Original Article

Influence of Alkaline Solution Ratio, Superplasticizer, and Curing Method on Geopolymer Paste Compressive Strength under Sulfuric Acid Exposure

Ratni Nurwidayati^{1,2*}, Akhmad Rizalli Saidy¹, Henry Wardhana², Nursiah Chairunnisa², Putri Retno Ayu Kinanti², Noor Sasmita²

¹Doctoral Program of Agricultural Science, Postgraduate Program, Lambung Mangkurat University, Indonesia. ²Civil Engineering Program, Engineering Faculty, Lambung Mangkurat University, Indonesia.

**Corresponding Author : ratninurwidayat@ulm.ac.id*

Abstract - This paper presents an experimental investigation focusing on the short-term durability of geopolymer paste derived from fly ash. The study involves the activation of Class F fly ash using sodium silicate and sodium hydroxide at varying ratios alongside different proportions of superplasticizer. The assessment encompasses an analysis of compressive strength, visual alterations, and mass fluctuations, considering the influence of two distinct curing methods. The results indicate that an increase in the ratio of sodium silicate to sodium hydroxide and the percentage of superplasticizer leads to a concurrent rise in compressive strength. Notably, a sodium silicate to sodium hydroxide ratio of 2.5 exhibits more excellent sulfuric acid resistance than lower ratios. Additionally, it is observed that test specimens subjected to moist curing with a higher alkaline solution ratio demonstrate increased resilience in sulfuric acid environments.

Keywords - Durability, Fly ash, Geopolymer paste, Sulfuric acid, Superplasticizer.

1. Introduction

Although it frequently negatively impacts the environment, construction is essential for infrastructural development and economic growth. The large amount of cement used in construction is one of the main environmental issues. Usually making up between 10 and 12 percent of the total volume of concrete produced, cement is the main component of concrete [1]. Carbon dioxide $(CO₂)$ emissions per kilogram of cement range from 0.66 to 0.82 kg. Greenhouse gas emissions rise as a result of the circumstances. Between 5 and 7% of the world's $CO₂$ emissions come from the manufacturing of cement [2–4]. Geopolymer is an emerging and sustainable alternative to traditional Portland cement. It is formed by reacting aluminosilicate materials with an alkaline solution, resulting in a unique chemical composition and microstructure binder. Geopolymer concretes are made from high-volume industrial waste materials to produce concrete that is low in energy consumption, has a low carbon footprint, is sustainable, and is free of Portland cement [5]. Geopolymer reduces 44-64% of greenhouse gas emissions over binder-containing OPC [6]. Turner and Collins [7] presented a just about 9% value, significantly less than previous estimates. Nonetheless, this demonstrates that geopolymers offer a potential remedy for the problems associated with sustainable building. Some

researchers utilized a curing temperature to achieve geopolymer's high early compressive strength [8]. However, it is worth noting that higher curing temperatures may necessitate increased energy consumption during the curing process, and curing temperature is challenging to implement in geopolymer field applications [7,9,10]. Strengthening with age was more evident in ambiently cured specimens than in heat-cured specimens [8]. Hence, this research evaluates the effect of the curing method on room temperature and moist curing. Moist curing provides a controlled environment with consistent moisture levels. Workability and setting time are still challenges in geopolymer manufacture. Geopolymer workability is improved by adding more water. Geopolymer concrete has a comparatively low liquid content. Water is found only in the alkaline solution and the aggregate.

However, this activity can degrade the geopolymer's mechanical characteristics [11]. Adding a superplasticizer improves workability and the setting time [12-13]. So, this study will evaluate the addition of superplasticizers to geopolymer mortar mixes. Concrete durability is one of the essential features of concrete since it refers to the ability of concrete to survive external conditions such as weather, chemical attack, and abrasion without substantial damage over its service life. The chemical attack may be acid, alkali, carbonation, chloride, leaching, or sulfate attacks [14]. Durability has a significant impact on the structure's service life. Different concretes require varying levels of durability, which are determined by the exposure environment and the required qualities [15]. Geopolymer durability must be thoroughly examined before they are used in the field.

The mechanical properties of concrete are affected by a low pH environment. Because of its alkaline composition, OPC concrete is vulnerable to acid damage. The acid ions in the peat water will break down the calcium (Ca) in the cement paste, causing concrete degradation [14]. As a result, the durability of concrete is a crucial factor in the long-term use of concrete in harsh situations [16]. Several investigations have demonstrated that geopolymer concrete is more resistant to acidic conditions than normal concrete or OPC concrete [17–21]. Evaluating the durability of geopolymers in both the short term and the long term is essential for assessing their suitability for various construction applications. This study investigates the short-term durability of geopolymers.

2. Materials and Methods

The raw material was local fly ash from the Asam-Asam Power Plant in South Borneo, Indonesia. ASTM C618 specifies a maximum grain fineness percentage of 34% [22]. After collecting the fly ash from the PLTU, the fly was filtered through a no. 200 sieve with a $75 \mu m$ aperture to achieve those requirements. Fly ash has a 1.45 gr/cm³ density and a specific gravity of 2.83. The percentage of fly ash grains retained in the no. 325 sieve determines the fineness. The fineness of fly ash was 24%. This percentage is less than the ASTM C618 maximum fineness requirement.

The smaller the fly ash particles, the higher the compressive strength [2,3]. The chemical content of fly ash is shown in Table 1 based on X-ray fluorescence (XRF) test findings. These results reveal that the fly ash utilized is class F since the overall chemical content is higher than 70%, precisely 86.29%, with a CaO level of 6.74%, less than 10% [22]. The $Fe₂O₃$ content of 26.14% in this investigation was substantially more significant when compared to several fly ash sources [2,4,5]. The morphology of fly ash particles was evaluated using a scanning electron microscope (SEM), as shown in Figure 1.

The fly ash grains can be seen to be spherical. The spherical shape of fly ash particles may extend the setting time, reduce viscosity and drying shrinkage, and increase flowability, compressive strength, and water resistance [23,24]. Because fly ash is spherical, the geopolymeric mixture can be extruded much more quickly, producing a denser microstructure [25]. The XRF analyses are expressed as a percentage of oxide compounds from the primary constituent components, and the energy dispersive X-ray (EDX) results are expressed as mass percentages of the constituent elements.

Table 1. Chemical composition of fly ash	
Oxide	Weight %
SiO ₂	48.86
Al_2O_3	11.29
Fe ₂ O ₃	26.14
CaO	6.74
MgO	4.46
Na ₂ O	0.11
K_2O	0.43
MnO ₂	0.36
TiO ₂	0.78
P_2O_5	0.05
SO ₃	0.34

Table 2. Fly ash chemical elements

Figure 2 and Table 2 show the results of the EDX spectrum. The primary constituents of fly ash were observed to be silica (Si) and iron (Fe), which are 16.36% and 16.87% by weight, respectively. The identification results from the EDX test are compatible with the chemical composition of the fly as determined by the XRF test, which has a high silica and iron content (Table 1). The elements carbon (C) 9.62%, calcium (Ca) 7.36%, alumina (Al) 7.07%, and magnesium (Mg) 2.97% are then listed.

The fly ash utilized has a Si/Al ratio of 2.31. Because the combustion process results are always in the form of oxide, which means it contains oxygen, oxygen (O) has the highest percentage by weight, namely 39.77%. Fly ash was activated using sodium hydroxide (NaOH) and sodium silicate $(Na₂SiO₃)$. The ratio of fly ash to alkaline solution was 1.5. Sodium silicate has 30.62% SiO₂, 9.42% Na₂O, 59.96% H₂O, and a $SiO₂/Na₂O$ ratio by weight. Dissolving 320 grams of sodium hydroxide flakes in tap water produced one liter of 8M NaOH solution. This procedure was conducted the day before the mixing process. Sodium silicate and sodium hydroxide were mixed before being combined with fly ash. This study evaluated the effect of the weight ratio of sodium silicate and sodium hydroxide or alkaline solution ratio (R) and the percentage of type D admixture as a retarder and water reducer on paste geopolymer's compressive strength and durability. The alkaline solution mixtures had 1, 1.5, 2, and 2.5 weight ratios. The composition of the admixture is polyhydroxy carbon salts. The Superplasticizer (Sp) percentages were 0.1%, 0.2%, and 0.3%. The weight ratio of fly ash to alkaline solution was 1.5. The compositions of geopolymer paste are listed in Table 3.

The geopolymer mortar specimen size was a cylinder with a diameter of 38 mm and a height of 76 mm. This experiment was comprised of two steps with a total of 192 specimens: first, the compressive strength of the geopolymer paste was evaluated, and then the durability of the geopolymer paste was evaluated. The alkaline solution was added over the fly ash in the mixer bowl and blended for two minutes. The machine was stopped, and any unmixed material from the edges of the bowl was reassembled and mixed for another two minutes. Pour the admixture into the geopolymer paste mixture according to the dose in Table 3.

This fresh geopolymer paste was poured into a cylinder mold. The molds were opened once the paste had hardened. The specimens were then cured. There were two curing systems; the specimens were covered with a moist cloth and then kept in a closed container, and the specimens were just left at room temperature. The compressive strength of

geopolymer mortar was evaluated at 14 and 28 days. It should be noted that the effect of the curing system on compressive strength was evaluated at 28 days, with an Sp of 0.3%. Following a curing period of 28 days, the study's second phase involved assessing the impact of an acidic environment on the geopolymer paste. It is essential to mention that this second evaluation step was exclusively conducted on specimens with a 0.3% SP content. The specimens were exposed to a simulated acid environment created using manufactured sulfuric acid (H2SO4) with a pH level of 3. Visual observations, mass loss measurements, and assessments of compressive strength for the geopolymer paste were conducted after 7, 14, and 28 days of submersion in the acidic solution.

3. Results and Discussion

3.1. Visual Observation

A visual assessment of the consequences of sulfuric acid immersion was explicitly carried out on specimens characterized by an SP percentage of 0.3%. Subsequent observations were then undertaken to evaluate the effects of moist and room-temperature curing before exposing the specimens to an acidic environment, with variations in alkaline solution ratios being considered. Table 4 presents the outcomes of the visual inspections conducted on geopolymer paste samples before and after exposure to an acidic environment.

Notation A denotes that the test specimens underwent an ambient curing process, while notation M signifies moist curing. In terms of visual changes, specimens subjected to moist curing and featuring an alkaline solution ratio of 1 exhibited minimal alterations, with no discernible white crystal formation even after 28 days of exposure to acidity. In contrast, white crystals were observed in specimens subjected to ambient curing under similar conditions. However, when the alkaline solution ratio was increased to 1.5, a small number of white crystals became evident on the specimens undergoing moist curing. In contrast, specimens subjected to ambient temperature curing displayed both white and yellow crystals, marking a noticeable contrast.

Fig. 1 SEM image of fly ash particles: (a) Magnification 1000 times, (b) Magnification 5000 times

As the alkaline solution ratio increases to 2 and 2,5, white or yellow crystals appear in both curing scenarios. The visual percentage of white and yellow crystals increased with the sodium silicate percentage of the total quantity of the alkaline solution. The increase of sodium silicate was represented by increasing the sodium silicate to sodium hydroxide from 1 to 2.5. The presence of these white crystals can primarily be attributed to the formation of gypsum and calcium

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sulphoaluminate (ettringite) [26]. Indeed, both gypsum and ettringite have expansive properties [27]. The expansion can give rise to internal stresses, potentially causing damage to the concrete and reducing its strength [17,28]. Chindaprasirt et al. [29] used aluminum hydroxide $AI(OH)_{3}$ and high concentration NaOH, while Chen et al. [30] used $SO₃$ and CaO to control the synthesis of ettringite in fly ash-based geopolymers.

Table 3. Visual appearance of geopolymer paste before and after exposed to an acidic environment

Fig. 3 Alterations weight after immersion in a sulfuric acid solution

3.2. Mass Change

Figure 3 illustrates fluctuations in geopolymer paste weight, involving loss and gain weight, after being submerged in a sulfuric acid solution for 28 days. The percentage change in mass was computed as the difference in mass between the exposed specimens and their corresponding unexposed counterparts relative to the mass of the unexposed specimens. Notably, specimens subjected to moist curing exhibited no change in weight during the initial seven days of submersion in sulfuric acid. The moist curing process makes the specimen saturated with free water in its pores. So, there is no weight change when soaked in an acid solution following the curing time. Conversely, specimens exposed to ambient temperature curing demonstrated a gain in weight ranging from 1.2% to 1.9% during the same period.

Voids occurred during the curing process. After being submerged for seven days, the sulfuric acid fluid permeated the material through its pores and formed crystals, so the weight was elevated [31]. As the duration of immersion in sulfuric acid prolonged, specimens that underwent moist curing consistently exhibited weight loss, and this trend persisted for the entire 28 days. Conversely, specimens subjected to ambient curing remained comparatively heavier than their initial weight before exposure to the acidic environment.

This difference was noticeable, although their weight decreased compared to the 7-day measurement. It is worth noting that the specimens with an alkaline solution ratio of 1 experienced weight losses of 1.2% and 1.9% at 14 and 28 days, respectively, which were exceptions to this pattern. This observation was appropriate for the visual findings, particularly regarding the presence of white or yellow crystals on the surface of the test specimens on specimens with an alkaline solution ratio of more than 1 (Table 4). In the moistcured, after soaking in acid solution for 14 and 28 days, the weight reduction rose when the alkali ratio was 1.5 compared to the alkali ratio of 1. However, the weight loss decreased when the alkali ratio climbed to 2 and 2.5. The highest weight loss percentage was 2.4%. Fernando et al. [32] mentioned an average mass loss of just 2.6% after being submitted to the attack of (sulphuric, hydrochloric, and nitric) acids during 28 days, while the mass loss for Portland cement concretes is more than twice that value.

3.3. Compressive Strength

Figure 4 depicts the compressive strength of test specimens at 14 and 28 days of aging across different alkaline solution ratios and Sp percentages before subjecting the specimens to a sulfuric acid environment. The compressive strength is the average of three specimens. The compressive strength demonstrates a noticeable upward trend as the specimen ages 14 to 28 days, as illustrated in Figure 4. This increase in compressive strength varies, ranging from 3.8% to as much as 31.2%. Sp0.3-R2.5 achieved the highest compressive strength of 7.73 MPa among all tested specimens.

A noticeable improvement in compressive strength at 14 and 28 days was achieved by increasing the Sp percentage across all alkaline solution ratios. The greater the Sp percentage, the more substantial the enhancement in compressive strength. At the 28 days, the boost in compressive strength for specimens with an alkaline solution ratio of 2.5 was particularly striking: at Sp levels of 0.1%, 0.2%, and 0.3%, the increase compared to specimens labeled as Sp 0% was 29%, 91%, and 139%, respectively.

This condition signifies that the increase in compressive strength is nearly threefold at Sp 0.3% compared to Sp 0%. Figure 4 presents the impact of the alkali solution ratio on the compressive strength of geopolymer mortar. As the alkali solution ratio (R) increases, there is a pronounced and noteworthy enhancement in compressive strength. For specimens without Sp, the compressive strengths at 28 days were 1.51, 2.20, 3.10, and 3.23 MPa at alkali solution ratios of 1, 1.5, 2, and 2.5, respectively. The result implies a significant increase in compressive strength of 46%, 106%, and 115% at alkaline solution ratios of 1.5, 2, and 2.5, respectively, compared to an alkaline solution ratio of 1. These findings are consistent with those of Mariamah et al. and El-Hassan and Ismail [33]. Mariamah used fly ash as a raw material, and El-Hassan and Ismail combined fly ash and Ground Granulated Blast Furnace Slag (GGBS). However, the compressive strength obtained in this study was lower than Mariamah et al. reported using the identical mixture composition. It should be noted that the CaO percentage in the fly ash utilized by Mariamah et al. was 12.86%, whereas in this study, it was just half or 6.74%. The high CaO content increases compressive strength; however, CaO also promotes the dissolution of the glass phase from the fly ash and forms a three-dimensional gel network. Adding GGBS or Ordinary Portland Cement (OPC) is one way to increase the CaO content and compressive strength [28].

However, if the CaO level is too high, additional reactions occur in the form of a C-S-H oligomeric gel, reducing compressive strength. The compressive strength of the geopolymer will be increased with the appropriate CaO component [34]. A CaO concentration of 11% is optimal for achieving high compressive strength [30]. The percentage increase in compressive strength becomes even more pronounced with Sp. At an Sp percentage of 0.3%, the compressive strength of geopolymer paste at 28 days was as follows: 2.96 MPa at an alkaline solution ratio of 1, 5.15 MPa at 1.5, 5.73 MPa at 2, and 7.73 MPa at 2.5. It indicates an increase in compressive strength of 74%, 94%, and 161% at alkaline solution ratios of 1.5, 2, and 2.5, respectively, compared to an alkaline solution ratio of 1. Figure 5 shows the compressive strength before and after three months of immersion in a sulfuric acid solution.

The zero (0) days point in Figure 5 signifies that the specimens underwent a 28-day moist and ambient curing period but had not yet been exposed to an acidic solution. It is important to note that this evaluation in an acidic environment pertains exclusively to a 0.3% Sp. The 28-day curing period before exposure to an acidic environment (0 days) did not significantly impact the compressive strength at an alkaline solution ratio of 2, showing only a marginal 1% increase in moist curing compared to ambient curing. In contrast, the other alkaline solution ratios resulted in an increased compressive strength ranging from 11% to 15%. The result is consistent with the findings of Bai [35]. Across immersion durations of 7, 14, and 28 days, a consistent pattern of heightened compressive strength with increasing alkaline solution ratios was observed under both moist and ambient curing conditions. In the case of moist curing, the increase in compressive strength was particularly pronounced, with improvements of 63.9%, 90.9%, and 175.0% after 7, 14, and 28 days of immersion, respectively, as the alkaline solution ratio escalated from 1 to 2.5.

Under ambient curing conditions, a similar trend was observed, with compressive strength experiencing substantial gains of 175.3%, 131.8%, and 185.2% after 7, 14, and 28 days of immersion, respectively, as the alkaline solution ratio increased from 1 to 2.5. Notably, with the alkaline solution ratio raised to 2.5, the compressive strength witnessed a remarkable increase at the 28-day mark, surpassing threefold the value obtained at an alkaline solution ratio of 1. The geopolymer paste test specimens exhibited increased compressive strength after exposure to an acidic environment for 7 and 14 days across all alkaline solution ratios. However, compressive strength decreased after 28 days of sulfuric acid immersion. Hydrating calcium silicates and pozzolanic processes are responsible for the initial rise in strength. Because of the pressure produced by the expanding components, these reactions result in internal confinement [36]. The percentage change in compressive strength was likewise determined by assessing the strength difference between the exposed specimens and their corresponding unexposed counterparts relative to the strength of the unexposed specimens.

At an alkaline solution ratio of 1, the compressive strength increased by 64.21% and 43.40% after 7 and 14 days of immersion, respectively. However, it decreased by 8.30% after 28 days. With an alkaline solution ratio of 1.5, there was an increase of 20.82% after seven days, a marginal increase of 0.72% after 14 days, and a reduction of 21.54% after 28 days. Similarly, at an alkaline solution ratio of 2, there was a 20.29% increase after seven days, a 10.65% increase after 14 days, and a 3.82% decrease after 28 days. However, after 28 days of immersion in the acid environment, the compressive strength declined to 7.45 MPa. The compressive strength increased by about 6.34% after seven days, 4.67% after 14 days, and a decrease of 3.55% after 28 days of immersion in sulfuric acid. This result indicates that an alkaline solution ratio of 2.5 exhibits more excellent resistance to acidic environments.

Fig. 4 Compressive strength of geopolymer paste: (a) 14 days, (b) 28 days

Fig. 5 Compressive strength of geopolymer paste after exposure to sulfuric acid: (a) Moist curing, (b) Ambient curing

4. Conclusion

This research focused on assessing geopolymer paste's compressive strength and short-term durability. The findings indicated a progressive increase in the compressive strength of geopolymer paste as the specimens aged. Among various combinations, the weight ratio of sodium sulfate to sodium hydroxide at 2.5 exhibited the highest compressive strength, surpassing ratios of 1, 1.5, and 2 in moist and ambient curing systems. This superiority was held properly before exposure to an acidic environment and during immersion. The incorporation of 0.3% superplasticizer led to the highest compressive strength outcomes. Regarding acid resistance evaluation, the moist curing method demonstrated superior resilience to the acidic environment compared to the ambienttemperature curing method. Furthermore, geopolymer paste formulated with an alkaline solution ratio of 2.5 exhibited

enhanced resistance to the acidic environment. The compressive strength decreased by 3.55% and 5.41% at moist and ambient curing, respectively, after being submerged in sulfuric acid for 28 days.

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