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The effect of sodium hydroxide molarity on setting time, workability, and compressive strength of fly ash-slag-based geopolymer mortar

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Abstract. This research investigates the influence of sodium hydroxide (SH) molarity on fly ashslag-based geopolymer mortar's workability, setting time, and compressive strength. The molarit[y of SH varies between 1M, 2M](mailto:ernawatisri@staff.uns.ac.id), 4M, 6M, 8M, and 10M. The geopolymer mixture was proportioned to contain 30% slag as a substitution for fly ash, with the alkaline activator to binder (Al/Bi) ratio of 0.45 and the sodium silicate to sodium hydroxide ratio (SS/SH) of 1.5. The results showed that at a range of low molarity SH (1M, 2M, and 4M), the setting time was faster in a mixture containing higher molarities. In contrast, at a range of high molarity (6M, 8M, and 10M), the setting time is slower in a mixture with higher molarity SH. Based on the setting time, the concentration of SH that can be applied in the field is 1M, 2M, 8M, and 10M. Increasing the molarity of SH will decrease the workability but increase compressive strength. However, when SH molarity increased after optimal molarity was established, the compressive strength was reduced. The maximum compressive strength of mortar is 46.86 MPa, obtained at an SH molarity of 6M.

1. Introduction

Cement is commonly utilized as a binder in the production of mortar and concrete. Cement consumption significantly affects the environment because cement manufacture produces enormous carbon dioxide (CO2) emissions. Davidovits [1] stated that to create one ton of Portland cement, around 0.85 to 1 ton of CO² is discharged into the atmosphere. In addition, it was also reported that ordinary Portland cement produces around 1.35 billion tons of $CO₂$ per year and contributes 5–7% of global emissions of $CO₂[2]$. Therefore, alternative binder materials are required as a substitute for cement.

New binding materials have been the subject of significant research in recent years, mainly geopolymers, which are more environmentally friendly than ordinary Portland cement (OPC). Geopolymer is formed when a material with a high silica and alumina content reacts with an alkaline

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activator solution [3]. Geopolymer binder is a 2-component inorganic system, consisting of solid components having $SiO₂$ and $Al₂O₃$, such as fly ash, pozzolan, slag, and alkali activator consisting of alkali hydroxide, silica, alumina, carbon, and sulfate [4]. In addition, geopolymer cement produces 80- 90% fewer carbon emissions than Portland cement [5].

Many researchers have investigated the influence of SH molarity and curing temperature on fly ashbased geopolymer mortar characteristics. In fly ash-based geopolymer mortar, heat curing is required with a temperature varying from 60°C to 100°C. Görhan and Kürklü [6] noted that the highest compressive strength was reached at 6M of SH concentration and high curing temperature (85°C). Chithambaram et al. [7] recognized that the highest compressive strength occurs at an SH molarity of 12M and a curing temperature of 90°C. The requirement of high-temperature curing causes a problem for field applications.

Alternative materials are needed to produce geopolymer mortar without requiring high-temperature curing. Ground granulated blast furnace slag (slag) is one of the materials that can be utilized in this case. Research on the addition of slag in fly ash-based geopolymer mortar shows that even in small quantities, slag can replace high-temperature curin [8]. Using precursors with high calcium oxide (CaO) content in concrete cured at room temperature provides higher strength, reduces setting time, and improves the mechanical characteristics of geopolymer concrete [9]. Geopolymer mortar with high slag volume results in fast setting time and high initial strength so that it can be considered as a substitute for mortar for rapid rehabilitation materials in construction [10].

In addition to the presence of slag as a fly ash replacement, the most influential factor on the characteristics of geopolymer mortar is the concentration of alkaline activator. With the molarity of the SH solution increasing, the compressive strength also increases. Using SH with high molarity results in higher binder dissolution and accelerates the geopolymerization reaction [11]. Although the workability is decreased, increasing SH's molarity improves the mortar's compressive strength. The SH molarity of 12M produces the greatest compressive strength [12]. The use of 8M-14M SH molarity and slag as a substitute for 0%-40% fly ash obtained the highest compressive strength of mortar at 30% slag content with an SH solution molarity of 12M [7].

Research on fly ash-slag-based geopolymer mortar is mostly found using a medium-high concentration of SH solution [7], [11], [12]. Meanwhile, the most significant $CO₂$ gas emissions in the geopolymer mortar manufacturing process are from the production of alkaline activator solution [13]– [16]. In this study, variations in SH solution from low to medium molarity were used, which are 1M, 2M, 4M, 6M, 8M, and 10M. Moreover, this study will investigate how the molarity SH affects the workability, setting time, and compressive strength of geopolymer mortar. The results can be used to guide in proportioning fly ash-slag-based geopolymer mortar for field applications and are environmentally friendly.

2. Experimental

2.1. Materials

Fly ash-slag-based geopolymer mortar was made from a mixture of fine aggregate (sand), a precursor consisting of fly ash and slag, and an alkali activator with a specific ratio. The aluminosilicate material used in this study was class F fly ash from PLTU Tanjung Jati B, Jepara, Indonesia, according to ASTM C618. Meanwhile, slag as a substitute for fly ash was obtained from PT. Krakatau Semen Indonesia, Cilegon, West Java, Indonesia. The fly ash has a diameter of 75 μm or passes the No. 200 sieve. The chemical content of fly ash and slag was determined using an X-ray fluorescence (XRF) test, with the results shown in table 1. The total SiO2, Al2O3, and Fe2O3 content of fly ash was 83.83% (>70%), and the calcium oxide content was 8.64% (<10%), categorized as fly ash of class F. The CaO content of slag is categorized as very high, at 62.1%.

This research used an alkaline activator mixture of SS and SH. Sodium Silicate solution is a gel type with the production code BE-58 obtained from a chemical store. The SH solution was made by dissolving the SH flakes with distilled water according to the specified molarity. Fine aggregate using

sand from Kulon Progo, Yogyakarta, Indonesia, passed the No. 8 sieve with a diameter of 2.36 mm. The sand used has passed the material test and is suitable for use as a geopolymer mortar material%.

2.2. Methods

2.2.1. Mix Proportions, Mixing, and Curing. The mix design used to determine the proportion of the mixture is based on the weight ratio. The ratio of the binder to sand is 1 binder:2 sand. The independent variables in the mix design are SH molarity of 1M, 2M, 4M, 6M, 8M, and 10M. In this research, the alkali activator to binder (Al/Bi) ratio was 0.45, the ratio of SS/SH was 1.5, and the percentage of slag as a substitute for fly ash was 30%. The proportions of the complete mixture and the requirement of NaOH flakes for each concentration of SH solution are shown in table 2 and table 3.

Description	Material Requirement Per \mathbf{M}^3
The density of mortar $(kg/m3)$	2400
Slag percentage $(\%)$	30
Al/Bi	0.45
SS/SH	1.5
Sand (kg)	1600
Fly ash (kg)	560
Slag (kg)	240
Sodium silicate (kg)	216
Sodium hydroxide (kg)	144

Table 2. Details of mixed proportions of mortar

The solution of SH was prepared 24 hours before being mixed with sodium silicate. Mixing of alkaline activator solutions (SS and SH) was carried out 30 minutes before mixing with other ingredients. While waiting for the alkaline activator solution to cool, a binder consisting of fly ash and slag was prepared by mixing them in a container. The stages of making mortars were as follows: first, sand and binder (fly ash and slag) were mixed evenly, then an alkaline solution consisting of SS and SH at a specific ratio was added and stirred until the mixture was homogeneous.

After the mortar was evenly mixed, the workability was tested using the flow table test. The last step was to make a compressive strength test sample with a cube-shaped mold. The sample-making process

was carried out in two layers, and each layer was compacted so that the sample had no pores. After 24 hours, the sample was removed from the mold and cured by wrapping it in plastic until testing.

Mix ID	Designation	NaOH Flake (gr/L)
GPM1	$SH-1M$	40
GPM2	$SH-2M$	80
GPM4	SH-4M	160
GPM ₆	SH-6M	240
GPM8	SH-8M	320
GPM10	$SH-10M$	400

Table 3. NaOH flake requirement per liter of sodium hydroxide solution

2.2.2. Test Methode. The setting time test was carried out according to ASTM C 191-04 [17]. A conical ring with a 60 mm upper diameter, a 70 mm lower diameter, and a 40 mm height. There are two types of setting time tests: initial and final setting time. The initial setting time corresponds to when the Vicat needle penetrates at a depth of 25 mm. In comparison, the final setting time is determined when the Vicat needle no longer penetrates the paste (the paste has already hardened).

A flow table test according to ASTM C1437-07 was used to test the workability of mortar [18]. The conical mold utilized in the flow table test had a 50 mm height, a bottom diameter of 100 mm, and a top diameter of 70 mm. The test step begins by pouring fresh mortar into the conical mold in two layers, compacting each layer 20 times evenly. After removing the mortar from the mold, a flow table test was conducted by lifting and dropping 25 times in 15 seconds. Then measure the diameter of the mortar's spread four times.

The compressive strength test of mortars was determined following ASTM C109/C109M-02 [19] after it had cured for seven days. The compressive strength test mold is in the form of a cube with a side of 50 mm. Compressive strength test using the Universal Testing Machine (UTM) with a maximum capacity of 50 tons.

3. Result and Discussion

This research uses six variations of SH molarity. Thereby, each test also uses the six variations of the molarity to investigate how it affects setting time, workability, and compressive strength. The results and discussion will be presented separately according to this study's dependent variables, i.e., the effects of SH molarity on setting time, workability, and compressive strength.

3.1. Effect of SH Molarity on Setting Time

The setting time test is closely related to the stages of mortar application in the field, which includes mixing, transporting, pouring, and compacting. So, the setting time should not be too fast or too slow. The results of the analysis of the setting time test with a slag percentage of 30%, Al/Bi 0.45, and SS/SH 1.5 completely can be seen in figure 1.

Figure 1 shows that the fastest initial and final setting time of 44 minutes and 60 minutes is obtained at 4M sodium hydroxide molarity. In comparison, an SH molarity of 10M has the longest initial and final setting times of 129 minutes and 214 minutes, respectively. Additionally, it is evident from figure 1 that the lower the molarity of SH (1M and 2M), the setting time increases. At low molarity, Si and Al in the precursor dissolve more slowly because there is less NaOH in the SH solution. The slower dissolution process results in a lengthy decomposition of ions in the precursor into monomers and subsequently forming polymers. However, the presence of high calcium percentage in the slag causes

an increase in geopolymer reaction by creating an amorphous structure of Ca-Al-Si gel. On the contrary, calcium (Ca) dissolution is not disturbed at low molarity sodium hydroxide [20] [21].

Figure 1. Relationship of sodium hydroxide molarity with initial and final setting time

Meanwhile, the initial and final setting time also increases at higher molarity of sodium hydroxide (6M, 8M, and 10M). This condition is due to silica and alumina's much better dissolution process. However, calcium leaching is inhibited, causing the calcium content in the mixture to be reduced. So the setting time is regulated by the geopolymerization mechanism and results in a longer setting time [22].

According to ASTM C191-04, with minimum initial and final setting time values of 45 minutes and 122 minutes, the recommended molarity of SH to be applied is 1M, 2M, 8M, and 10M. It is because the molarity of 1M, 2M, 8M, and 10M has the required setting time for the production process and mortar application in the field. The results confirm that low molarity SH can be used as an alkali activator in fly ash-slag-based geopolymer paste according to the setting time.

3.2. Effect of SH Molarity on Workability

The workability test in this study used the flow table method, intended to determine the flow of mortar containing binders instead of hydraulic cement. The flow diameters at 1M, 2M, 4M, 6M, 8M, and 10M are 160.5, 155.5, 148.25, 146.25, 143.5, and 120 mm, respectively.

Figure 2. Relationship of sodium hydroxide molarity with workability

Figure 2 shows that workability is inversely related to the molarity of SH. The workability decreases with increasing SH molarity. The higher molarity of SH means more NaOH content, thus raising the solution's viscosity. Consequently, mixtures containing high molarity of sodium hydroxide solution are more cohesive and have lower flowability [12], [23]. Reddy et al. [24] also indicated that the workability of geopolymer concrete decreases as the SH molarity increases.

3.3. Effect of SH Molarity on Compressive Strength

The compressive strength of mortar is one of the most fundamental mechanical characteristics requirements. Higher mortar quality is associated with increased compressive strength. This study tested the compressive strength of mortar at seven days. Based on figure 3, the compressive strength of mortar with SH molarity of 1M, 2M, 4M, 6M, 8M, and 10M was 33.61MPa, 37.86MPa, 41.7MPa, 46.86MPa, 38.15MPa, and 38.46MPa, respectively.

Figure 3. Relationship of sodium hydroxide molarity with mortar compressive strength

Initially, increasing the concentration of SH solution causes the compressive strength of the mortar increases. However, the increasing SH molarity above 6M leads to a lower compressive strength. The compressive strength reduction is noted at the high molarity of sodium hydroxide (8M and 10M). At the ICAMBF-2022

high molarity of sodium hydroxide, the compressive strength is dominated by the formation of a less dense NASH gel due to the geopolymerization reaction of silicates and aluminates present in fly ash. However, the dissolution of calcium in the slag is inhibited, so the CSH gel produced is also lower, causing a decrease in compressive strength. The maximum mortar compressive strength of 46.86 MPa is obtained at 6M sodium hydroxide molarity.

4. Conclusions

This study investigated the effects of varying the molarity of SH solution on the workability, setting time, and compressive strength of fly ash-slag-based geopolymer mortar. The following conclusions are offered: at low molarities of SH (1M, 2M, and 4M), the greater the molarity, the faster the setting time. Meanwhile, at high molarity of SH (6M, 8M, and 10M), the setting time decreases with increasing molarity of SH. Considering the setting time requirements, SH solutions of 1M, 2M, 8M, and 10M are recommended for field application. Increasing the concentration of SH solution increases the compressive strength but reduces the workability of fresh mortar. After reaching the optimum value, the compressive strength of the mortar decreases as the SH molarity rises. The highest mortar compressive strength is 46.86 MPa at 6M SH molarity. The study confirms that SH solutions with low molarities (1M and 2M) can be applied as an alkaline activator in fly ash-slag-based geopolymer mortar.

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