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Mechanical Characterization of Geopolymer Paste and Mortar Fabricated from Alum Sludge and Fly Ash

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Abstract: This study evaluated the effect of alum sludge as an alternative to fly ash in fabricating geopolymer paste and mortar. The blending of this industrial waste (alum sludge and fly ash) is not only for the benefit of sustainable construction and disposal of industrial waste but also for the reduction of CO_2 emissions due to the increasing production of Portland cement from the cement production industry. A laboratory investigation was carried out on the workability and mechanical properties of geopolymer paste and mortar produced with alum sludge replacement in different proportions (0, 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100). A combination of an alkaline solution of sodium silicate and sodium hydroxide of 14 molarity was employed as an activator for the manufacturing of both paste and mortar geopolymer specimens. It was observed from the findings that geopolymer paste and mortar was flowable and workable when alum sludge is replaced for fly ash at higher replacement content. The addition of alum sludge to the mix improved some properties such as density, strength, water absorption, and the elevated temperature behavior. It was observed that the addition of alum sludge was optimum at the 50% replacement level. The addition of alum sludge up to 50% significantly increased the compressive strength of mortar (up to 80% increase in 28 days strength). The compressive strength of the paste and mortar increased with an increase in curing age. Thus, alum sludge and fly ash can be employed together in the production of eco-friendly cementing material for environmental sustainability.

Keywords: geopolymer; paste; mortar; alum sludge; fly ash

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1. Introduction

Some countries in Asia and Africa are among the largest consumers and producers of cement in the world, due to a swift increase in infrastructural development. This chase for development has drastically increased the rate at which cement is consumed in construction industries for infrastructural and industrial development. In developing countries such as Malaysia, cement consumption is increasing in this region due to a swift turn on infrastructural development. It is imperative to underscore that the infrastructural development process increased the production of cement in these regions, causing an upsurge in the release of carbon dioxide (CO_2) into the ether. This activity greatly causes environmental pollution which is now a global challenge. There is proven evidence that a significant amount (0.87 tons) of CO_2 is released into the ether when producing 1 ton of cement [1], causing environmental pollution that affects human health. Hence, the adoption of alum sludge–fly ash geopolymer as a replacement for cementitious binders seems to be ideal, in an optimistic stance, to reduce or eliminate the CO_2 release and combustion of carbon fuel during cement manufacturing.

The mix of alum sludge-fly ash-based geopolymer provides a helpful alternative in reduction to the consumption of Ordinary Portland Cement (OPC). Not limited to the total eradication of environmental hazards associated with cement manufacturing. One considerable approach is the total purging of negative effects associated with the disposal of alum sludge and fly ash by utilizing them as an alternative to cementitious materials. The conventional disposal practice of some industrial waste (alum sludge and fly ash) is not always a desirable option, and it attracts high transportation costs to landfill sites and the burning of waste (alum sludge) before disposal. Thus, due to the enormous benefits for both economic and environmental sustainability, studies are now aligned and determined to source different sustainable methods to reuse waste materials in geopolymer [2]. However, [3] indicated that material such as fly ash and alum sludge is pozzolanic, and being dissolvable in alkaline solution is ideal as a potential geopolymer precursor and could aid in geopolymerization. Alum sludge (waste from water treatment plant) is an unavoidable waste that is generated when purifying drinking water [4]. This waste is rich in aluminum and silica and other minor elements [5,6], which makes it a good material for the production of geopolymer material (paste or mortar). This is due to its ability to dissolve and react under an alkaline solution [3]. To improve bonding between materials, alum sludge in the past has been utilized as an alternative substitute material in the production of different civil engineering materials [4]. Therefore, it can be employed in the production of geopolymer paste or mortar to eliminate disposal hazards and reduce carbon dioxide emissions when used as a substitute for cementitious material. This outcome makes alum sludge a suitable replacement material for the production of geopolymer paste or mortar. Unlike alum sludge, the fabrication of geopolymer with fly ash has been studied [7,8] previously with encouraging results. In a review by Aleem and Arumairaj [9], it was explained that geopolymers are considered a sustainable substitute for cement, and they have somewhat similar behavior to ordinary Portland cement.

Fly ash-based geopolymers have been studied [10–13]. It was suggested by [14] that investigation was necessary to provide evidence of the behavior of produced fly ash-based geopolymer paste and concrete. Thus, using different alkali dosages on the effect of the hardened state of the material and the rate of hardening of geopolymer paste or concrete at different times of curing was examined. However, a more rigorous examination was necessary for this outcome. A study by Saludung et al. [12] demonstrated strong evidence of the physical and mechanical properties and time of curing for the production of most geopolymer materials. This was not limited to the rate of strength achieved due to different times of curing. Another point raised on the physical and mechanical properties of geopolymer binders [12] affirmed that both of the binders employed were somewhat similar to that of the conventional binder (OPC). Hence, these findings were highly affected by the concentration of the activator and the chemical composition of the binder employed. Thus, the fabrication of geopolymer with fly ash and other constituent materials could be blended using a solution with a relatively suitable alkali activation that is capable of curing conventionally under normal temperatures [13]. It was determined that geopolymer paste or concrete fabrication is economically feasible, and its hardened properties could be comparable to Portland cement [13]. Having these facts underscored, geopolymer mortar or concrete can be utilized essentially for concrete repairs and the production of precast concrete.

Furthermore, it was reported that a higher compressive strength of 10.06 MPa was achieved by employing 14 M (morality ratio) of sodium hydroxide solution, in the production of geopolymer mortar. When the molarity ratio was increased to 16 M, there was a decrease in strength of about 9.16 MPa when compared to the mortar with 14 M. In contrast, 8 M yielded the lowest compressive strength of 3.95 MPa [15]. This behavior indicates that the optimum molarity of NaOH reported was 14 M. Thus, a higher molarity of NaOH of 16 M will result in a decrease in strength. An experimental investigation with silica fume and fly ash that is abundant in calcium showed that geopolymer specimens with silica fume up to 40%, significantly enhanced performance on strength when compared to other samples [16]. Meanwhile, when compared with geopolymer mortar and conventional mortar, it was observed that geopolymer mortar yielded better results in both elevated and ambient temperatures [17]. Based on existing literature [18], there is an indication that geopolymer mortar manufactured with fly ash can achieve 19.28 MPa at 28 days of curing age. Green production of geopolymer materials using fly ash as a substitute for Ordinary Portland cement can be significantly achieved within acceptable parameters [19,20]. This result can only be achieved with a proper selection of materials and proportions. A study in Australia examined several methods of interaction with the determination of amorphous aluminum–silicate material. To understand how much material did/did not react but acted as filler material, X-ray diffraction test and X-ray fluorescence was used with the alkaline dissolution of fly ash. The results showed that 39% by weight of fly ash was the amount of fly ash dissolved as filler out of 50% total weight of fly ash that was employed in the geopolymer mix [21]. It was emphasized in the analysis that “little of the amorphous component could be present to participate in dissolution and polycondensation reaction during the conventional geopolymerization”. This observation implies that the amorphous iron oxide had no important role to play during this formation but accumulated in the structure as filler.

An experimental investigation by Saloma et al. [15] on the density of geopolymer mortar indicated that higher molarity decreased slump flow but increased density. A ternary blend of different industrial waste materials (fly ash ground-granulated blast-furnace slag, and high magnesium nickel slag-based) was employed and studied [1]. The findings showed that the strength and ultimate strain of geopolymer paste increased with an increase in the rate of strain, with a change in the failure mode for all samples under different impact speeds [1]. A durability study on geopolymer paste and concrete was studied [8]. It was observed that the strength property of produced specimens with fly ash and ground granulated blast slag (GGBS) in the production of geopolymer concrete tended to increase with the increase in the curing period [8]. Another study affirmed that geopolymer paste produced with fly ash and GGBS with high sodium hydroxide content increased the compressive strength of geopolymer mortar. Studies then suggested that the production of geopolymer concrete mixed with fly ash and GGBS under outdoor conditions could be feasible [22]. The microstructure and strength of fly ash (class C) and GGBS-based geopolymer were also investigated [23]. However, it was reported that an increase in the addition of fly ash content in the geopolymer mix resulted in a lower compressive strength value. It was further explained that specimens with NaOH yield a lesser effect in compressive strength gain and denser microstructure when compared to paste activated with NaOH and Na_2SiO_3. This means that the main cause of silica was produced by Na_2SiO_3 which gave the paste a denser structure and enhanced the hardening process of the produced geopolymer paste. Meanwhile, another investigation with a similar view affirmed that fly ash-based-geopolymer paste and mortar achieved a density of 2.34 g/cm^3 and a high compressive strength of 48.10 MPa with 12.5 molarity [24]. It was further explained that 12.5 molarity in the mix influences the workability of the produced geopolymer sample. A laboratory investigation also proved that a molar ratio of 10 to 15 usually produces a workable fly ash geopolymer mix [25]. An investigation from Brazil by Santos et al. [26], shed light on the feasibility of employing calcined sludge at a temperature of 750 °C for 6 h and grinding for 2 h as a source of silica and alumina. The result from this experiment indicated that Negro River sludge with a lower silica–alumina ratio yielded a lower workability property but a higher compressive strength of over 50 MPa after 28 days. Lin et al. [27] stated that a geopolymer sample with an optimum replacement content ratio produced a binding effect that permits an improvement in the geopolymerization reaction and strength of the geopolymer sample.

However, alum sludge from water treatment plants has limited experimental study on the production of geopolymer materials. Thus, due to the high reactive content of Al_2O_3 and SiO_3, it has been used in concrete as a replacement for cement [28–30]. However, the application and behavior of alum sludge in geopolymer paste and mortar have limited experimental investigation and need to be studied. Santos et al. [26] investigated calcined water treatment sludge employed in geopolymers as a source of silica and alumina with a minimal percentage content (8%) of Portland cement. Their investigation encouraged the use of water in geopolymeric synthesis. A phosphate-based geopolymer cement composite was also used for the removal of lead ions from an aqueous solution and was found to be suitable for the removal of heavy metals from wastewater from the mining industry and other industries [31]. An experimental study on strength and setting time of geopolymer paste produced with fly ash with different water ratios. The result showed that 0.2 ratio resulted in higher strength and faster setting time, while a 0.4 ratio gave a lower strength and longer setting time [32]. Past studies have utilized different waste materials in the production of geopolymer materials for a sustainable environment [33–35].

Meanwhile, this study employed untreated/uncalcined alum sludge without the addition of Portland cement as a binder and added water in the production of geopolymer paste and mortar using alum sludge and fly ash. Moreover, the investigation of the aforementioned literature was in Brazil, and this study will be focused on alum sludge locally generated from Malaysia, although it is understood that alum sludge from different locations may have different physical/chemical properties and may react differently. Hence, is important to investigate the alum sludge locally generated from Malaysia and establish the mechanical properties and optimum replacement content of the produced geopolymer product. The major objective of this investigation was to study the behavior of alum sludge-fly ash-based geopolymer paste with 14 molarity, using alum sludge as a source of aluminum-silicate for geopolymer production. This investigation also highlights the optimum replacement of fly ash using alum sludge in geopolymer paste and mortar, and the behavior of the geopolymer paste and mortar when alum sludge replaced fly ash in different proportions.

Furthermore, the study focused on comparing the mechanical characterization of geopolymer paste and mortar fabricated from alum sludge as a replacement material for fly ash. There is limited or no laboratory investigation performed on geopolymer paste and mortar using alum sludge to replace fly ash. Therefore, this utilization method will not only help to eliminate hazards associated with the disposal of industrial waste but will open up an innovative approach to applying this industrial waste in the production of geopolymer paste and mortar. Furthermore, the manufacturing of alum sludge-geopolymer paste and mortar will also minimize the environmental hazard involved in the production of cement for building and construction purposes.

2. Materials and Methods

2.1. Materials

The fly ash employed in this investigation was locally generated from Tenaga Nasional Berhad (TNB) from Jimah Power Plant located in Negeri Sembilan, Malaysia. The fly ash was collected from the power plant and put in a plastic container and thereafter transported to the laboratory for sample preparation. The fly ash was later stored in an airtight container and kept for use. The alum sludge utilized in this study was collected from a water treatment plant in Putrajaya, Malaysia. It was collected in a plastic bag from the sludge dumping site at the treatment plant and then transported to the laboratory for sample preparation. The sodium silicate and sodium hydroxide were collected from a local chemical store. Sodium hydroxide was dissolved and diluted using water (preparation of the alkali solution) fit for drinking. The alkali utilized for the geopolymerization was a mixture of sodium hydroxide (NaOH) and sodium silicate solution. The sodium hydroxide (NaOH) used as an activator had a purity of 99%. A steel mold and heavy-duty plastic mold of size 50 mm by 50 mm were used to produce geopolymer samples (paste and mortar).

2.2. Methods

2.2.1. Preparation of Samples

Alum sludge was kept in a flat metallic plate and placed to dry inside an oven at 200 °C temperature to burn out possible organic material, crushed with Los Angeles Abrasion test machine, and then sieved to get desired particle size. Crushing was necessary because after the drying process, the alum sludge formed lumps that needed to be crushed to achieve the desired particle size for testing. Alkaline activators sodium hydroxide solution (NaOH) and sodium silicate (Na_2SiO_3) were employed and prepared to produce geopolymer paste (fly ash and alum sludge) and mortar (sand, fly ash, and alum sludge). The preparation of the sodium hydroxide solution was performed by dissolving the sodium hydroxide pallet in distilled water at the desired molarity (14 M), then stirred and allowed to be completely liquified. Thereafter, the solution was allowed to cool off for 24 hours at room temperature. The molarity of 14 M was kept constant throughout the mixing of the geopolymer paste and mortar.

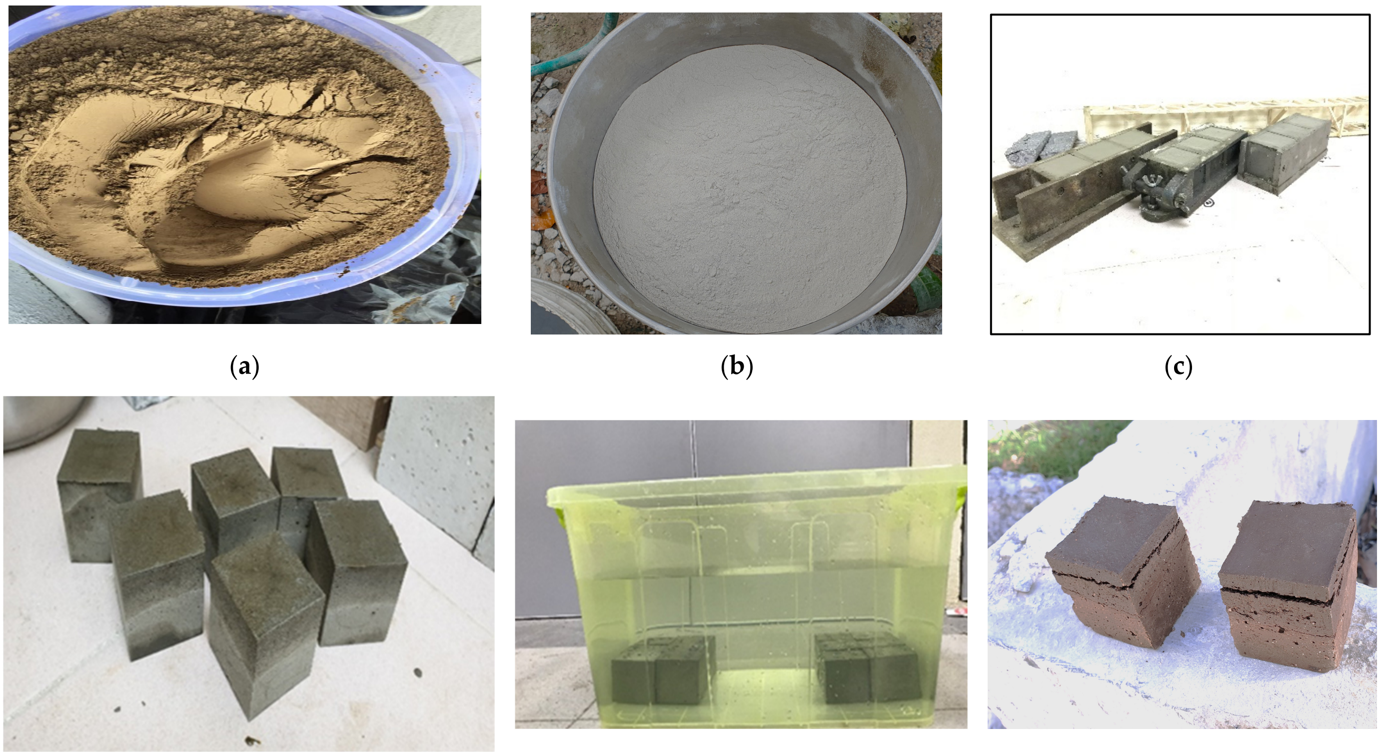
2.2.2. Blending and Casting of Geopolymer Paste and Mortar

All samples (paste and mortar) were mixed in a mini electric pan mixer. For the paste samples, the fly ash and alum sludge were first mixed to be sure there were no lumps, thereafter, sodium silicate and sodium hydroxide solution were slowly poured into the mixing pan as the fly ash was mixed. In the case of mortar, all the solid materials (fly ash, alum sludge, and fine aggregates) were thoroughly mixed until a homogenous color was obtained. After mixing was performed, the mixed materials were used to conduct a flow test before casting into a $50 \times 50 \times 50$ mm^3 cube size. This process was adopted from a study by Kuri, et al. [36] and Yahaya et al. [24]. The design mixture and procedure were adopted from past studies after considering the findings reported by Yahaya et al. [24]. The 14-molarity and mix method employed in this study was adopted from a study by Saloma et al. [15] and Yahaya et al. [24]. All the samples were left to self-compact in the mold while pouring the mixed materials. This means the mixed materials were not compacted nor vibrated. A flow test was adopted for the flowability of all geopolymers (paste and mortar). This was done to observe the effect and behavior of alum sludge as a substitute to fly ash for the production of geopolymer paste and mortar, and the workability properties of the geopolymer samples. For replacement samples, alum sludge was incorporated into the mix to partially replace fly ash in different percentages (see Table 1), for both geopolymer paste and mortar. For geopolymer mortar, the sand content was 250 g while the fly ash was replaced with alum sludge in different percentages (0, 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100) to study the behavior and effect of alum sludge on geopolymeric structure. This proportion was adopted to easily understand the effect of alum sludge on geopolymer paste and mortar when used as a replacement for fly ash-based geopolymer. After casting, the geopolymer samples were carried immediately (with the mold) into the oven for drying under a temperature of about 60 °C. The heating was adopted to facilitate and achieve a proper hardening process before the samples were immersed in the curing tank for the curing process. The geopolymer paste and mortar were retrieved from the oven after 7 h and then left to cool off at room temperature. Thereafter, the conventional curing method was employed, and the samples were completely immersed in a curing tank (see Figure 1). The samples were left in the curing tank at a room temperature of about 20 °C to 25 °C until each testing day was attained (1 day, 7 days, 28 days, and 180 days), as shown in Figure 1.

Heating of the geopolymer paste and mortar immediately after casting for 7 h in the oven was initiated after observing that the samples (geopolymer paste and mortar) started debonding/deteriorating when immersed in the curing tank after 24 h. This is to say that immersing the geopolymer paste or mortar in water without heating it in a low temperature (the conventional curing process) affected the geopolymer samples negatively; see Figure 1.

Table 1. Mix proportioning.

Sample No.	Molarity	Sand (gm)	Fly Ash (%)	Alum Sludge (%)	NaOH Solution (gm)	SiO_2/Na_2O (gm)
			Geopolymer paste			
FA0	14	0	100	0	71.5	178.5
FA1	14	0	90	10	71.5	178.5
FA2	14	0	80	20	71.5	178.5
FA3	14	0	70	30	71.5	178.5
FA4	14	0	60	40	71.5	178.5
FA5	14	0	50	50	71.5	178.5
FA6	14	0	40	60	71.5	178.5
FA7	14	0	30	70	71.5	178.5
FA8	14	0	20	80	71.5	178.5
FA9	14	0	10	90	71.5	178.5
FA10	14	0	0	100	71.5	178.5
			Geopolymer mortar			
MA0	14	250	100	0	71.5	178.5
MA1	14	250	90	10	71.5	178.5
MA2	14	250	80	20	71.5	178.5
MA3	14	250	70	30	71.5	178.5
MA4	14	250	60	40	71.5	178.5
MA5	14	250	50	50	71.5	178.5
MA6	14	250	40	60	71.5	178.5
MA7	14	250	30	70	71.5	178.5
MA8	14	250	20	80	71.5	178.5
MA9	14	250	10	90	71.5	178.5
MA10	12.5	250	0	100	71.5	178.5



(**a**) (**b**) (**c**)
(**d**) (**e**) (**f**)

Figure 1. (**a**) Raw alum sludge (**b**) Raw fly ash (**c**) Geopolymer paste in a steel mold. (**d**) The alum sludge-fly ash geopolymer paste samples were removed from the mold after oven drying for 7 h. (**e**) Alum sludge-fly ash geopolymer paste sample in the curing tank. (**f**) Geopolymer mortar sample debonded, cracked, and deteriorated when immersed in the curing tank after 24 h.

2.2.3. Properties of Materials

Table 2 presents the physical properties of fine aggregate, fly ash, and alum sludge employed in this investigation, while the chemical composition was analyzed using X-ray fluorescence (XRF) and shown in Table 3. A scanning electron microscope (SEM) machine was used to generate the microparticle image of the materials (presented in Figure 2). The chemical composition of fly ash indicated a high concentration of SiO_2 and Al_2O_3 with a percentage concentration of 59.18% and 22.82%, respectively. From Figure 2, it was observed that fly ash appeared spherical in shape, as seen from the micro-structure image.

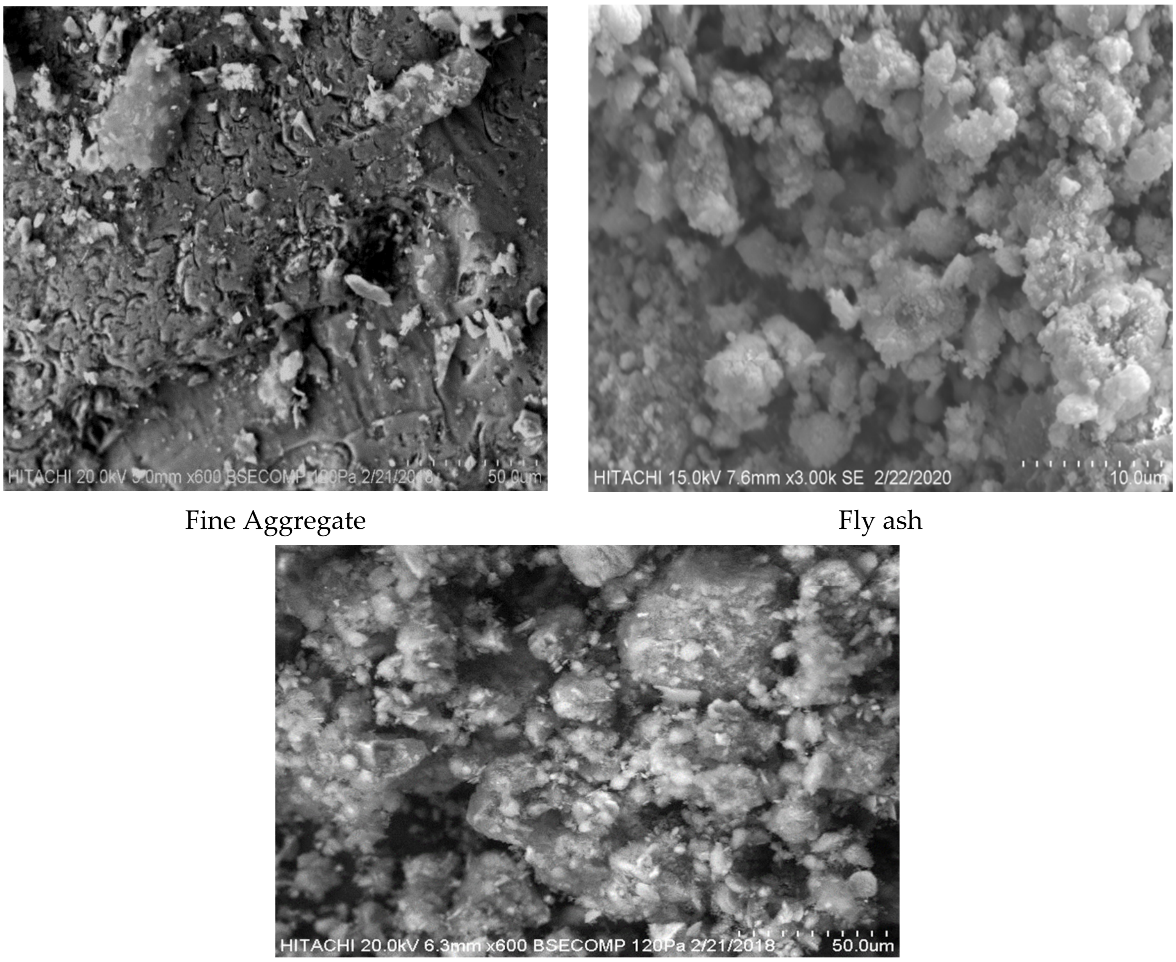


Figure 2. Microstructure image of samples.

Table 2. Physical properties of materials.

Test	FAG	ALS	FLD
Specific gravity	2.63	2.37	2.54
Blain's fineness (cm^2/g)		5369	5283
Passing 45 µ sieve (%)	68	100	98
Color	Off-white	Light brown	Grayish

Fine aggregate (FAG); Alum sludge (ALS); Fly ash (FLD).

Alum sludge's chemical composition is shown in Table 2, and it can be seen that alum sludge has higher SiO_2 (46.54%) and Al_2O_3 (38.75%) with other minor elements dictated. The microstructure image from Figure 2 shows that alum sludge is made up of particles that are irregular in shape, with an obvious rough surface morphology, pervious structure, and amorphous in nature [37–39]. In contrast, river sand had SiO_2 as the highest element concentration of about 80.76% and Al_2O_3 up to 11.64%, with an angular particle shape and specific gravity of 2.63.

Table 3. Chemical composition of materials.

Samples	SiO_2 %	Al_2O_3 %	CaO %	Fe_2O_3 %	Na_2O %	K_2O %	MgO %	MnO %	TiO_2 %
FAG	86.76	11.64	3.58	1.78	1.43	1.21	0.77	0.05	-
ALS	46.54	38.75	0.43	4.91	-	1.88	0.32	0.02	0.6
FLD	59.18	22.82	10.54	14.13	1.09	1.77	3.2	-	-

Fine aggregate (FAG); Alum sludge (ALS); Fly ash (FLD).

3. Results

3.1. Geopolymer Paste/Mortar Workability

The fresh state of geopolymer flowability is an important property that affects the behavior in the workability of geopolymer paste or mortar. A flow table test was employed (see Figure 3) to examine the workability behavior of the geopolymer product. This was performed to ascertain its flowability when alum sludge was used as a substitute for fly ash in the mix.

Figure 3. Slump flow of alum sludge-fly ash geopolymer paste/mortar.

The fresh property (workability) of the produced geopolymer samples (paste and mortar) was measured according to ASTM C1437-20 (standard test method for the flow of hydraulic cement mortar) and shown in Figure 4. The conventional flow test was employed to understand the effect of fly ash blended with alum sludge in different replacement content. From the results, the slump flow of the geopolymer mix decreased when alum sludge was increased in the mix for paste and mortar, respectively. Hence, with increased replacement content with alum sludge up to 60%, the slump flow decreased. In contrast, the results indicated that the replacement of fly ash with alum sludge from 10% to 30% showed a slight decrease in slump flow for both paste and mortar when compared with the control sample (FA0). This is an indication that these replacement content (FA0, FA1, FA3, MA0, MA1, MA2, and MA3) have more viscosity and flowability than other replacement content in the mix. The mix with only fly ash for both geopolymer paste and mortar showed a higher flowability. This could be because alum sludge has more absorbing capacity than fly ash [6,35]. Thus, when alum sludge replacement content increases in the mix, the absorption capacity increases due to higher dust content in the mix that was not fully consolidated by the alkalis. However, a slight decrease in flowability was observed in the result of geopolymer mortar from the control mix (MA0) to (MA2). The experimental result presented shows that all geopolymers mixed with alum sludge as a substitute to fly ash had no sign of bleeding or segregation during the casting process as all the materials were properly blended and flowable as shown in Figure 3. However, it is noticed in Figure 4 that the alum sludge had a slight variation in the flowability of produced geopolymer materials (paste and mortar) at higher replacement content.

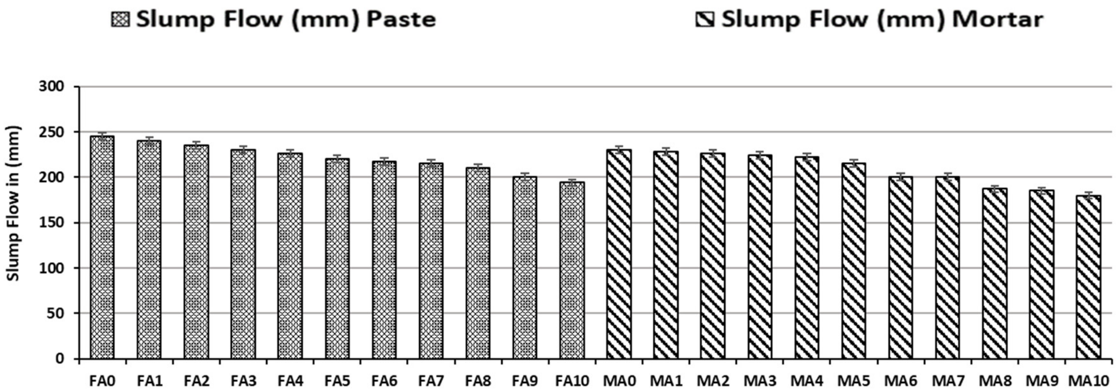


Figure 4. Effect of alum sludge-fly ash geopolymer paste/mortar on flowability.

Furthermore, it was observed that the addition of alum sludge as a replacement material to fly ash yielded an increase in the setting time of the geopolymer paste. For example, the control sample (PA0) had an initial setting time of 168.4 min and a final setting time of 257.6 min. In contrast, when alum sludge was introduced to the mix, the initial setting time increased to 175.6 min and the final setting time increased to 268.2 min. The same behavior was also seen in all mixes with alum sludge as a replacement for fly ash.

3.2. Density of Alum Sludge-Fly Ash Geopolymer Materials

The evaluation of density on geopolymer paste using alum sludge as a substitute to fly ash was studied and presented in Figure 5, while the density of geopolymer mortar is shown in Figure 6. All samples were evaluated for different curing ages 1, 7, 28, and 180 days. The curing of produced geopolymer material at an age of about 180 days was performed to ascertain the behavior in the density of alum sludge fly ash-based geopolymer samples at a long-term curing age (180 days). From the graph shown in Figure 5, the density of alum sludge-fly as geopolymer paste increased with an increase in curing age. This indicates that as the age of curing progresses the hydration of geopolymer paste and mortar progressively improved, and improved the packing effect of the internal structure due to the fineness of the alum sludge. Subsequently, the geopolymer samples increased due to the substitution of fly ash with alum sludge for replacement mix up to 50%. This behavior could be because alum sludge fine particles progressively filled the void in the internal structure of the geopolymer samples, and also improved the density of the geopolymer paste and mortar. It was also observed that an increased content of alum sludge from 60% to 100% in replacement of fly ash showed a decrease in density. This decrease can be attributed to the high dust content of alum sludge at replacement content above 50% for both geopolymer paste and mortar.

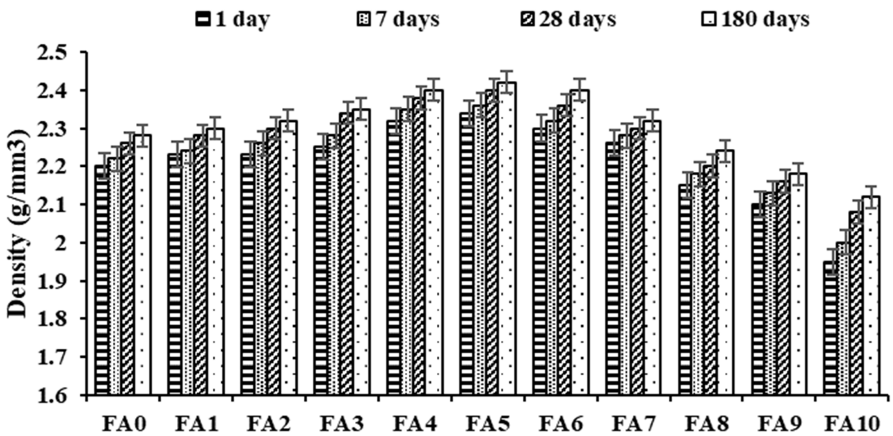


Figure 5. Density of alum sludge-fly ash geopolymer paste.

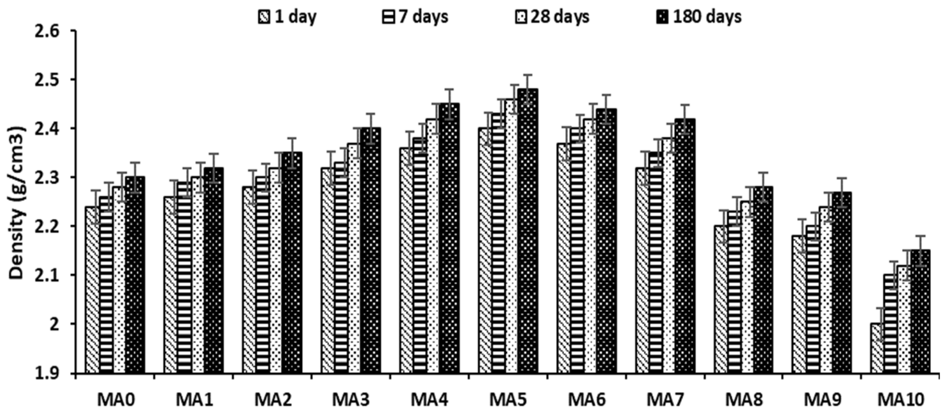


Figure 6. Density of alum sludge and fly ash-based geopolymer mortar.

A similar behavior was found in the case of geopolymer mortar from Figure 6, and the results indicate that the density of mortar improved (increased) with an increase in curing age and with replacement content from 10% to 50%. In contrast, an increase in alum sludge content above 50% attracted a reduction in the density of mortar. However, this is to state that the high substitute content of alum sludge reduced the density of both paste and mortar.

From Figure 6, it is seen that the decrease in density is because of the increase in dust content in the mix (for both geopolymer paste and mortar) and due to the lower specific gravity of alum sludge when compared with fly ash. Meanwhile, from 10 to 50 percent replacement content, it was observed that alum sludge improved the density of both paste and mortar.

3.3. Behavior of Alum Sludge-Fly Ash Geopolymer Paste and Mortar on Compressive Strength

Figures 7 and 8 show the compressive strength of geopolymer paste and mortar at different curing ages, with different replacement content of fly ash with alum sludge. It was observed that the replacement of alum sludge up to 50% significantly increased the compressive strength. The results also showed that the compressive strength of the paste increased with an increase in curing age. For example, on day 1, the strength of the geopolymer paste of the control sample was 24.55 MPa and at 7 days the strength increased to 28.42 MPa, then when the curing age was increased from 28 days to 180 days the strength increased progressively from 35.7 MPa to 40.8 MPa, respectively. A similar increase in strength due to curing age was also reported by Lee et al. [8] and Saludung et al. [12]. It is also seen that the replacement of fly ash with alum sludge up to 50% yielded a positive behavior in compressive strength. Hence, 50% replacement content of alum sludge blended with fly ash is the optimum replacement content in geopolymer paste.

However, the compressive strength behavior of geopolymer paste was somewhat similar to the compressive strength behavior of the geopolymer mortar as presented in Figure 8. For geopolymer mortar, it was observed that the strength also increased with an increase in age of curing for every replacement content, but decreased when replacement content was increased (increase in alum sludge above 50%). For example, the compressive strength of the control sample (MA0) at 1 day of curing was 19.55 MPa, while the geopolymer sample at 50% replacement content of fly ash had a strength increase of 40.6 MPa. The increase in compressive strength of the produced geopolymer mortar might be attributed to the concentration of the alkali solution in the mix [12,24]. This indicates that proper concentration and blending of alkali solution with suitable molarity is another defining factor in the compressive strength of geopolymer mortar. It has been established in past that 10 to 15 molar ratios yield more improved workability and strength of geopolymer [25].

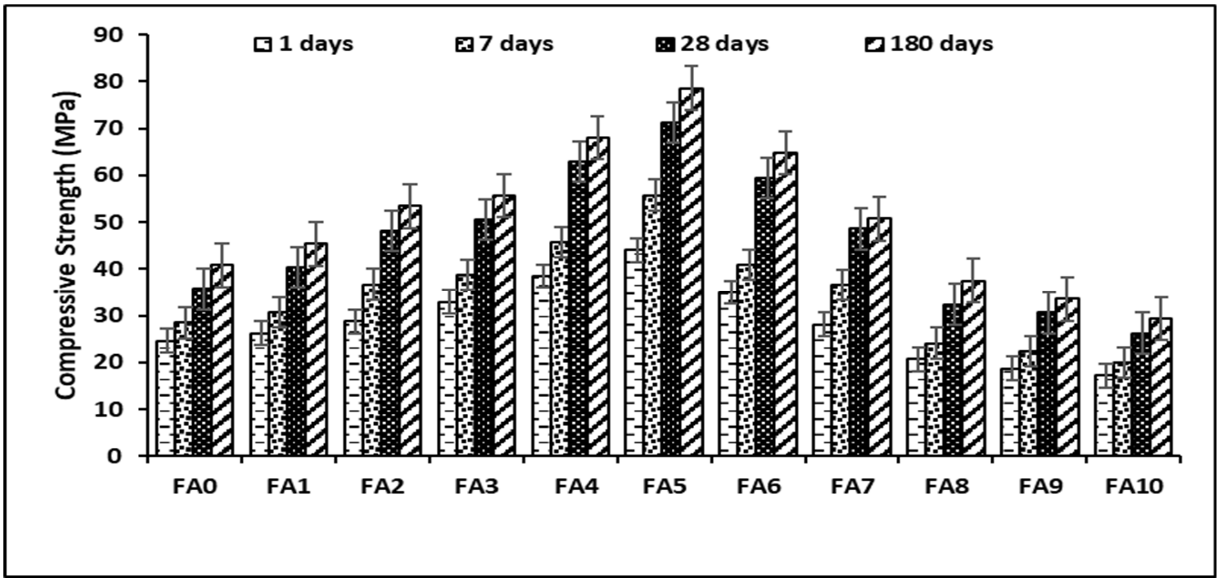


Figure 7. Effect of alum sludge -fly ash on the strength of geopolymer paste.

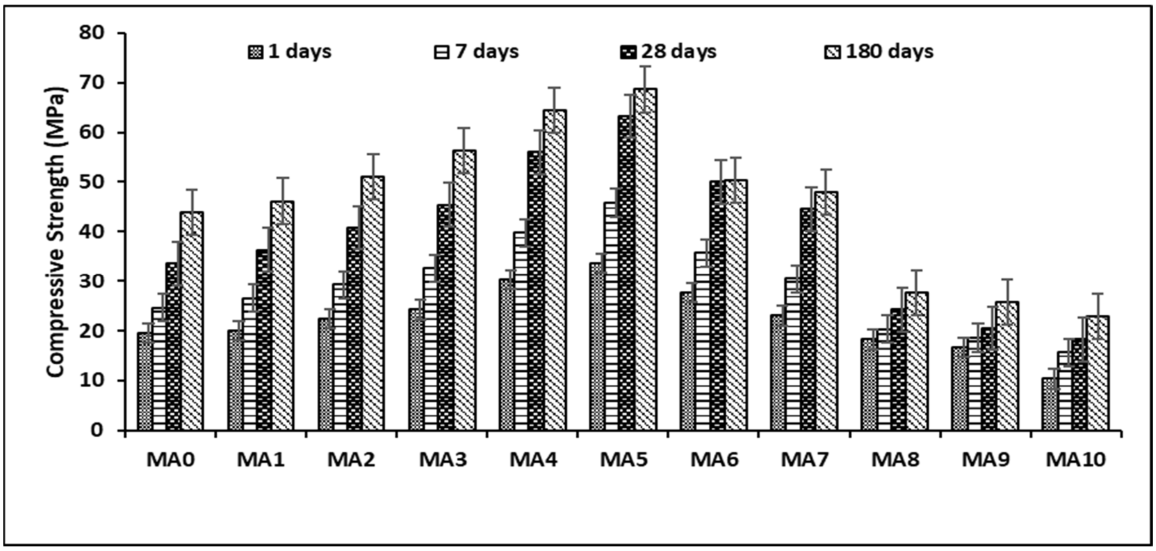


Figure 8. Behavior of alum sludge-fly ash on the strength of geopolymer mortar.

Another factor that contributed to the strength increase in geopolymer mortar was the reaction that enhanced the bond between the alkali solution with Si and the additional release or supply of aluminum (AI) by alum sludge. This reaction contributed to the polymerization of the mix that yielded an improved bonding between the constituent materials and increased the compressive strength of the geopolymer mortar [12]. It is seen from the behavior that the presence of Si and AI in alum sludge formed a denser structure that led to an improved packing effect of the geopolymer mortar, and hence, improved the strength of the geopolymer mortar [2,23]. Meanwhile, the increase in compressive strength when the alum sludge content was increased (replacing fly ash) indicates that the addition of alum sludge at an increased proportion released alumino-silicates which aid in significant improvement of the geopolymeric structure of the mortar. There was a more significant increase in compressive strength of the geopolymer mortar with alum sludge at 50% content and fly ash at 50% (equal content). This explained that both alum sludge and fly ash at this content increased the supply of alumino-silicates that promoted a dense geopolymer matrix and strength [13]. This behavior was obvious when the reference sample was compared with other replacement content. The result presented showed that

50% replacement content of alum sludge is the optimum replacement of fly ash with alum sludge, as this was also observed from the results presented in Figure 7 (geopolymer paste).

Figure 9 shows the pattern of failure for the geopolymer mortar specimen for MA5 (50% alum sludge replacement) after compressing the sample. It is seen from Figure 9 that the failure crack pattern was developed on the sides of the cube samples without any visible damage on the top and bottom of the cube sample touching the bottom and top plate of the compressive strength machine. Geopolymer mortar samples with alum sludge replacing fly ash and samples with fly ash displayed distributed cracks. No form of an explosive pattern of failure was observed, and all cracks were vertical in appearance with an hourglass shape. This failure pattern was observed in all the geopolymer paste and mortar samples in all curing ages.

Figure 9. Failure pattern of geopolymer mortar specimens.

3.3.1. Increase in Compressive Strength due to Alum Sludge Content

Figures 10 and 11 represent the effect and behavior of alum sludge on the increase in the strength of geopolymer paste and mortar. From the graph presented in Figure 10 (geopolymer paste), it was observed that the rate of strength increase was progressive even after 28 days, which showed a sign of a chemical effect (pozzolanic effect) due to the slow and constant release of increased concentration of silica and aluminum in the internal structure of geopolymer paste. This process yields a firm geopolymerization by forming a stronger bond due to the rich reactive aluminosilicate in both constituent materials (fly ash and alum sludge) that were dissolved in the alkali solution [13]. This behavior was significant in the sample with alum sludge replacement contents of 40, 50, 60, and 70 (MA4, MA5, MA6, and MA7), respectively. The graphs from both Figures 10 and 11 show a decrease in the compressive strength when the alum sludge content was above optimum. At this stage, the rate of decrease in the compressive strength increased in both replacement content and curing age.

The results presented showed an improvement in geopolymer paste and mortar that is within the optimum content, which is entirely influenced by alum sludge that was employed in the mix. This means that the optimum content of alum sludge to replace fly ash in geopolymer paste and mortar forms a stronger bond that progressively increased above 28 days of curing. However, for geopolymer mortar, the rate of strength increase was higher at 28 days when compared to the other curing ages, as seen in Figure 11. The rate of strength increase was higher for the mix with alum sludge when compared with the mix without alum sludge.

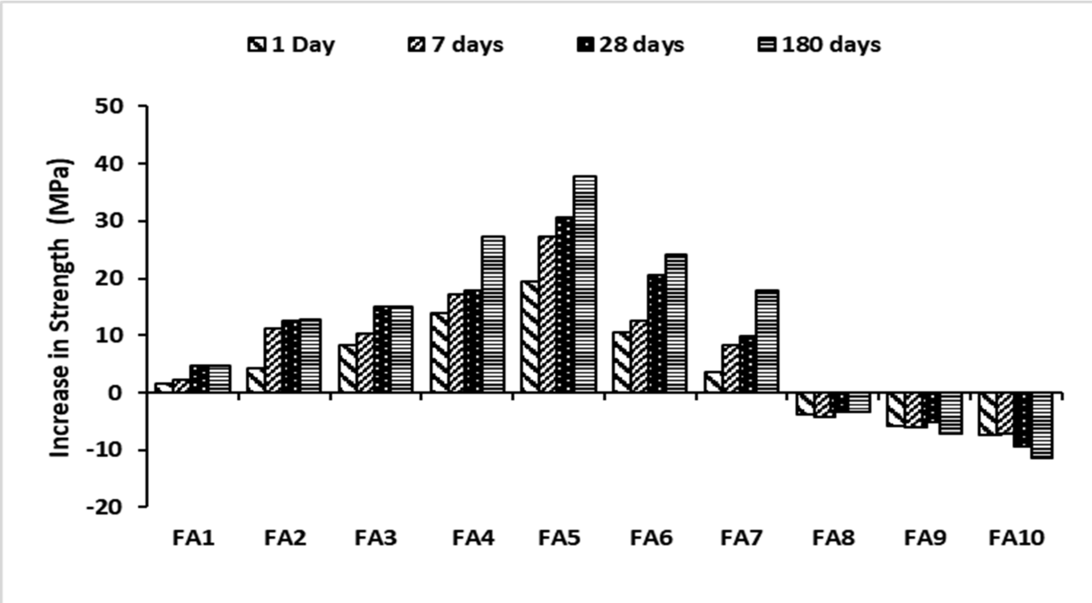


Figure 10. Effect and behavior of alum sludge on strength increase of geopolymer paste.

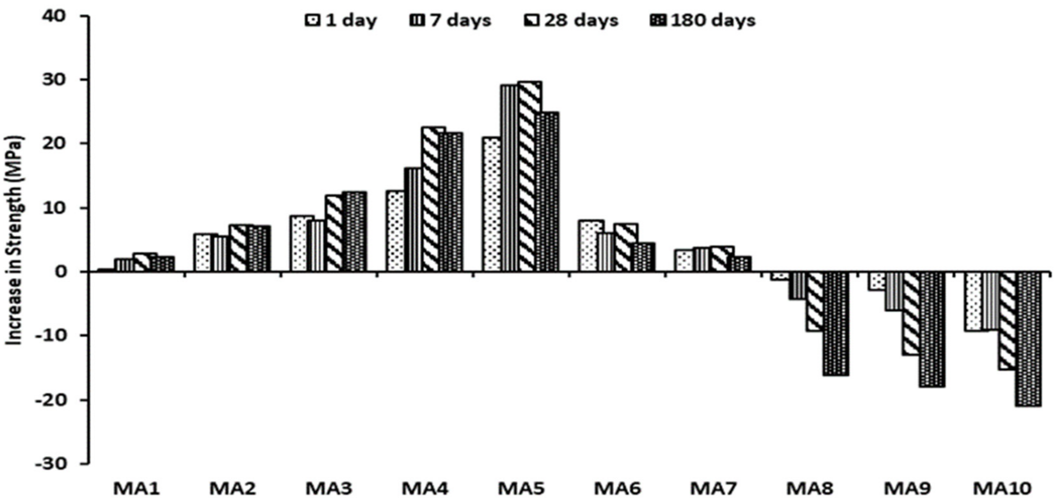


Figure 11. Increase in compressive strength of geopolymer mortar due to alum sludge.

3.3.2. Percentage Increase in Compressive Strength due to Alum Sludge Content

Figures 12 and 13 present the results for the percentage increase in strength for geopolymer paste and mortar. These results were derived by calculating the percentage increase in strength between the control sample (FA0, MA0) and other replacement content in the mix for both geopolymer paste and mortar. From the results, it was observed that the addition of alum sludge to the mix aided the percentage increase for both paste and mortar, respectively, with a significant percentage increase at 28 days. The behavior was observed in both geopolymer paste and mortar at 28 days curing age; for example, geopolymer paste FA1 at 1 day curing age had a percentage increase of 6.7%. However, when the curing age progressed to 7 days, 28 days, and 180 days, the percentage increase became 7.9%, 13.4%, and 11.3%, respectively. This indicates that a significant increase occurred at 28 days of curing age, but the percentage rate of increase decreased when the curing age progressed to 180 days when compared with the percentage increase at 28 days of curing age.

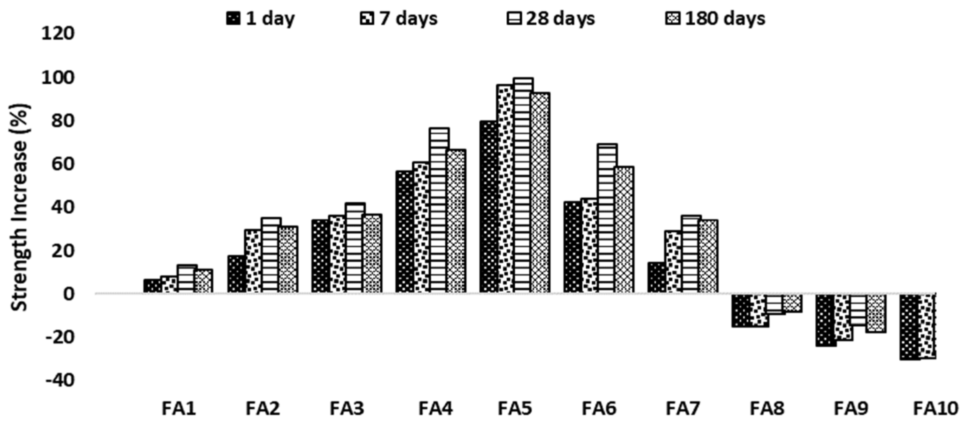


Figure 12. Percentage increase in strength of geopolymer paste due to alum sludge.

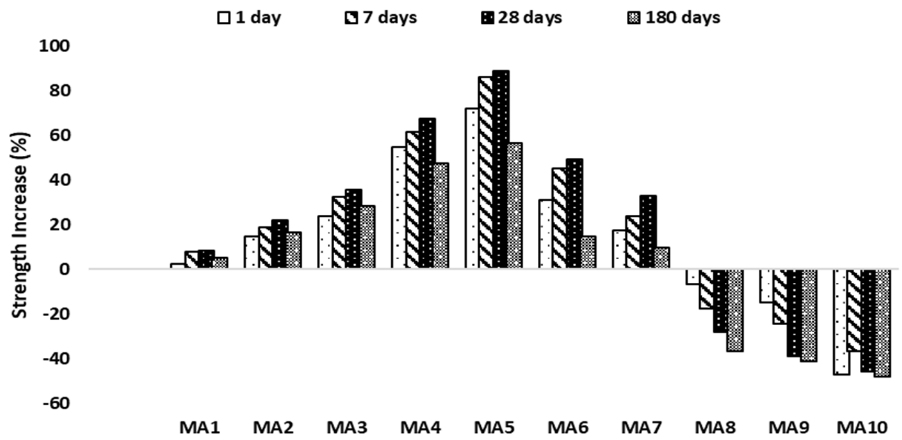


Figure 13. Percentage increase in strength of geopolymer mortar due to alum sludge.

From Figure 12, the results showed that the percentage strength increase progressively increased when curing age increased, and this was observed in both geopolymer paste and mortar. Alum sludge replacement content of 50% for geopolymer paste and mortar (FA5 and MA5) showed the highest percentage increase at all curing ages when compared to the control sample and other replacement content in the mix. This behavior shows that 50% replacement of fly ash with alum sludge is the optimum replacement content in the mix.

At replacement content of 80% (FA8 and MA8), a decrease in percentage increase was recorded and the decrease became significant when the alum sludge content increased from 80% to 100%. However, the negative percentage strength value showed a decrease in the strength of both the geopolymer paste and mortar at replacement content of 80% to 100%. For example, 70% (FA7) replacement of fly ash with alum sludge at 28 days had a percentage strength increase of 28.9%, but when the replacement content was increased to 80% (FA8), 90% (FA9), and 100% (FA10), there was a significant decrease in strength with percentages −15.2%, −21.5%, and −29.6%, respectively. This behavior was also observed in similar replacement content of geopolymer mortar. This implies that alum sludge replacement content above 70% attracts a significant decrease in compressive strength.

3.4. Water Absorption of Alum Sludge-Fly Ash Geopolymer Paste and Mortar

Investigation of water absorption by geopolymer paste and mortar was necessary for the evaluation of the behavior and effect of alum sludge as a substitute to fly ash on the produced paste and mortar. The study of water absorption is very important, especially when a new sample is introduced in the production of any construction material. The test was performed on samples at 1 day, 7 days, 28 days, and 180 days. Figures 14 and 15 present the results for geopolymer paste and mortar with alum sludge partially and fully replacing fly ash in different curing ages. From the results, it was detected that there was a decrease in water absorption when the curing age was increased and this behavior was observed in all replacement contents. A progressive reduction in water absorption with an increase in alum sludge content was also detected. Alum sludge at 50% replacement showed improved resistance to water absorption, compared to the control sample and the other replacement content (FA0 and MA0) for both paste and mortar samples. For instance, geopolymer paste FA0 at a 1 day absorption rate was 6.2% while FA5 at a 1 day absorption rate was 4%, respectively, which is the lowest absorption rate when compared to other samples in this study. However, the decrease in absorption could be related to the addition of alum sludge as a substitute to fly ash which improved the geopolymer structure by reducing the void within the internal structure of the samples. Another factor could be due to the improvement of density and compact structure by alum sludge at optimum replacement content that produced a new formation of aluminosilicate gel which enhanced the filling effect by the materials. This can also be related to the improved compressive strength as reported in Section 3.2 for both geopolymer mortar and paste.

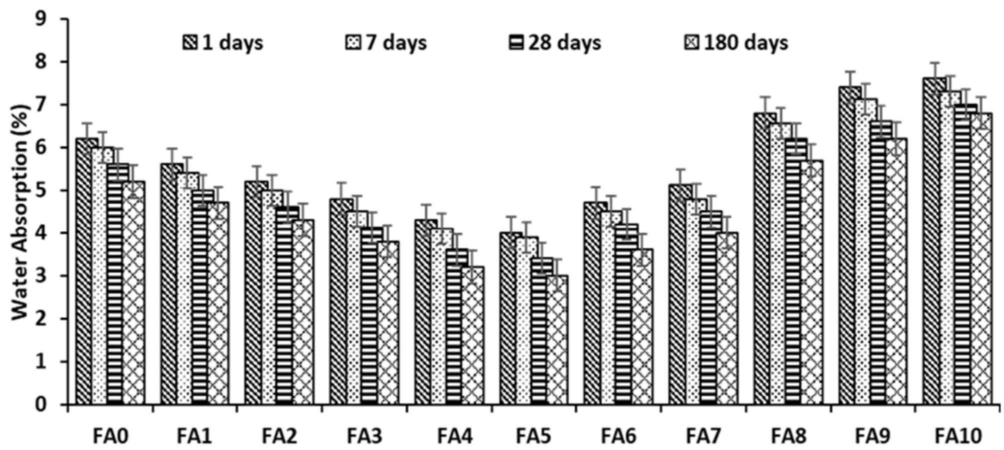


Figure 14. Behavior of alum sludge on water absorption of geopolymer paste.

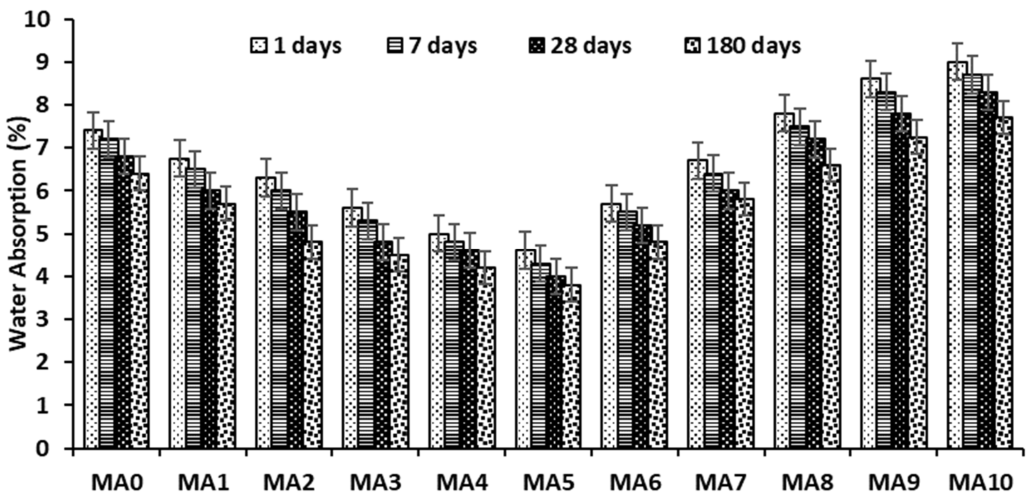


Figure 15. Behavior of alum sludge on water absorption of geopolymer mortar.

The results presented also indicate that water absorption increased with increases in the replacement of fly ash with alum sludge above 50%. It was seen that alum sludge with replacement content up to 100% yielded a higher rate of absorption for both geopolymer paste and mortar, with an absorption rate of about 6.8% for paste and 7.7% for mortar, as presented in Figures 14 and 15. This behavior could be related to the increase in dust content in the mix that could have caused the alkali not to consolidate all the materials and thus, affect the geopolymerization process that caused the internal structure of the sample to have a higher porous structure and higher water absorption behavior.

3.5. Behavior of Geopolymer Paste and Mortar due to Elevated Temperature

Geopolymer samples were subjected to elevated temperatures of 400 °C and 600 °C and studied using two methods; loss in weight and loss in compressive strength. These investigations were performed on geopolymer samples at 28 days of curing age when the geopolymer samples were assumed to have attained their strength. However, there are limited findings in this aspect of the durability test on geopolymer paste or mortar. Therefore, this was used as a yardstick to investigate geopolymer paste and mortar manufactured with alum sludge as a substitute to fly ash. Samples produced in this study were transferred to the electric furnace for an elevated temperature test for 1 h. This was performed for all the desired temperatures adopted in this study, and then, after 1-h heating, the strength of the sample was compared with the original strength of all samples (paste and mortar sample) before heating. This test was necessary to establish the effect of elevated temperature on the geopolymer paste and mortar, and further understand the resisting level of the geopolymer paste and mortar to high temperatures. Thus, this investigation will assist in the design of geopolymer paste and mortar where its structural member can resist high temperatures that could affect the durability/integrity of the paste and mortar with time.

3.5.1. Weight Loss due to Elevated Temperature on Geopolymer Paste and Mortar

Figures 16 and 17 present the results of weight loss for geopolymer paste and mortar manufactured using alum sludge as a substitute to fly ash. From the presented graph (Figures 16 and 17), it can be seen that the weight loss of both geopolymer paste and mortar increased due to an increase in elevated temperature. Meanwhile, a different behavior was observed when the replacement material (alum sludge) was utilized as a substitute to fly ash for the production of geopolymer paste and mortar. The results indicated that the loss of weight reduced with an increase in alum sludge up to 70% but increased in weight loss at replacement content above 70%. From Figure 14 for instance, FA0 (control sample) had a weight loss of 2.18 g/cm^3 when the temperature was elevated to 400 °C but when it was increased to 600 °C, the weight loss increased to 3.4 g/cm^3. However, it was seen that alum sludge employed in the mix as a substitute to fly ash for FA, yielded a slight weight reduction (2.15 g/cm^3) at a temperature of 400 °C and when the temperature was increased to 600 °C, the weight loss was 3.37 g/cm^3. This behavior affirmed that there is an improvement in resistance to loss of weight when alum sludge was utilized in optimum content as a substitute to fly ash for the production of geopolymer paste and mortar. Moreover, when alum sludge was added to the geopolymer mix above optimum (70%), a progressive increase in loss of weight was observed in FA8 when compared with FA0 and FA5. With a weight loss of about 2.25 g/cm^3 and 3.46 g/cm^3 for the elevated temperature of 400 °C and 600 °C, respectively, while for FA5, weight loss was seen to be 1.16 g/cm^3 at 400 °C and 2.16 g/cm^3 for temperature elevated up to 600 °C. This behavior by geopolymer paste and mortar using 50% alum sludge as a substitute to fly ash (FA5 and MA5) is an indication that blending alum sludge in an optimum content significantly improved the resistance to loss of weight when subjected to elevated temperature.

Geopolymer paste and mortar produced with alum sludge showed better resistance to loss of weight than the produced sample with only fly ash but when alum sludge was increased up to 70% as a substitute to fly ash, an increase in loss of weight was observed.

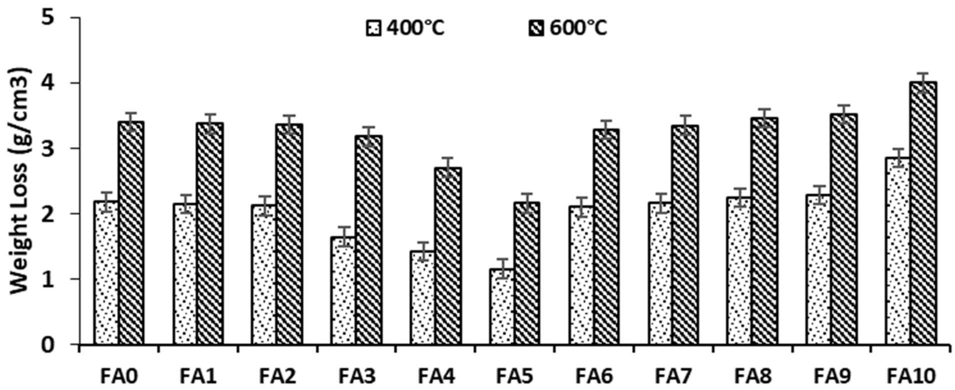


Figure 16. Geopolymer paste weight loss due to exposure to elevated temperature.

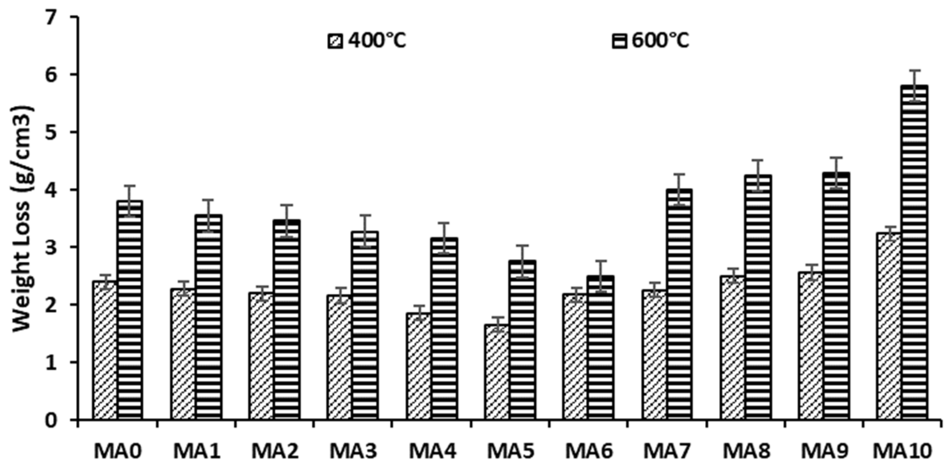


Figure 17. Geopolymer mortar weight loss due to exposure to elevated temperature.

3.5.2. Strength Loss due to Elevated Temperature for Geopolymer Paste and Mortar

Strength loss due to elevated temperature for geopolymer paste and mortar is presented in Figures 18 and 19. The results from this experiment indicated a decrease in compressive strength when the temperature was increased. This decrease in strength was due to exposure to elevated temperatures of 400 °C and 600 °C. It was observed that loss of strength was less at 400 °C when the loss in compressive strength was compared to the strength loss at 600 °C. However, the behavior of the manufactured geopolymer paste and mortar showed high strength loss at an exposure of temperature up to 600 °C than the samples subjected to a temperature of about 400 °C. This means that a temperature of about 400 °C showed no significant effect on the strength loss of geopolymer paste and mortar. For example, at an elevated temperature of 400 °C, FA0 and MA0 strength losses were 3 MPa and 3.75 Mpa at 400 °C, but at 600 °C the strength loss was 3.8 MPa and 5.83 MPa, respectively. For FA5 and MA5, strength loss was 0.8 MPa and 1.6 MPa, while at 600 °C temperature, strength loss was 1.4 MPa and 2.15 MPa. Additionally, the decrease in resistance to strength loss due to elevated temperature of geopolymer samples with alum sludge as a substitute to fly ash compared with control samples (FA0 and MA0), shows that the resistance to strength loss decreased progressively with an increase in alum sludge content. This performance was found in all geopolymer samples manufactured with alum sludge as substitute material to fly ash for both paste and mortar. Thus, this means that geopolymer paste and mortar with alum sludge as a substitute to fly ash yielded low resistance to relative loss in strength due to elevated temperatures above 400 °C. This was except for geopolymer paste and mortar above 70% replacement content (FA7 and MA7), which was seen to have a progressive increase in loss of strength (lower resistance to compressive strength loss).

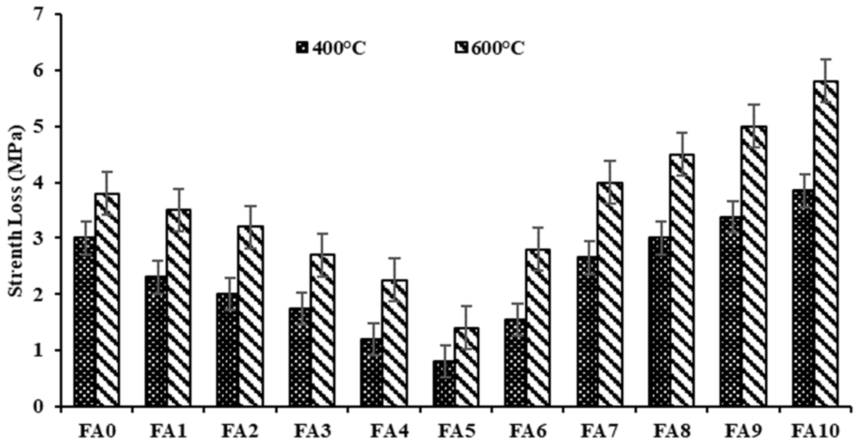


Figure 18. Strength loss of geopolymer paste due to elevated temperature.

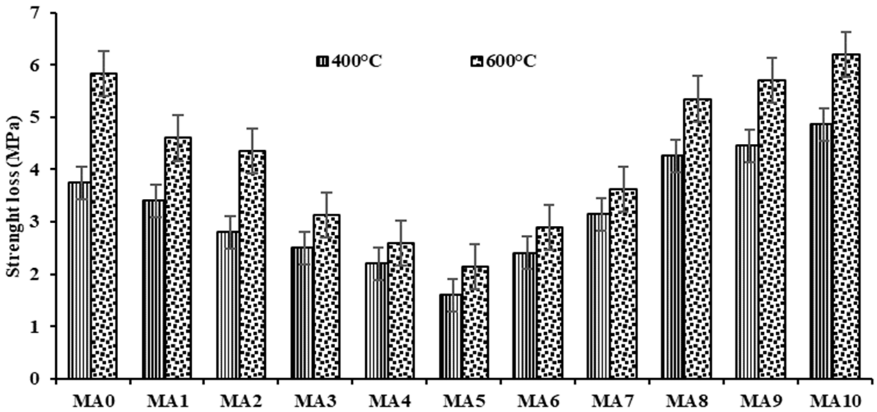


Figure 19. Strength loss of geopolymer mortar due to elevated temperatures.

For example, FA8, FA9, and FA10 for geopolymer paste at 400 °C lost 3 MPa, 3.38 MPa, and 3.85 MPa, meanwhile, at 600 °C, geopolymer paste lost 4.5 MPa, 5 M Pa, and 5.8 MPa, respectively. There is clear evidence of a higher loss in strength compared to the loss of strength for the control sample (FA0) and other replacement content. This behavior was also observed in geopolymer mortar samples in this study. On the other hand, there is clear evidence that the optimum content of alum sludge as a substitute to fly ash for the production of geopolymer paste and mortar subjected to elevated temperatures showed higher resistance to compressive strength loss in both temperatures (400 °C and 600 °C). This behavior can be related to the improved performance of density and compressive strength properties as reported in Sections 3.1 and 3.2 in this study.

4. Conclusions

This paper presents the behavior of alum sludge as a substitute to fly ash for manufacturing geopolymer paste and mortar on the workability, density, compressive strength, water absorption, and elevated temperature. The following conclusions were drawn based on the experimental work and results discussed:

I. Replacement of fly ash with alum sludge has proven to yield a flowable mix that can be used where flowability is needed, although the mix with only fly ash had the highest value of slump flow.

II. From the performance of geopolymer paste and mortar based on density and strength properties, it was seen that the normal curing process adopted in this study did not affect the geopolymer samples negatively. The density of geopolymer paste and mortar increased with an increase in alum sludge as a substitute to fly ash. Alum sludge within optimum replacement content showed higher density compared to other samples in the study by forming a stronger and denser geopolymer structure.

III. The compressive strength of geopolymer paste and mortar increased with an increase in alum sludge up to 50% and decreased with an increase in alum sludge above 50%. The optimum replacement content of fly ash with alum sludge was 50%, which gave the highest strength compared to other geopolymer mixes in this study.

IV. Both alum sludge and fly ash contributed immensely to improving the strength development of both geopolymer paste and mortar. This was due to the availability of SiO_2 and Al_2O_3 from both alum sludge and fly ash that reacted with the alkalis to produce a stronger bond between the raw materials.

V. The replacement of fly ash with alum sludge yielded a decrease in water absorption. Thus, the water absorption decreased with an increase in alum sludge content up to 50%, while the increase in alum sludge above 50% increased the rate of water absorption.

VI. The weight loss and strength loss due to elevated temperature decreased as alum sludge partially replaced fly ash up to 50% in both geopolymer paste and mortar. Meanwhile, alum sludge content above 50% yielded an increased weight loss and strength loss of geopolymer paste and mortar.

VII. The optimum replacement content of fly ash using alum sludge was shown to be 50% which gave the best improvement results for density, compressive strength, water absorption, and elevated temperature.

The experimental results affirm that the optimum content of alum sludge to substitute fly ash employed in this research is the influential parameter that yields the improved mechanical properties of the geopolymer paste or mortar. The improvement in density, compressive strength, water absorption, and elevated temperature were seen to be due to the addition of alum sludge in the mix at optimum content. Thus, it is important to conclude that blending industrial waste materials such as alum sludge and fly ash can produce an eco-friendly cementing material for environmental sustainability.

From the results and findings obtained from this investigation, it is recommended that further durability studies (sulphate attack and chloride attack on geopolymer mortar) should be carried out using these materials (alum sludge and fly ash). This should be done to understand the behavior of fly ash-based geopolymers replaced with alum sludge when exposed to a chemical environment and the ability to resist sulphate and chloride attack. Different percentage replacements should be adopted to investigate the compressive strength, water absorption, and elevated temperatures of alum sludge as a replacement for fly ash-based geopolymer paste and mortar.

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