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Performance Changes in Mass and Compressive Strength of High-Calcium Fly Ash Based Geopolymer Concrete Due to Sodium Sulphate Exposure

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Abstract

The sulfate environment is one of the conditions that significantly affect the durability of concrete. Sulfate attack causes cracks and affects the quality of the concrete. Therefore, it is necessary to innovate concrete that can withstand the aggression of this sulfate attack. Geopolymer concrete is a breakthrough that can be developed as an alternative material without using Portland cement as the ingredients, yet by using class F fly ash as the primary source material. On the other hand, the availability of class C fly ash with high CaO content is highly abundant in Indonesia. The high calcium content in this fly ash will cause the fresh geopolymer concrete to harden rapidly. Previous studies used sucrose as an additive to overcome this rapid hardening problem. However, its effect on the geopolymer concrete exposed to sulfate ingression requires further investigation. In this study, the geopolymer concrete was produced by using class C fly ash and sucrose. The sulfate ingression was simulated by immersing the concrete specimen in the sodium sulfate solution while the mass changes and compressive strength were examined. pH measurement of the sodium sulfate solution was also carried out to provide a different perspective on the data analysis. Cylindrical specimens, measuring 10 cm x 20 cm, were produced using the dry-mixing method and the characteristic strength of 28 MPa according to SNI 2847–2019. From the results, geopolymer concrete with sucrose showed a high workability performance through the high slump value obtained. Nevertheless, the experimental results also indicated the declining strength of geopolymer concrete after the sulfate exposure. However, only a slight reduction was observed. The strength residue still satisfies the minimum requirement of SNI 2847-2019. It shows the potential of geopolymer concrete to be used as a construction material in a harsh environment with high sulfate content.

Keywords: Geopolymer concrete; High-calcium fly ash; Sulfate exposure

1. Introduction

The Portland cement production process releases carbon dioxide (CO_2), a major contributor to greenhouse gas emissions in the atmosphere. The production of each ton of cement clinker results in the release of one ton of carbon dioxide (CO_2) into the atmosphere. World cement production contributes 1.6 million tons of carbon dioxide, about 7% of CO_2 released into the atmosphere [1]. Some things need to be considered, namely the impact of Portland cement production on environmental sustainability. In addition, Portland cement concrete is highly alkaline, making it susceptible to chemical attack [2]. Experts have been trying to find a substitute for Portland cement, one of which is offered: using a mixture of alumina-silica with an alkali activator through the geo-polymerization process, resulting in a mixture of geopolymers [3]. Fly ash-based geopolymer concrete offers excellent mechanical properties and durability, which is not offered by OPC concrete [4]. Researchers found geopolymer-based fly ash has good-performance concrete [28–36]. Concrete structures are expected to have good durability and resistance during the planned time. Several factors reduce concrete durability and resistance. Lack of resistance is caused by many factors, including chemical attack factors like sulfate. Sulfates can be found in marine and underground environments. The content of seawater is 96.5% pure water and 3.5% salts, and within this 3.5% salt, it has chloride (55%); sodium (31%); sulfate (8%), magnesium (4%); calcium (1%); potassium (1%) and others [5]. Sulfate ingression in concrete is considered harmful since it can cause cracks in its microstructure.

Observing sea salt's content, it is discovered that sulfate in the sea is mainly found in sodium sulfate (Na_2SO_4). Construction damage that usually occurs in the marine environment is very aggressive because seawater consists of chlorides and sulfates. Both ions are highly harmful to the durability of concrete. Chlorides affect durability by initiating corrosion of steel reinforcement, whereas sulfates affect it by damaging the concrete.

A sulfate attack is a chemical breakdown process that occurs between the hydrated cement compounds (alumina and calcium hydroxide) and sulfate ions (SO_4), which are bound to different cations such as Ca^+ and Na^+ [6]. Each type of sulfate works by another mechanism and can cause some problems related to the resistance of the material [7]. The degradation mechanism appears due to the formation of crystalline compounds in the material's pores, causing cracks in the concrete [8]. A sulfate attack occurs when water is contaminated with sulfate penetrating concrete by diffusion or capillary [9]. Research shows that using fly ash increases the resistance of concrete to the entry of aggressive agents by reducing the permeability and diffusion coefficient [10]. Damage to concrete in a sulfate environment is caused by the reaction between calcium ions in concrete and sulfate ions. Studies of sulfate attack on the concrete show that the response of calcium ions in concrete with sulfate ions to form ettringite and gypsum, which causes cracks on the concrete surface, reduces the durability of the concrete [6]. Gypsum causes a reduction in stiffness and strength, expansion, mass change, and cracking by ultimately turning the material into a mushy and non-cohesive mass. The formation of gypsum and ettringite due to sulfate attack can absorb moisture so that the volume of the solid phase of concrete can increase to about 124% and 227% [11]. This study discusses the mass changes in geopolymer concrete made from type C fly ash after exposure to sulfate ingression. Geopolymer concrete was cured at ambient temperature for 28 days prior to the immersion in a sodium sulfate solution to determine its impact on the alteration of geopolymer concrete mass and compressive strength.

2. Method

2.1 Specimen

The manufacture of geopolymer concrete started with calculating the composition of the wet method [12]. Furthermore, the calculation based on the wet method is changed to a calculation based on the material composition of the dry method [13]. The design of dry-mixed geopolymer concrete can be seen in Table 1. The activator composition ratio of Na_2SiO_3: NaOH is 1:1 and uses one percent sucrose by weight of Fly Ash.

Table 1. Mix Design Dry-mixed Geopolymer Concrete

Materials	Weight (kg/m^3)
Fly ash	467.36
Fine Aggregate	548.83
Coarse Aggregate	1242.61
NaOH	48.60
Na_2SiO_3	55.45
Water	134.30
Sucrose	4.67

2.2 Materials

2.2.1 Fly Ash

Fly Ash in this study came from PT. Paiton Jawa Power Probolinggo. Table 2 shows the composition of fly ash, which is indicated by XRF (X-Ray Fluorescence). XRF fly ash results show that the CaO level is above 18%. Therefore, fly ash can be categorized as C-type.

Table 2. Fly Ash Composition

Oxide	wt %
$A1_2O_3$	9.45
SiO_2	24.85
P_2O_5	0.45
SO_3	0.35
K_2O	1.26
Cao	27.2
TiO_2	1.3

V_2O_5	0.06
Cr_2O_3	0.02
MnO	0.31
Fe_2O_3	30.7
ZnO	0.09
MoO_3	3.4
BaO	0.57
Na_2O	-

2.2.2 Sodium Hydroxide

This study uses Sodium Hydroxide NaOH) with a content of 14 M as an Alkali activator. High molarity releases more Si and Al ions in fly ash. Therefore, a better polycondensation reaction increases the long-term compressive strength [19].

2.2.3 Sodium Silicate

Another alkaline activator used in this study is sodium silicate ($Na_2SiO_{3}.5H_2O$). The specifications of Sodium Silicate are described in Table 3.

Table 3. Specification of Sodium Silicate

Parameter	Content	Unit
Sodium Oxide (Na_2O)	29	%
Silica (SiO_2)	29.11	%
Water insoluble	0.02	%
Fe	54	ppm
Whiteness	92	%
Bulk Density	0.95	g/cc
the pH of 1% Solution	12.5	
Melting Point	72.2	°C
Particular Size	95	%

2.2.4 Fine Aggregate

Sand used as a fine aggregate in this study was from Lumajang, Indonesia. The specification is given in Table 4.

Table 4. Fine Aggregate Test Results

Parameter	Content	Unit
Fine Modulus	2.25	%
Volume Weight	1.60	ton/m^3
Moisture	0.57	%
Specific gravity	2.82	ton/m^3
Absorption	0.68	%

2.2.5 Coarse Aggregate

The gravel used in the study was collected from PT. Calvary, as shown in Table 5.

Table 5. Coarse Aggregate Test Results

Parameter	Content	Unit
Fine Modulus	3.59	%
Volume Weight	1.30	ton/m^3
Moisture	0.52	%
Specific gravity	2.72	ton/m^3
Absorption	2.19	%

2.3 Geopolymer Cement Manufacturing

In this study, geopolymer cement was manufactured through several stages. First, using a ball mill machine, geopolymer cement consisting of fly ash, NaOH(s), and $Na_2SiO_3.5H_2O$(s) was ground. It was then followed by adding the fly ash, NaOH, and $Na_2SiO_{3}.5H_2O$ according to the calculated mix design. Next, the steel ball was inserted into the ball mill machine. In this stage, the ball mill machine was closed, and the purpose was to prohibit the material from leaking out. The ball mill was closed by tightening the locking screw, and the ball mill machine was then turned on and set to rotate at 13 rpm for 500 cycles. After the machine had stopped, the ball mill machine cover was opened, and the steel ball was removed.

2.4 Geopolymer Concrete Manufacturing

Before making concrete, coarse aggregate or gravel must be washed first to clean the mud still attached to the gravel and then sieved on a no. 4 sieve before the casting. The fine aggregate or sand must be dry to avoid adding extra water to the concrete mixture. Geopolymer concrete mixing was carried out by applying the dry method. The process started by putting gravel and sand into the concrete mixer machine. In this process, sucrose was incorporated into the geopolymer concrete mix. Geopolymer cement was added to the mixture and continuously stirred when the mixture had been homogeneously mixed. The mixing process was then concluded by adding the water. After the fresh concrete was collected from the mixer, a slump test was immediately performed to determine the workability of this concrete. Lastly, the geopolymer concrete was cast into the mould and moulded after 24 hours.

Table 6. Cylindrical Geopolymer Concrete

Test	**Time Immersion (days)**	**Number of Samples**	
		Sulfate Immersion	**Air Curing**
Compressive Strength Test	0	-	3
	28	3	3
	56	3	3
	84	3	3
Mass Change Measurement	0, 7, 14, 28, 56, 84	3	3

Figure 1. Geopolymer concrete samples (a), air ambient curing of geopolymer concrete samples (b), sulfate immersion of geopolymer concrete samples (c)

2.5 Geopolymer Concrete Air Curing and Exposure by Sulfate Immersion

Concrete curing was carried out using the air-ambient curing method for 28 days, as shown in Figure 1 (b). After air curing, the concrete specimens were immersed in a sulfate solution using a sodium sulfate (Na_2SO_4) immersion with a 50 g/L based on ASTM C 1012 for 84 days, as shown in Figure 1 (c). The samples were measured for mass change at 0, 7, 14, 28, 56, and 84 days and conducted compressive strength at 0, 28, 56, and 84 days—the results after sulfate immersion were compared to those samples with air ambient curing.

3. Results and Discussion

3.1 Slump Test

Due to many samples, the manufacture of geopolymer concrete was carried out in 3 stages. A slump was done to determine the workability of concrete after casting specimens. Table 7 shows the slump value after the Abrams cone was lifted.

Table 7. The Result of Slump Test

Casting	**Slump**	**Standard**
	mm	**SNI 1972:2008**
1	155	150-230 mm

2	180
3	160

The first, second, and third slump measurements from the 3 castings were 155, 180, and 160 mm, respectively. Slump results from the three castings have met the workability standards by SNI 1972:2008 code because of still in the value range of 150 – 230 mm.

3.2 Visual Observation

The pores of the concrete are filled with the formed crystals, which might give initial advantages to the mechanical strength. However, these crystals did not stop reacting and caused internal stress on the concrete elements, which can cause cracking and other related problems. This reaction causes the concrete to undergo more intense degradation, resulting in the expansion and dissolution of cement compounds. Due to this aggressive ingression, the structure's useful life will be reduced and might lead to the hazard mechanism that can be initiated only three years after exposure [6]. Sodium sulfate (Na_2SO_4) reacts with a calcium-containing binder to form gypsum (calcium sulfate dihydrate, $CaSO_4.2H_2O$) and ettringite ($Ca_6Al_2(SO_4)_3.(OH)_{12}.26H_2O$) [14].

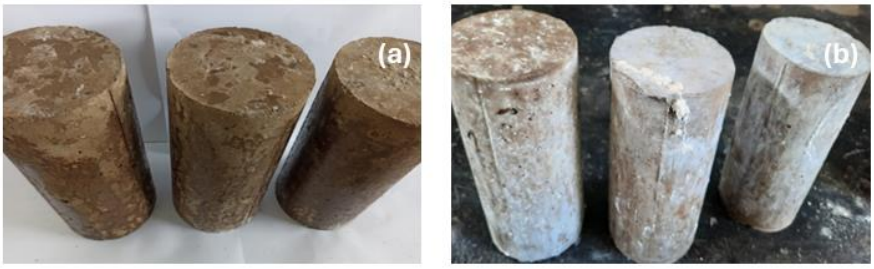

Figure 2. Visual geopolymer concrete before sulfate immersion (a), visual geopolymer concrete after sulfate immersion (b)

Figure 2 (b) shows the visual appearance of specimens after being immersed in sodium sulfate for 84 days. There is a change in appearance, the formation of several layers of sediment on the concrete surface. White layers were formed on the sample surface due to the long immersion. Visually there was also a reaction with air which resulted in a color change on the geopolymer concrete.

3.3 Mass Change

The procedure for evaluating concrete exposed to sulfate attack refers to [22] ASTM C 1012 and [21] ASTM C267. The concentration of sodium sulfate solution used in this study was 50 grams/liter, as suggested by ASTM C 1012.

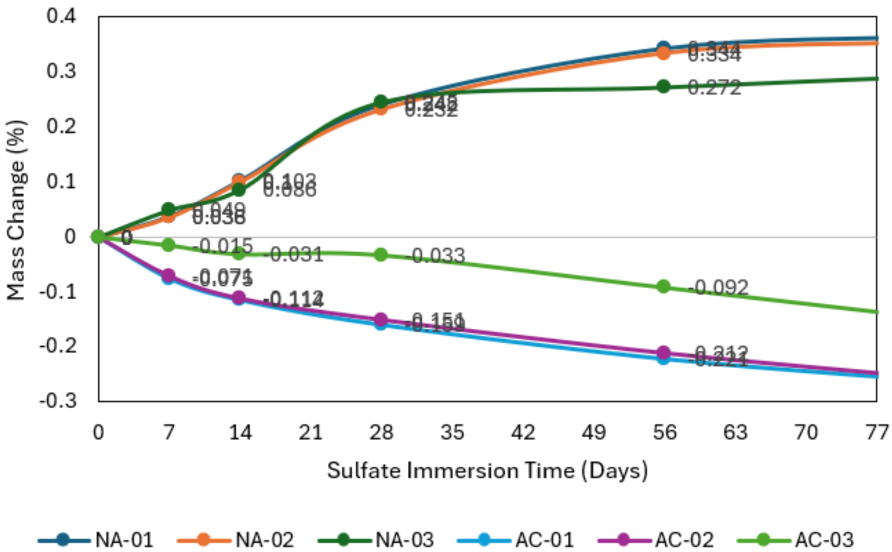

Figure 3. Result change in mass

*NA = sodium sulfate immersion
AC = air - ambient curing

Figure 3 shows the mass changes of geopolymer concrete after being immersed in Sodium Sulfate (Na2SO4), where the specimens gained mass after the exposure. Until the immersion age of 84 days, the average mass change was 0.338 percent. This outcome shares a similar finding with previous research where the mass also escalated up to 0.360 percent after 84 days of sodium sulfate exposure [11]. Rashidian [15] also reported a consistent trend, with the increase in mass at the age of 120 days of immersion being 2.2 percent. The mass gain is believed to be caused by the formation of gypsum and ettringite, which filled the voids in the concrete, yet might make the concrete prone to crack and break [11]. Figure 6 also shows the change in mass of geopolymer concrete based on fly ash type C with ambient curing. Until the age of 84 days, the difference in mass was 0.224 percent. A similar trend was also reported in another study [16]. The change in ambient curing mass at 180 days was 0.972 percent. In Ambient curing treatment, when the water present in the sample evaporates, water reduction occurs, which causes weight loss over some time [16].

3.4 Compressive Strength

The compressive strength test of ambient air-curing treated geopolymer concrete was carried out at 0, 28, 56, and 84 days. However, the compressive strength of those samples after sodium sulfate immersion was conducted at the age of 28, 56, and 84 days. The results of the compressive strength test are presented in Figure 4. The compressive strength of the concrete reaches 38.25 MPa in the zero-day air-ambient curing and sulfate immersion on two kinds of 28-day-old cylindrical samples. Therefore, it meets the standard [20] of SNI 2847-2019 for class S1 sulfate exposure classification, which was more than 28 MPa. After exposure to sulfate for 0, 28, 56, and 84 days, the compressive strength decreased respectively to 38.25, 41.14, 39.84, and 36.45 MPa.

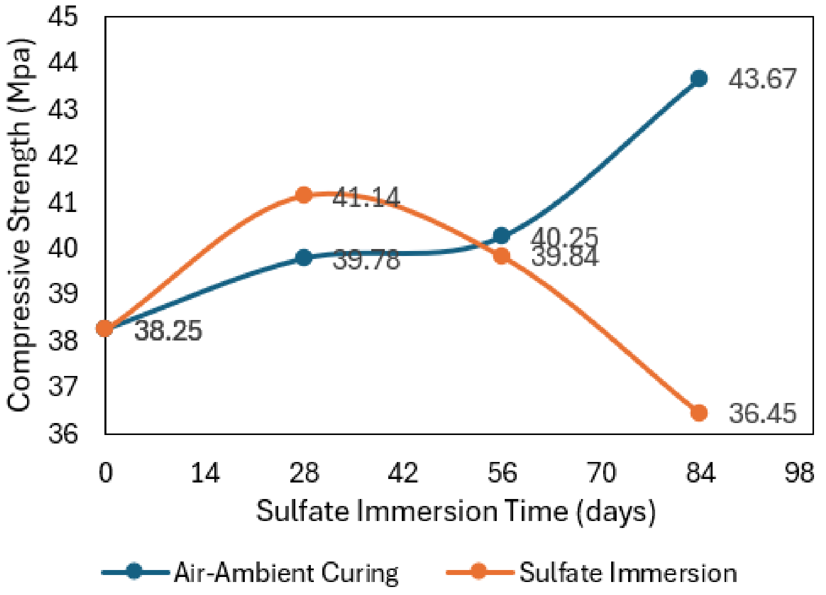

Figure 4. Results of Geopolymer Concrete Compressive Strength

However, after being exposed for 84 days of sulfate immersion, the compressive strength decreased by 5%, which allowed the possibility of increased compressive strength at the immersion beginning of 28 days. This occurs due to the filling of the pores by the formed crystals, which are gypsum and ettringite [17]. Figure 4 shows a difference in the compressive strength of concrete between ambient curing and sulfate immersion at some ages. The increase in compressive strength along with ambient curing age was also reported by Harjitno's 2005 study [18]. The compressive strength with air-ambient curing increases with the age of the concrete. The compressive strength of those for 0, 28, 56, and 84 days increased were 38.25, 39.78, 40.25, and 43.67 MPa, respectively.

The water-cement (w/c) factor is one of the most influential factors for compressive strength because w/c is the ratio of the amount of water to the amount of cement in a concrete mixture. The smaller the w/c value used, the better the strength of the concrete will be. Table 8 shows the water per geopolymer cement factor of 0.235.

Table 8. Water Cement Factor

Material	Weight (Kg)	Water per Geopolymer Cement Factor
Geopolymer Cement	571.41	0.235
Water	134.30	

3.5 pH Test

Figure 5 depicts the pH changes of sulfate solution for immersion of geopolymer concrete samples. The pH was measured at 0, 1, 3, 7, 14, 21, 28, 56, and 84 days. The result obtained after measuring using a pH meter showed that the pH of the solution increased during the first 14 days. However, in the following week, the pH of the solution was almost constant at a pH value of 9.0-10.00. This was also reported by a study conducted by Karakoc in 2015. During an initial immersion, the pH also increased, and then the pH had a constant value at pH 9.0-10.00.

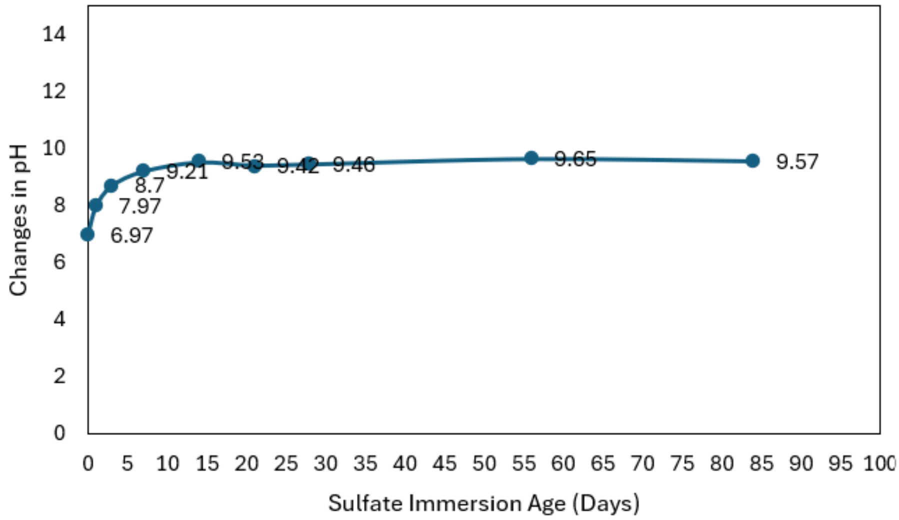

Figure 5. pH Test Results

4. Conclusions

Based on the experimental results of this study, it can be concluded that: The geopolymer concrete mass change test results show an increase in mass of 0.338 percent for 84 days of sulfate immersion. The increase in mass was probably due to the reaction of the formation of gypsum and ettringite, which filled the voids in the concrete and caused the concrete to crack and break [11]. Compressive strength test at the age of 28 days attained an average value of 38.25 MPa, then increased to 41.14 MPa at 56 days. Beyond this period, the strength decreased to 36.45 MPa at 84 days. The compressive strength of high calcium fly ash-based geopolymer concrete with dry-mixed method still meets the SNI 2847-2019 Indonesian concrete standard for implementation in the sulfate environment of class S1 classification, which is more than 28 MPa.

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