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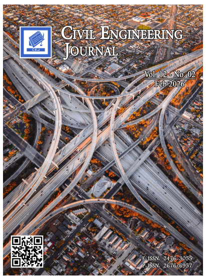

Compressive Strength and Acid Resistance of Fly Ash Based One-Part Geopolymer

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Abstract

This research studied the properties of one-part geopolymer mortar using a binder from high calcium fly ash. Sodium metasilicate (SM) and sodium hydroxide (SH) were used as solid alkali activators at ratios of 1:1 and 1:2. This study focused on the effect of the dosage and the solid ratio of the alkali activator from SM and SH for the potential to produce a one-part geopolymer. The compressive strength and corrosion resistance of mortar due to sulfuric acid and hydrochloric acid were investigated. The results showed that using a high amount of sodium metasilicate and sodium hydroxide could enhance the development of compressive strength. The fly ash-based one-part geopolymer using sodium metasilicate and sodium hydroxide (SM: NH) at a ratio of 1:1 at 18% achieved the highest compressive strength of 13.3 MPa at 60 days. For the acid attack, it was found that the fly ash-based one-part geopolymer mortar using SM: NH at a ratio of 1:1 had a lower weight change than a ratio of 1:2 after immersion in sulfuric acid. Meanwhile, the fly ash-based one-part geopolymer mortar with SM: NH at a ratio of 1:2 showed higher resistance to hydrochloric acid than at a ratio of 1:1.

Keywords: One-Part Geopolymer; Solid Alkali Activator; Fly Ash; Compressive Strength; Acid Resistance.

1. Introduction

CO_2 has a major effect on the environment and the planet, causing damage to the atmosphere and leading to impacts on climate change. Portland cement-based building materials are widely used in construction [1]. However, the cement industry is a major source of CO_2 emissions. Critical processes such as the calcination of limestone and the combustion of fuels release significant amounts of CO_2 into the atmosphere. Furthermore, the growing need for infrastructure development, particularly in developing countries, has exacerbated the problem. This underlines the critical need to explore sustainable alternatives to Portland cement to minimize its environmental impact. Thus, both the construction sector and researchers have focused on reducing the use of OPC in concrete binders [2-4]. Geopolymer is one material found to be sustainable for use as a concrete binder that addresses environmental impacts and promotes a healthier planet.

Geopolymer is an inorganic material created from a chemical reaction between aluminosilicate precursors and an alkali activator [3, 5]. The conservative geopolymer (two-part geopolymer) is produced using solid precursors and an alkaline activator based on aqueous alkaline solutions. One-part geopolymer is essentially a type of geopolymer with

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slight differences in terms of activation conditions compared to conventional geopolymers or two-part geopolymer, which uses a solid alkaline activator. In one-part geopolymers, the alkali activator is provided in a solid powdered form, eliminating the need to prepare or handle corrosive aqueous alkali solutions, such as sodium hydroxide or sodium silicate, separately. This characteristic makes one-part geopolymers more suitable for large-scale construction applications. Furthermore, transporting solid mixtures for concrete production in industrial practice is more convenient than handling liquid-based mixtures. Recent studies on one-part geopolymers have attempted to overcome the practical limitations of two-part systems, particularly the transportation and handling of alkaline solutions. Singh et al. [6] summarized that, one-part geopolymers offer clear advantages over both ordinary Portland cement (OPC) and conventional two-part geopolymers in terms of carbon footprint and economic performance. The embodied CO_2 of one-part geopolymers was approximately 65% lower than OPC, largely due to the reduced reliance on high–carbon–intensity sodium silicate solutions. one-part geopolymers also exhibited slightly lower emissions than two-part systems (≈ 2%), alongside a reduction in embodied energy of up to 47% relative to OPC. These environmental benefited parallel the life-cycle cost outcomes, namely, one-part geopolymers demonstrated a 16.8% lower production cost compared with OPC, whereas two-part geopolymers remain 9.4% more expensive due to the requirement for liquid activators and more complex processing.

Ghazy et al. [7] reported that the one-part geopolymer method simplifies the preparation process while allowing effective strength development at ambient temperatures. Compared to two-part geopolymers, one-part geopolymers generally offer better processability when the activator concentration and activator modulus remain the same. This workability improves mixing and possibly contributes to higher strength development. In addition, one-part geopolymers have a lower environmental impact due to the absence of corrosive activators, further supporting their sustainability in construction applications. Zhang et al. [8] demonstrated that the mechanical performance of one-part fly ash/metakaolin-based geopolymers strongly depends on the geopolymer type, concentration, and modulus of solid activators. Anhydrous sodium silicate proved significantly more reactive than hydrous Na_2SiO_3–NaOH blends, achieving higher reaction heat, degree of reaction, and compressive strength up to 49.2 MPa at 7 days. Although the early-age strength of one-part mixes remained lower than that of two-part systems due to incomplete dissolution of solid activators, the optimal mixture (35% concentration, modulus 1.4) provided mechanical properties approaching those of two-part geopolymers. Reaction calorimetry and acid dissolution tests confirmed that the dissolution rate and resulting geopolymer gel formation predominantly control microstructural densification and strength development in one-part systems.

Among the various materials used as geopolymer precursors, fly ash is used extensively as a precursor to produce geopolymer, especially low-calcium fly ash. In fact, fly ash is categorized by ASTM C618-22 [9] into two classes, including low-calcium fly ash (class F) and high-calcium fly ash (class C). The properties of the geopolymer are different due to the different raw materials. Nath & Sarker [10] examined the key parameters affecting the fresh and hardened properties of alkali-activated fly ash–slag (AAFS) geopolymer concrete. The results indicated that the predominant influencing factors were the slag replacement level for fly ash, as well as the type and dosage of the alkaline activator. Puligulla & Mondal [11] studied GGBS-FA blend pastes. They found that the dissolution of calcium from slag significantly impacts both early and late age properties. The availability of free Ca ions enhances geopolymer gel formation, and rapid hardening continues due to accelerated geopolymerization. The composition of fly ash varies significantly, and different chemical components affect the reaction, strength development, setting time, and durability of aluminosilicate binders [12]. Therefore, this study focuses on using high-CaO fly ash, which may also potentially be utilized as a precursor for geopolymer.

Moreover, the reaction between sulfuric acid (H_2SO_4) and calcium compounds in ordinary Portland cement also leads to the formation of gypsum. This process significantly affects the integrity of the cement matrix and leads to a significant reduction in strength, loss of mass, and deterioration of the surface [13]. In general, the cement matrix is also brittle when exposed to hydrochloric acid (HCl). The degradation mechanism consists of a combination of chemical reactions, pH reduction, ion migration, and physical changes, which together accelerate the degradation of the cement matrix [14]. An effective approach to reduce corrosion in mortar or concrete exposed to acidic environments has been the use of geopolymer as a binding agent. Ariyadasa et al. [15] reported that low-calcium fly ash geopolymer exhibited only 6% mass loss after exposure to sulfuric acid, which is significantly lower than the 49% mass loss observed in OPC. This report indicated that low-calcium fly ash geopolymer was much more resistant to acid corrosion than traditional cement systems. Teshnizi et al. [16] stated that the slag-based geopolymer mortar with an 8 M KOH mixture was the most effective mixture for resisting acid attack due to its superior compressive strength, lower weight loss, advantageous chemical composition, and overall durability in acidic environments. Furthermore, Wong [17] concluded that the superior acid resistance of geopolymer concrete compared to cement-based concrete is due to its chemically stable structure, slower degradation rate, and lower compressive strength loss when exposed to harsh acidic environments. These properties made geopolymer concrete a viable alternative for construction applications where acid exposure is a critical issue, as opposed to cement-based concrete.

Despite the growing interest in geopolymers, there is little research on the use of high calcium fly ash in one-part geopolymer systems, particularly with regard to its acid resistance. In this work, high calcium fly ash was used as a precursor for a one-part geopolymer. Sodium metasilicate (Na_2SiO_3: SM) and sodium hydroxide (NaOH: SH) were chosen as solid alkaline activators. The compressive strength of the fly ash-based geopolymers mortar with solid alkaline activator was investigated, and the effects of the SM: SH ratio and activator dosage were compared. In addition, the corrosion resistance of the mortar when exposed to sulfuric and hydrochloric acids was examined.

2. Experimental Investigation

2.1. Methodology Method

Figure 1 summarizes the workflow diagram of the research investigation.

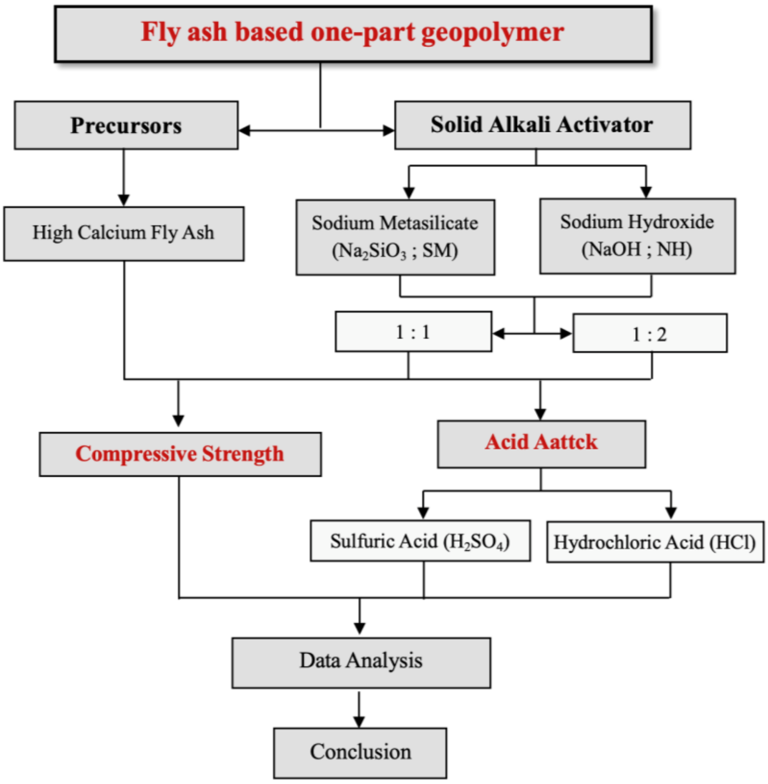


Figure 1. Methodology flow chart

2.2. Materials

The raw material used as the main precursor in this investigation was fly ash (FA) obtained as the by-product of burning coal for the electricity-generating process at the Mae Moh power plants, Thailand. The chemical composition of FA is presented in Table 1. The FA was comprised of 29.6% CaO, 24.2% SiO_2, 14.4% Al_2O_3, and 18.3% Fe_2O_3. Thus, it was classified as Class C fly ash or high calcium fly ash by ASTM C618-22 [9].

Table 1. Material properties of fly ash

Composition (%)	Fly ash
Silicon dioxide (SiO_2)	24.2
Aluminum oxide (Al_2O_3)	14.4
Ferric oxide (Fe_2O_3)	18.3
Calcium oxide (CaO)	29.6
Sulfur trioxide (SO_3)	6.5
Magnesium oxide (MgO)	2.4
Sodium oxide (Na_2O)	2.1
Potassium oxide (K_2O)	0.3
Loss on ignition (LOI)	0.6

2.3. Mix Proportion

Table 2 summarizes the mix proportion of fly ash-based one-part geopolymer in this study. The one-part geopolymer mortar was prepared using high calcium fly ash as the main precursor (Figure 2-a). The alkali activator was used in powder form or solid alkali activators, which consisted of sodium metasilicate (SM) (Figure 2-b) and sodium hydroxide (SH) (Figure 2-c). The alkali activator was a combination of sodium metasilicate and sodium hydroxide at ratios of 1:1 and 1:2 to study the influence of the activator on high calcium fly ash-based one-part geopolymer. The amount of activator powder addition was also investigated. The activator powder was used in amounts of 9, 12, 15, and 18 % by weight of the binder, corresponding to the symbols in Table 2 of 9FA, 12FA, 15FA, and 18FA, respectively. All geopolymer mortar mixtures were maintained at a constant binder-to-sand ratio of 1:2.75 and a liquid to binder (L/B) ratio of 0.5.

Table 2. Mix proportion of fly ash based one-part geopolymer

Mix	Mix Proportion (g)					Na_2SiO_3:NaOH (SM:NH)	L/B
	Fly Ash	Sodium metasilicate (Na_2SiO_3 : SM)	Sodium hydroxide (NaOH : NH)	Water	Sand		
9FA-1:1	740	33.3	33.3	370	2035	1:1	0.5
12FA-1:1	740	44.4	44.4	370	2035	1:1	0.5
15FA-1:1	740	55.5	55.5	370	2035	1:1	0.5
18FA-1:1	740	66.6	66.6	370	2035	1:1	0.5
9FA-1:2	740	16.6	50.0	370	2035	1:2	0.5
12FA-1:2	740	22.2	66.6	370	2035	1:2	0.5
15FA-1:2	740	27.8	83.2	370	2035	1:2	0.5
18FA-1:2	740	33.3	99.9	370	2035	1:2	0.5

(a) Fly ash (b) Sodium metasilicate (c) Sodium hydroxide

Figure 2. Precursors and solid alkali activator

2.4. Test of the Compressive Strength of Mortar

The compressive strength of mortar was tested using a standard cube mortar specimen with dimensions of 50 x 50 x 50 mm according to ASTM C109/C109M-20 [18]. After 24 hours, the mortar samples were demolded, sealed with a plastic sheet, and cured at a temperature of 60 ˚C for 24 h. After that, all mortar specimens were kept at room temperature until testing. The compressive strength of mortar was tested to determine age at 7, 28, and 60 days.

2.5. Test of the Acid Resistance of Mortar

The acid resistance of one-part geopolymer mortar was investigated in terms of weight change. The acids used to evaluate the change in weight of the mortar included sulfuric acid (H_2SO_4) and hydrochloric acid (HCl). A 3% concentration of H_2SO_4 and HCl solution was prepared for immersing the mortar samples. The mortar specimen was prepared as a cube specimen with dimensions of 50 × 50 × 50 mm. After 28 days, the mortar was unsealed from the plastic sheet and immersed in acid. The weight of the mortar due to acid was recorded every 14 days for 90 days. The weight change of one-part geopolymer mortar was calculated using Equation 1.

$$W_{change} = \frac{W_A - W_B}{W_A} \times 100 \quad (1)$$

where, W_{change} is Change in weight of specimen (%), W_A is Initial weight of specimen (g), and W_B is Weight of specimen at any time of immersion (g).

3. Results and Discussion

3.1. Compressive Strength

Figure 3 presents the compressive strength development of fly ash-based one-part geopolymer mortar. The results show that the compressive strength of all fly ash-based one-part geopolymer mortars with sodium metasilicate and sodium hydroxide increased with increasing curing time. The fly ash-based one-part geopolymer mortar, which uses SM: NH activator at a ratio of 1:1, had compressive strength ranges of 5.02 – 11.84, 6.27 – 12.31, and 6.46 –13.30 MPa at 7, 28, and 60 days, respectively. Meanwhile, the mortar using SM: NH activator at a ratio of 1:2 had compressive strength ranges of 7.65–10.90, 7.95–10.98, and 8.3 –11.72 MPa at 7, 28, and 60 days, respectively. The results indicate that the compressive strength of the fly ash-based one-part geopolymer mortar varied due to several factors, including the ratio of alkali activator powder (SM: NH) and the dosage of activator. The structure matrix of one part geopolymer binder was created from the dissolution of aluminum, silica, and calcium oxide in raw material by alkali solution during the reaction process, leading to the formation of C-S-H, C-A-H, and/or C-A-S-H phases during the hardening process, which contribute to the strength of the mortar [19, 20].

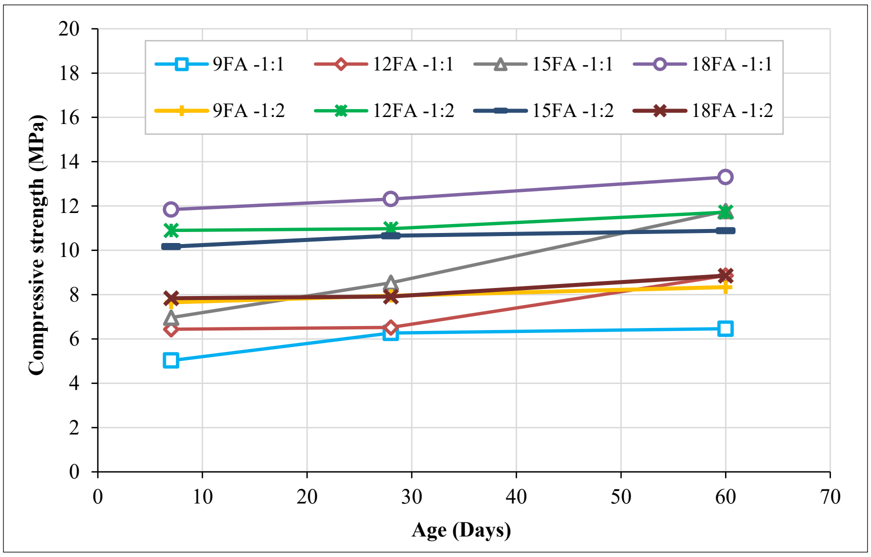
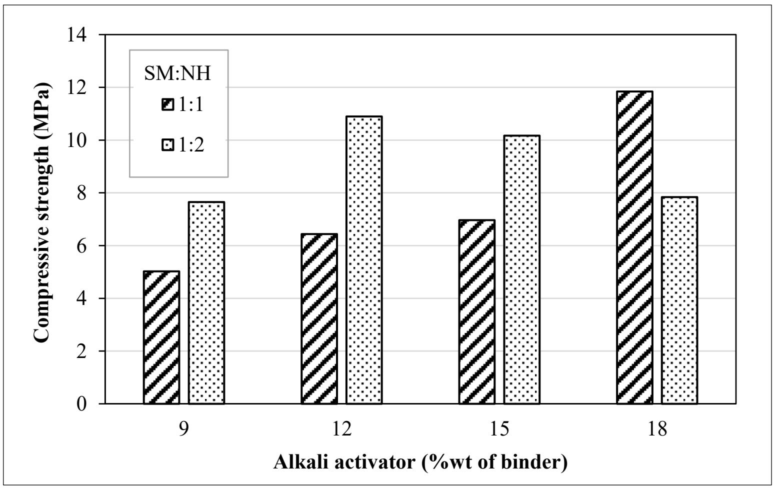
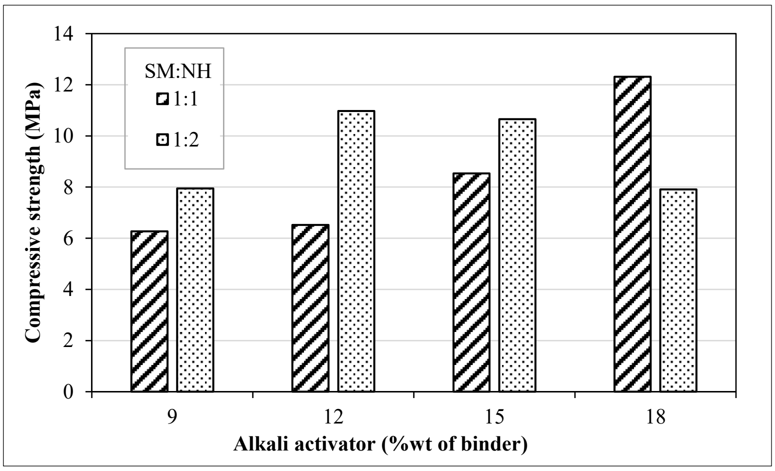


Figure 3. Compressive strength of fly ash based one-part geopolymer mortar

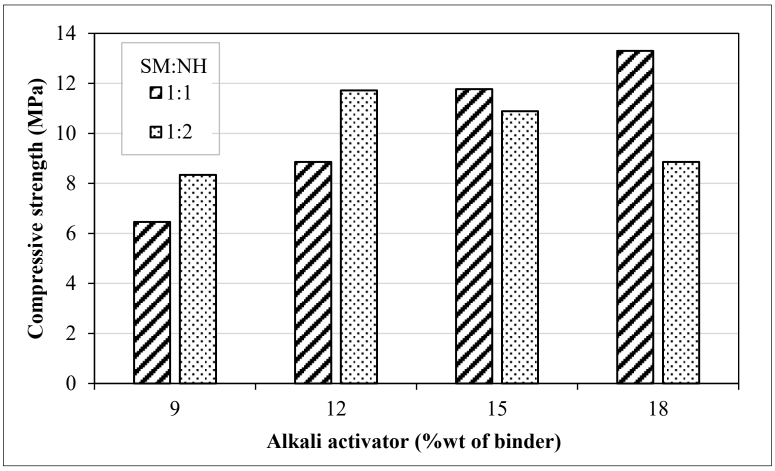
The effect of the alkali activator and the dosage of the activator is shown in Figure 4. The compressive strength of one-part geopolymer fly ash mortar with SM: NH at a ratio of 1:1 was increased with increasing dosage of alkali activator at all curing ages. The behavior may be due to the increase in dosage of alkali activator, which could improve the leaching of silica and alumina from the FA particles [21]. Moreover, the silica content in the binder also increased with the addition of more alkali activator; extra silica was contributed from sodium metasilicate, resulting in an enhanced reactivity process of the geopolymer mixture, which led to the improved strength of the binder. Reig et al. [22] confirmed that the lower silicate modulus of the alkali activator was unfavorable to the performance development. A similar result was reported by Ma et al. [23], who found that an appropriate increase in the silicate modulus of alkali-activator contributes to the performance enhancement of geopolymer mortar.



(a) At 7 days



(b) At 28 days



(c) At 60 days

Figure 4. Effect of alkali activator on compressive strength

In the case of fly ash-based one-part geopolymer mortar with an SM: NH ratio of 1:2, the result shows that the use of 9% wt and 12 %wt of alkali activator dosage into the binder had a stronger influence on the strength development than using 15% and 18% of NaOH. NaOH was a strong alkali and provided a high pH, which improved the dissolution of the aluminosilicate materials in fly ash and activated the reactivity process. Sodium metasilicate (Na_2SiO_3) provides both sodium ions and silicate ions. Although the higher NaOH content can lead to more aggressive activation, too much NaOH can also cause excessive dissolution of the aluminosilicate source. This over-dissolution reduces reaction efficiency and can potentially lead to microstructural instability [24]. In addition, the fly ash used in the study was classified as high calcium fly ash. High calcium fly ash had lower alumina and silica content than low calcium fly ash or traditional aluminosilicate precursors that are rich in silicon and aluminum [24]. Thus, the reactivated process may require more silica content from the alkali activator source, leading to the improved strength of the binder when precursors have low alumina and silica content, such as high calcium fly ash. From the study of the alkali activator powder effect on compressive strength of one part fly ash geopolymer, it was found that 12% of activator powder could provide the optimum rate for the geopolymerization process when using an SM: SH ratio at 1:2.

3.2. Weight Changes Due to Sulfuric Acid

The weight change of alkali activator mortar after immersion in 3% sulfuric acid is presented in Figure 5. The results indicate that the weight of all mortar samples made with binder from fly ash-based one-part geopolymer was increased after immersion in sulfuric acid. The increase in weight of mortar after exposure to sulfuric acid was probably attributed to the high content of CaO in the raw material (high calcium fly ash). Sulfuric acid can react with the calcium components in the geopolymer, leading to the formation of expansive products such as calcium sulfate (gypsum, $CaSO_4$) and then create additional volume [25, 26]. The one-part high calcium fly ash geopolymer with SM: SH at a ratio of 1:1 had weight gains ranging from 0.48 – 1.31 % after immersion in sulfuric acid for 98 days, while the one-part high calcium fly ash geopolymer with SM: SH at a ratio of 1:2 had weight gains ranging from 1.69 – 2.75 %. From the result, it was seen that the use of SM: SH at a ratio of 1:1 had significantly higher performance to resist sulfuric acid attack than a ratio of 1:2. The high NaOH content from the alkali activator showed the highly accelerated leaching of each oxide component from raw fly ash, especially calcium components (Ca^{2+}). The increased dissolution of calcium leads to a higher ion exchange between calcium ions and sulfuric acid. The free Ca^{2+} reacts with SO_4^{2-} ions from the sulfuric acid to form gypsum ($CaSO_4 \cdot 2H_2O$), resulting in the formation of expansive products and an increase in the weight of the mortar [27, 28]. In other words, although the high level of NaOH in the alkali activator creates a very alkaline environment that can enhance the geopolymerization process, this intense alkalinity may also increase reactivity with sulfuric acid, leading to a gain in the weight of mortars during immersion in H_2SO_4. Moreover, the sodium hydroxide could be reacted with sulfuric acid, producing sodium sulfate, and can also lead to the formation of an expanding geopolymer structure. Thus, the geopolymer mortar mixing with high sodium hydroxide content in alkali activator (SM: SH of 1:2) was weaker than that geopolymer mortar using SM: SH at a ratio of 1:1. This result was similar to the investigation by Abdulmatin et al. [29], which examined the effect of different NaOH dosages on the sulfuric acid attack of ternary binders made from palm oil fuel ash, slag, and calcium carbide residue. The investigation also reported that the addition of a high concentration of sodium hydroxide solution in one part of base fly ash geopolymer mortar increased the weight of the mortar after immersion in a 3% H_2SO_4 solution. The visual appearance of specimens exposed to a 3% H_2SO_4 solution is shown in Figure 6.

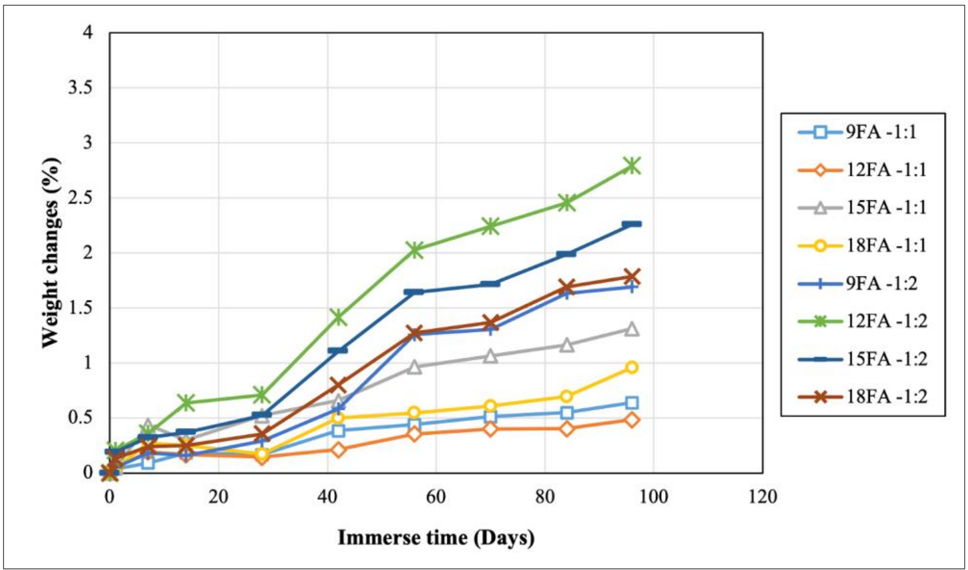


Figure 5. Weight changes due to sulfuric acid

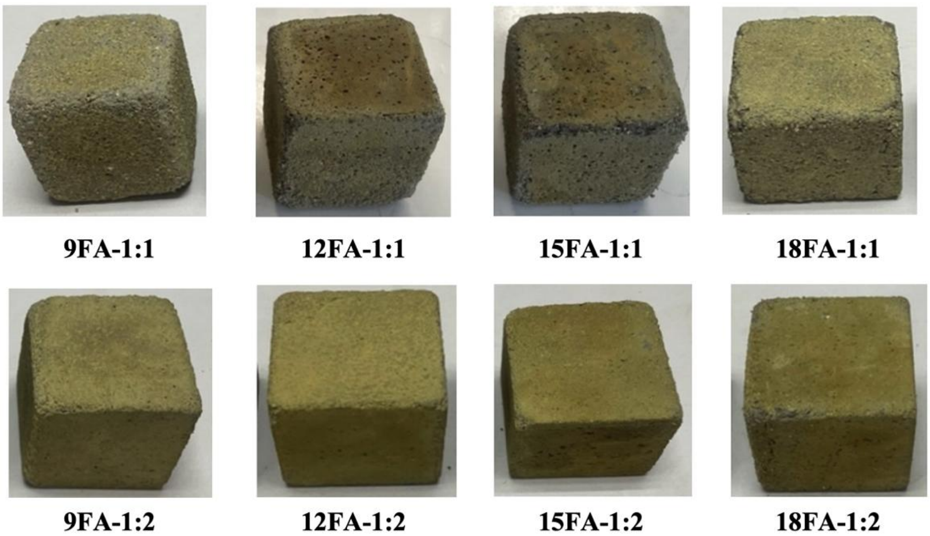


Figure 6. Specimens exposed to H_2So_4 solution

3.3. Weight Changes Due to Hydrochloric Acid

The result of hydrochloric acid resistance in terms of weight change of fly ash-based one-part geopolymer mortar after immersing in 3% hydrochloric acid is shown in Figure 7. Samples immersed in HCl exhibited a different behavior compared to H_2SO_4. All of the mortar specimens were dissolved in the hydrochloric acid solution, resulting in their deterioration in terms of weight loss. Hydrochloric acid (HCl) is a strong acid that fully dissociates into H^+ and Cl^- ions. The H^+ ions attack the aluminosilicate network by breaking Si–O–Al bonds, resulting in the dissolution of the geopolymer matrix in the mortar. This process leads to material degradation and a corresponding loss of weight. In contrast, sulfuric acid (H_2SO_4) dissociates into H^+ and SO_4^{2-} ions, and the sulfate ions can react with the calcium oxide (CaO) components in the mortar to form gypsum ($CaSO_4 \cdot 2H_2O$), causing an increase in weight [25, 26]. This mechanism shows a similar result to that reported by Xu et al. [30]. Regarding the effect of the SM: SH ratio, it was found that the weight loss of one-part base fly ash geopolymer with SM: SH at a ratio of 1:1 ranges from 2.15–6.51 %wt after immersion in hydrochloric acid for 98 days. In the case of one-part fly ash geopolymer with SM: SH at a ratio of 1:2, it was found that the mortar specimen had a weight loss range from 0.42 – 1.18 %wt. The results showed that the fly ash-based one-part geopolymer mortar with SM: SH at a ratio of 1:2 had higher hydrochloric acid resistance than SM: SH at a ratio of 1:1. The deterioration of the specimens is presented in Figure 8. Considering the effect of alkali activator powder on hydrochloric acid attack, fly ash-based one-part geopolymer at a high content of NaOH in alkali activator (SM: SH at a ratio of 1:2) gives a percentage of excess sodium hydroxide content in geopolymer, indicating higher hydrochloric resistance. The high concentration of NaOH solution in the alkali activator contributes to the alkalinity of the geopolymer system. This high alkalinity (OH^- ions) helps buffer the effects of acid, mitigating the immediate impact of HCl on the geopolymer structure. Yankwa Djobo et al. [31] reported that the sodium-rich specimens inhibited the extent of the reaction between the calcium in the geopolymer gel and the acid. Klingsad & Ayudhya [32] also reported that a high molarity NaOH solution and elevated curing temperature enhanced resistance to HCl attack more effectively than H_2SO_4 and $MgSO_4$. A similar result was found by Teymouri et al. [33], who also reported that the use of high molarity of NaOH solution resulted in reduced weight loss of alkali-activated slag concrete (AASC) specimens after exposure to HCl acid solution.

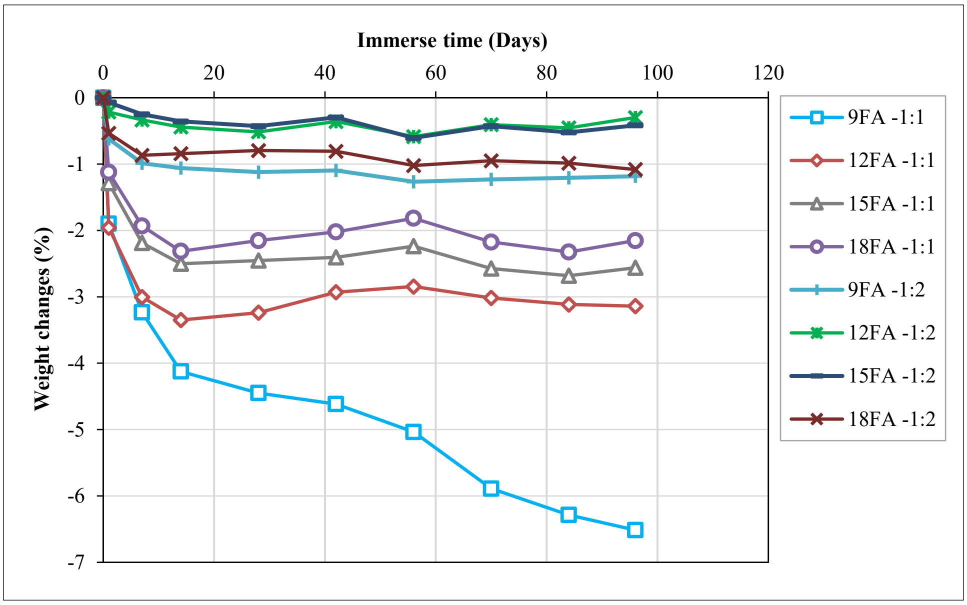


Figure 7. Weight changes due to hydrochloric acid

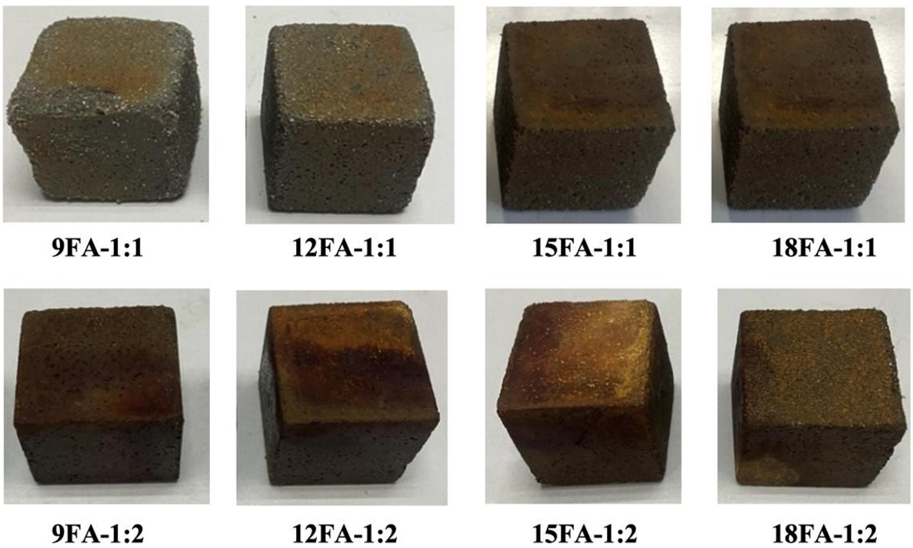


Figure 8. Specimens exposed to HCl solution

Additionally, the HCl acid resistance of fly ash-based one-part geopolymer mortars with both SM: NS ratios appears to be related to the compressive strength of the mortar. The test results also indicated that the weight loss of the mortar tends to decrease as compressive strength increases. This suggests that mortars with higher compressive strength have a denser and more stable microstructure, leading to greater degradation resistance [26, 29].

4. Conclusions

This study conducted an experimental investigation concerning the effects of the SM: SH ratio and activator dosage on fly ash–based one-part geopolymers. The compressive strength and corrosion resistance of the mortar under sulfuric and hydrochloric acid exposure were evaluated. The main findings can be summarized as follows:

- The high-calcium fly ash-based one-part geopolymer mortar exhibited variable performance depending on the sodium metasilicate to sodium hydroxide (SM: SH) ratio and the activator dosage. Mortar with an SM: SH ratio of 1:1 showed higher compressive strength when the activator powder dosage was 18% by weight of the binder. In contrast, the optimized activator powder dosage for mortar with an SM: SH ratio of 1:2 was 12% by weight of the binder. Mortar with an SM: SH ratio of 1:1 achieved the highest compressive strength of 13.3 MPa at 60 days.
- Regarding acid resistance, immersion in sulfuric acid (H_2SO_4) resulted in a weight increase for all mortar samples. However, the mortar with an SM: SH ratio of 1:2 exhibited lower resistance to H_2SO_4 compared to the mortar with a 1:1 ratio. Conversely, in hydrochloric acid (HCl), the 1:2 ratio mortar showed less weight loss than the 1:1 ratio, indicating that a higher NaOH content in the activator enhances resistance to HCl environments.

5. Declarations

5.1. Author Contributions

Conceptualization, A.A. and S.D.; methodology, A.A., S.D., and A.B.; validation, S.D., A.T., and S.P.; formal analysis, A.A. and S.D.; investigation, S.D. and A.B.; resources, S.D.; data curation, A.A.; writing—original draft preparation, A.A. and S.D.; writing—review and editing, A.B., A.T., and S.P.; visualization, S.D.; supervision, A.T. and S.P.; project administration, S.D.; funding acquisition, S.D. All authors have read and agreed to the published version of the manuscript.

5.2. Data Availability Statement

The data presented in this study are available on request from the corresponding author.

5.3. Funding and Acknowledgments

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5.4. Conflicts of Interest

The authors declare no conflict of interest.

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