the curing at 45 °C for two days, the compressive strength was increased by around 12 times.

Moreover, heat curing had a considerable effect on the 7, 28, and 56-day compressive strength of the AAM made by the 32% sodium metasilicate. When applying the heat curing, the compressive strength of G1A increased by 326%,

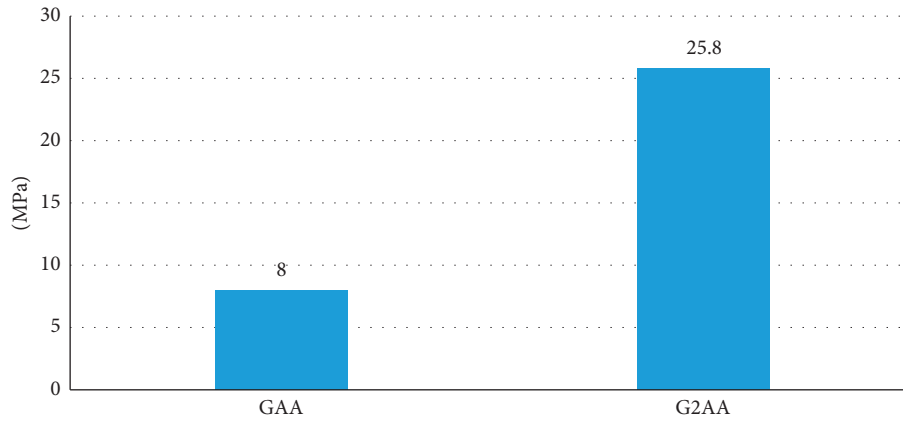


FIGURE 3: Influence of the change in concentration of sodium silicate on the 28-day compressive strength.

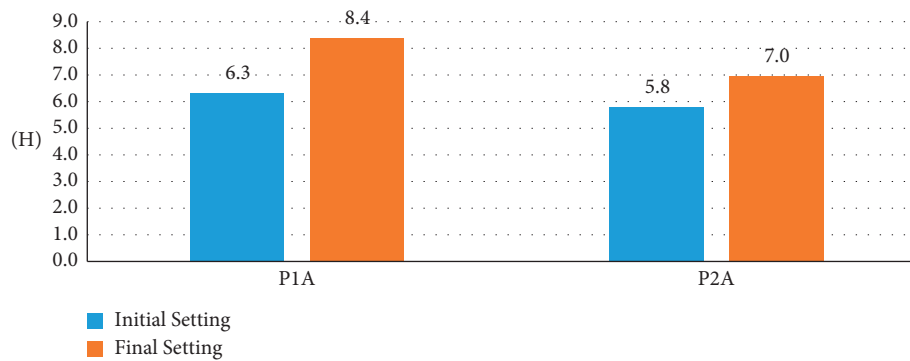


FIGURE 4: Influence of the SiO<sub>2</sub>:Na<sub>2</sub>O ratio on the setting time.

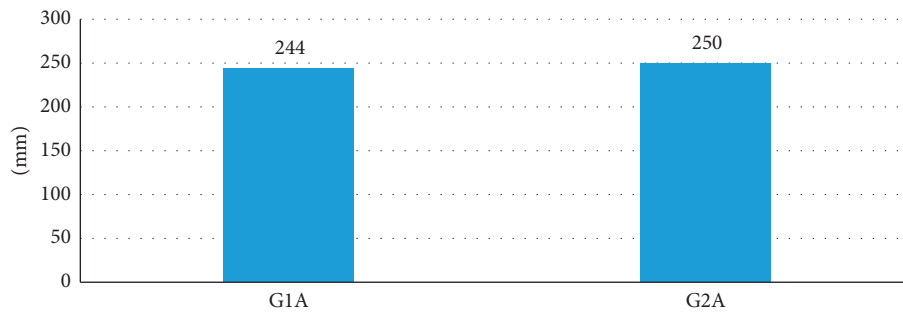


FIGURE 5: Influence of the SiO<sub>2</sub>:Na<sub>2</sub>O ratio on the flowability.

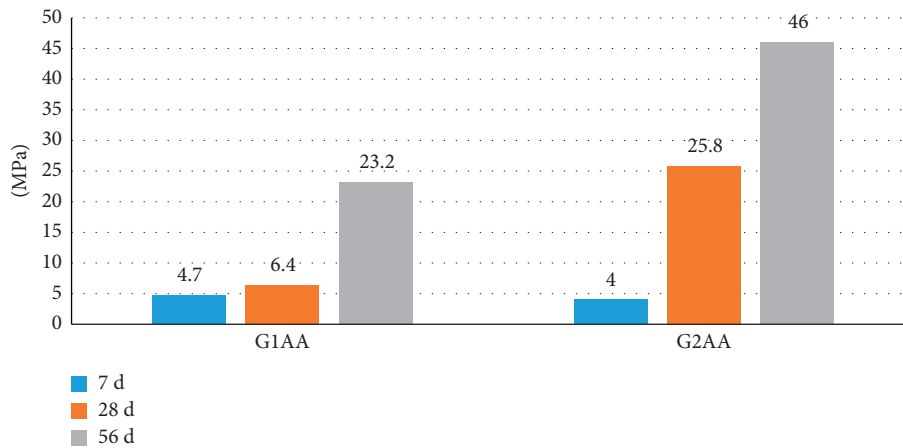


FIGURE 6: Influence of the SiO<sub>2</sub>:Na<sub>2</sub>O ratio on the compressive strength.

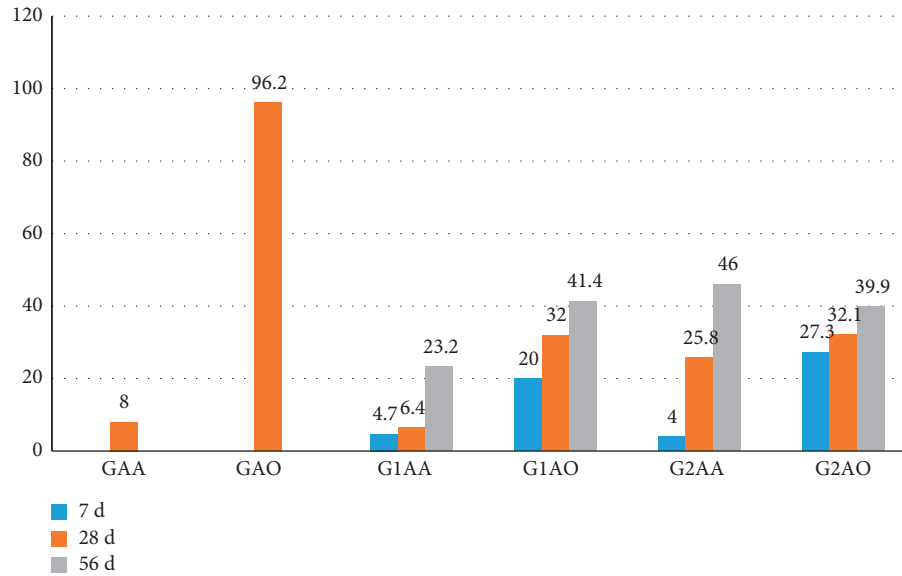


FIGURE 7: Effect of the curing condition on the compressive strength of AAM.

400%, and 78% at 7, 28, and 56 days, respectively. This indicates that heat curing had an excellent influence on the 7 and 28-day compressive strength of the AAM made by the sodium metasilicate.

For G2A mortar, while applying the heat curing, there was a remarkable increase in the 7-day compressive strength. Improvement of 583% was observed in the compressive strength of G2AO at the aforementioned age. At 28 days, there was a slight increase, but at 56 days, the heat curing had a negative influence on the compressive strength. The compressive strength dropped from 46 MPa to 39.9 MPa at 56 days. The result showed that heat curing had a significant influence on the early strength of G2A, but for the later ages, the compressive strength of the samples cured at ambient temperature and incubator had very little difference. This may be due to the higher content of  $\text{SiO}_2$  in the mortar, which helped to achieve its late strength without curing at the higher temperature. As the fly ash in the mortar was already activated by a lower alkaline environment. Therefore, sodium disilicate with a lower concentration than the conventional one will be effective for making low-calcium fly ash-based AAM cured at ambient temperature conditions.

#### 4. Conclusion

Low calcium-fly ash-based AAMs have some extraordinary geopolymeric properties such as high fire resistance and low drying shrinkage when heat curing is applied to them, but when they are cured at ambient temperature, they have some problems of high setting time and low compressive strength. Moreover, the workability problem due to the use of highly concentrated water glass is considered another problem that is existed in the material. Conventionally, fly ash-based-AAM is made by around 50% concentrated sodium silicate solutions.

This research was conducted to solve the aforementioned problems related to the conventional low-calcium fly ash-based AAM cured at ambient temperature conditions. Replacing some percentage of fly ash with slag can solve some of the problems but using slag increases the drying shrinkage of the materials.

Therefore, this research studied the influence of the types and concentrations of the sodium silicate solution on the setting time, flowability, and compressive strength properties of the AAM. These two types of sodium silicate were sodium metasilicate which has a  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio of 1 and sodium disilicate with the modulus of 2. Moreover, the effect of the curing condition was also investigated.

From the study, the following conclusion can be written.

- (1) The concentration of the sodium silicate has a considerable influence on the setting time of the AAM paste. Using 54% sodium silicate or sodium disilicate increases the initial and final setting times to more than 30 hours. However, while reducing the concentration, the initial and final setting time decrease to less than 10 hours.
- (2) Decreasing the concentration of sodium silicate solution increases the flowability of low-calcium fly ash-based AAM.
- (3) Ambient temperature-cured low-calcium fly ash-based AAM using 32% sodium disilicate has a much higher compressive strength than the one made by the 54% AAM.
- (4)  $\text{SiO}_2:\text{Na}_2\text{O}$  ratio of sodium silicate solution does not have a significant influence on the setting time and flowability of the paste and mortar samples, respectively.
- (5) The influence of the  $\text{SiO}_2:\text{Na}_2\text{O}$  is prominent on the compressive strength of the AAM. The compressive strength at 28 and 56 days increases 4 and 2 times, respectively, when sodium disilicate solution is used instead of sodium metasilicate solution.

- (6) For the ambient temperature curing, low-calcium fly ash-based AAM using sodium silicate with a concentration of around 50% has much lower compressive strength at 28 days. But when heat curing is applied, the compressive strength becomes much higher.
- (7) Heat curing has a significant influence on the initial strength of the AAM made by sodium metasilicate and sodium disilicate solution. However, the AAM made by the sodium disilicate solution has higher compressive strength at ambient temperature curing conditions than one cured at a higher temperature.
- (8) Using 32% sodium disilicate for making low-calcium fly ash-based AAM cured at ambient temperature conditions has advantages in terms of setting time, flowability, and compressive strength. However, the 7-day compressive strength is lower, but it may be improved by a slight increase in the concentration of sodium silicate or a reduction in AA solution/fly ash ratio.

### Data Availability

Data are available on request from the author.

### Conflicts of Interest

The author declares that there are no conflicts of interest related to the paper.

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