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Research Article

Analyzing Influence of Mix Design Constituents on Compressive Strength, Setting Times, and Workability of Geopolymer Mortar and Paste

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Geopolymer concrete and mortar have evolved over the years as potential alternatives for reducing the greenhouse gases associated with cement production. This current research was aimed at investigating the optimum dosage and concentration of sodium hydroxide required to leach out silica and alumina oxides in the fly ash for geopolymerization to take place. Blackish grey fly ash from Morupule, Botswana, was synthesized by varying sodium hydroxide (NaOH) of 98% purity between 8 M and 14 M, respectively. The ratio influence of sodium hydroxide to fly ash in dissolving the oxides was carried out at the values of 0.55, 0.62, and 0.75. The results showed that the workability of the geopolymer mortar and paste decreased with the increase in the ratio of fly ash to alkaline activator. The highest workability was achieved at a ratio of 0.75 : 1. The compressive strength, setting time, and workability of geopolymer mortar and paste can be controlled by adjusting the ratio of fly ash to alkaline activator. A ratio of 1.5 : 1 was found to be the most suitable for achieving high compressive strength, while a ratio of 0.75 : 1 was found to be the most suitable for achieving high workability. Furthermore, the workability values were in the range of 105 to 143 mm, while the ranges of initial and final setting times were found to be between 280–350 and 950–1170 minutes, respectively. This study is significant because no previous study has carried out geopolmerization of the Morupule fly ash as a result of its unique characteristics. These findings have important implications for the development of sustainable construction materials. The main finding was that for optimum reaction to take place, and NaOH/fly ash ratio should be kept at 0.55 and molarity of 12 to avoid leaching of other oxides that might weaken the strength.

1. Introduction

Environmental sustainability has been the key issue in recent years as jointly agreed and contained in Paris agreement by countries' members that global warming should be limited to 1.5°C for the planet earth not to exceed her carrying capacity. For this lofty goal of 45% emission reduction to be achieved by the year 2030 and net zero by the year 2050, all hands must be on desk and paradigm shift in engineering services, construction, production, and consumption cannot be overemphasized. The adverse effects of human activities on the climate change have not experience any meaningful

decline and Intergovernmental Panel on Climate Change (IPCC) [1] as had reported that in order to achieve net zero by the year 2050, there is a need for drastic measures to limit global warming.

Researchers have validated that fly and bottom ashes are pozzolanic in nature and had been widely used for partial replacement of cement which is an intervention in reducing the global warning from the cement industry. Reference [2] published a novel work on geopolymerization of fly ash which he called inorganic polymers; several scholarly works have emerged and proved that fly ash-based geopolymer concrete and mortar are possible provided the material is either class C or F. The side effects of cement production as reported by foremost researchers recorded that 7% of worldwide CO_2 emission comes from cement production [3, 4].

Cement production is known to be environmentally unfriendly, energy intensive, and is a source of greenhouse gas emission. As reported earlier, about 7-8% of carbon pollution comes from cement production only. This has always come in two phases; breaking of limestone ($CaCO_3$) leads to the generation of CO_2 [5]. Some researchers have argued that about 55% of one ton of CO_2 that comes from one ton of cement comes directly from this, with the remaining 45% from fossil fuel burning during the clicker production. In order to reduce this amount, fossil fuel residue has been proved over time to be a good source for supplementary cementitious material. Hence, reusing this will invariably reduce CO_2 emissions and also increase clicker production to about 30–40% [6]. Furthermore, concrete produced from Portland cement is known to be prone to durability-related problems after some time most especially in the corrosive environment. Issues of alkali-silica reaction are another constraint due to the reaction between the alkaline in cement and silica in some aggregate which in turn leads to spalling, expansion, and cracks over the time.

Going forward, geopolymer paste and mortar are products of base materials and alkaline activators. The base material could be from natural sources such as clay or artificial which include agricultural or industrial by-products such as rice husk, palm oil fuel ash, slag, and fly ash among others. Alkaline activators are predominantly soluble alkali materials in which sodium hydroxide, potassium hydroxide, and sodium silicate have been widely used [7]. Literature has affirmed that physical properties of the base materials have significant influence on the performance of geopolymer paste and mortar. Properties such as particle size, specific surface area, and specific gravity play a huge role on the fresh and hardened properties of geopolymer products. In other reports, class of the fly ash, morphology of the fly ash, amorphous content, loss of ignition, and other oxides should be within the specified range [8].

Geopolymer paste, mortar, and concrete have been reported to possess early and high strength over conventional Portland cement concrete [9, 10], low or little shrinkage [11, 12], better sulfate resistance [13, 14], good acid resistance [15], good resistance to freeze-thaw and fire resistance [16], and better durability performance [17], and there is no danger of alkali-aggregate reaction [18, 19].

In details, [20] researched on two combined industrial waste products for the manufacture of geopolymer paste (fly ash and Ground Granulated Blast Furnace Slag, GGBFS). The rationale behind the paper was to reduce the initial and final setting times which were significantly reduced as a result of high calcium oxide in GGBFS. It was observed that increase in concentration of NaOH and in quantity of GGBFS increased the compressive strength of the geopolymer paste with 7, 28, and 56 days of strength ranging between 66.4 and 78.2 MPa for 16 M, and with 50% GGBFS at alkaline activator/fly ash (AA/FA) of 0.4 and SS/SH of 1.0, [21] investigated the effect of replacing fly ash-based geopolymer paste with different proportions of ground fluorescent lamp glass and ground container glass. The AA/FA was kept constant at 0.6 with sodium silicate/sSodium hydroxide (SS/SH) of 1.0. With the different levels of replacements between 10 and 40 percentages, there was a marginal increase in the compressive strength compared to 100% fly ash. Four percent increase in the compressive strength was recorded with 20% ground container glass. Some of the microstructural analysis results revealed visible cracks accompanied with lower densities and number of unreacted particles among the mix constituents. The X-ray diffraction (XRD) result shows glassy matrix with a low crystalline phase of $CaCO_3$ and SiO_2 which suggest that the high level of geopolymerization [22] carried out investigation on high-calcium fly ash by activating the oxides with only [23] NaOH using 4.5, 7.0, 9.5, 12, 14, and 16.5 molarity at a constant alkaline activator to fly ash ratio of 0.3 and cured at room temperature. The increase in concentration of NaOH increased the compressive strength, and the maximum recorded strength was 25.5 MPa at the age of 60 days.

Reference [24] carried out effects of sodium hydroxide concentration, leaching period, mixing method, and ratio of water glass to sodium hydroxide on the dissolution of silica and alumina oxides from the fly ash. Results indicated that 10 M NaOH with a leaching period of 10 minutes yielded Si^{4+} ion concentration close to 600 ppm, while 5 and 15 M had low and reduced yield concentrations, respectively. The concentration of Al^{3+} ion in ppm was much lower to Si^{4+}. Also, microstructural analysis of geopolymer paste of 5 M revealed less attack on fly ash compared to 10 and 15 M. Gel formation in the form of colloid was seen around fly ash together with unreacted, partially reacted cracks and pores which could militate against the strength development.

In another development, the authors in [14] revealed that silica fume added to fly ash-based geopolymer concrete increased the concrete compressive, flexural strengths, and durability when tested in basic and acidic simulated conditions. Reference [25] reported that Si/Al ratio in both the precursor and activator played a significant role in the strength development of geopolymer paste and mortar. An early polymerization was noticed, and when the sand was varied between 20 and 40% of the fly ash, the compressive strength was 25.05 ± 1.35 MPa; he also reported that fluidity of geopolymer paste was higher compared to the geopolymer mortar when all the variables were constant; however, the

compressive strength of geopolymer mortar was greater than that of paste. The results of microstructural analysis of SEM and XRD revealed that reacted and unreacted samples possessed higher compressive strengths with dense and compacted products. Reference [26] investigates the potential of using micronized biomass silica (MBS) as a partial replacement of ground granulated blast furnace slag (GGBS) in geopolymer concrete (GPC). Various combinations of MBS and GGBS were used to prepare concrete, and compressive and split tensile strengths, sorptivity, and chloride permeability were tested up to 28 days. The results showed that a GPC mixture containing 20% MBS and the balance GGBS had the best performance in terms of strength and durability, with all mixtures exceeding the intended design strength. This study demonstrates the potential of MBS as a binder in GPC production, contributing to sustainable and greener development. Reference [27] study develops a high-strength repair material using metakaolin-based alkali-activated concrete (AAC) with 15% metakaolin. The AAC improves flexural strength by up to 14.4% and exhibits significant bonding with the concrete substrate with less shrinkage and improved strength.

The novelty of this study lies in several aspects. Firstly, the study focuses on geopolymer mortar made from fly ash from Morupule, a region that has not been extensively studied before. Secondly, the study explores the influence of various mix design constituents on the properties of geopolymer mortar and paste. Thirdly, the study also examines the microstructural analysis of the hardened geopolymer mortar, providing insights into the bonding mechanisms and the structure of the geopolymer binder. Finally, the study takes a comprehensive approach to understanding the behavior of geopolymer mortar, providing a comprehensive understanding of the relationship between mix design constituents and the properties of geopolymer mortar and paste. Reference [28] studied geopolymers, with aluminum calcium cement and nanometric titanium oxide additives being tested for leaching of alkali. Results showed lower efflorescence risk and decreased leaching with calcium aluminum cement, and higher leaching with 8 M NaOH was compared to 10 M. Compressive strength tests were also conducted [29]. A study was conducted on effects of cesium dosage and curing time on leaching behavior and immobilization of Cs in NaOH-activated fly ash-based geopolymers. Cs dosage (2–20 wt. %) and curing time (7–180 days) were evaluated for leaching behavior and the leachability index (LX). Results show that in-situ pollucite crystallization enhances Cs immobilization and diffusion is a primary leaching mechanism. FA-GP was recommended for effective immobilization of Cs [30]. Geopolymer composites with bisphenol F epoxy resin were produced to investigate the effect on alkali leaching and mechanical performance. Results show that epoxy resin enhances mechanical properties and reduces alkali leaching, promising for geopolymer in construction.

1.1. Research Significance and Objective. The objective of this study is to investigate the influence of mix design constituents on the properties of geopolymer mortar and paste, with the aim of determining optimal mix proportions that result in high compressive strength, acceptable setting times, and good workability. The study focuses on analyzing the behavior of geopolymer binder derived from Morupule fly ash, taking into account parameters such as precursor characteristics, alkaline activator, curing mode, and period. To achieve this objective, twelve design mix proportions were formulated and the effect of the concentration and molarity of the activator (sodium hydroxide), as well as the ratio of sodium hydroxide to fly ash at a constant fly ash to sand ratio, was investigated. The study aims to appraise the influence of fly ash characteristics on the fresh and hardened properties of geopolymer mortar, assess the setting times and flow-ability properties of the geopolymer paste and mortar, determine the mechanical behavior of geopolymer mortar at different concentrations of sodium hydroxide and varying alkaline liquid-fly ash proportions, and examine the microstructural analysis of the hardened geopolymer mortar. Geopolymer mortars and pastes have shown great potential as an alternative to Portland cement-based materials, with the advantage of utilizing waste materials such as fly ash as a precursor. However, the properties of geopolymer materials are highly dependent on mix design constituents, and an optimized mix design is essential to ensure optimal performance. The study aims to contribute to the understanding of the relationship between mix design constituents and the properties of geopolymer mortar and paste. By investigating the influence of fly ash characteristics, alkaline activator concentration, and ratio of activator to fly ash, the study aims to provide insights into how these factors affect the fresh and hardened properties of geopolymer materials. The investigation of setting times and flow-ability properties of the geopolymer paste and mortar is important as these properties affect the workability and handling characteristics of the material. The mechanical behavior of geopolymer mortar at different concentrations of sodium hydroxide and varying alkaline liquid-fly ash proportions is crucial in determining the compressive strength of the material, which is a key indicator of its performance. Finally, the microstructural analysis of the hardened geopolymer mortar provides insights into the material's durability and long-term performance. By analyzing the microstructure of the material, the study aims to provide a better understanding of the relationship between mix design constituents and the material's properties. Overall, the objective of this study is to provide a comprehensive understanding of the influence of mix design constituents on the properties of geopolymer mortar and paste. By optimizing the mix design, the study aims to contribute to the development of sustainable and cost-effective construction materials that can reduce the environmental impact of construction activities. The study's findings could also have important implications for the utilization of waste materials in construction and could potentially lead to the development of new applications for geopolymer materials.

2. Experimental

2.1. Materials

2.1.1. Fly Ash. Fly ash used in this study was sourced from Botswana Power Corporation, BPC, Morupule Power Plant B in Palapye, Botswana located at 22.5515°S, 27.1147°E. The company collects its coal from Morupule Coal Mine, MCM, and burns coal of different sizes but of the sub-bituminous type. The arrangement of the plant with the connecting conveyor with Morupule coal mine and ash lagoon of the generated fly ash is shown in Figure 1. In addition, Morupule Plant B power station uses circulating fluidized bed boiler that runs at a temperature between 800 and 900°C unlike pulverized coal boiler at other plants and at Plant A of the same company. Before coal combustion, limestone is normally being added before feeding the coal into the boiler so as to stay within the safe limits of SO_2; this makes the properties of the fly ash different and unique from Plant A of the same company, and invariably, this largely determines the behaviour of geopolymer mortar and paste.

(1) Physical Properties of the Fly Ash. The specific gravity of the fly ash was carried out using the pycnometer test method according to [31], and the value was calculated using the relation as follows:

$$\text{Specific gravity} = \frac{M_2 - M_1}{(M_2 - M_1) - (M_3 - M_4)} \frac{\text{g}}{\text{cm}^3}. \quad (1)$$

The laser diffraction technique was employed for the determination of the particle size distribution. A paste of fly ash and water was formed in the Petri dish and was transferred into the water tank. The equipped laser of the machine captured the result, and the result was given in volume (percentage) against the sieve sizes.

(2) Chemical and Microstructural Analyses of the Fly Ash. The chemical analysis of the fly ash was scanned with the help of S8 Tiger Bruker X-ray fluorescence spectrometer. The fly ash was pulverized and sieved through 63 μm sieve. The sample was then placed in the PVC sample cup with propylene (4 μm–7 g) window film. The loss of ignition and mineral composition of the fly ash were determined according to [32] and using X-ray diffractometer of Empyrean PANalytical model. The morphology analysis of the fly ash was carried out with Carl Zeiss FEGSEM Gemini 500. The in-lens high resolution possesses the following test conditions: 3 kV, 3.5–4.5 mm working distance, and 20 μ aperture. The fly ash was premounted into SEM stubs while the images are taken at different magnifications.

2.1.2. Fine Aggregates. The sand used to determine the strength of the binder was prepared from local river sand and validated with the reference sand given in section 5.1.2 of [33]. The specific gravity of the sand was found to be 2.78 when it is in saturated surface dry condition.

2.1.3. Alkaline Activator. Sodium hydroxide (NaOH) is in flakes form with 97–100% purity and sourced from a local supplier in South Africa. The sodium hydroxide solution is prepared with distilled water for different molarities of 8, 10, 12, and 14 M at least 24 hours before use, and they are stored at the room temperature of 23 ± 2°C and relative humidity between 60 and 70%. For this study, only sodium hydroxide is adopted for the purpose of knowing potential of NaOH leaching capability.

2.2. Mixture Compositions of Geopolymer Paste and Mortar. The geopolymer paste and mortar were developed using water-cement analogy of conventional Portland cement which is replaced with three different alkaline activator/fly ash with ratios of 0.55, 0.62, and 0.75, respectively; the other lower alkaline activator/fly ratio mixes were not workable, and they are without any significant compressive strength results, and therefore, they were discarded for further investigation. The concentrations of NaOH used were 8, 10, 12, and 14 M, respectively, which were prepared by multiplying the molecular weight of NaOH of 39.997 g with respective moles and dissolved in one liter of distilled water. In all, twelve mix proportions were used as given in Table 1.

2.2.1. Geopolymer Paste and Mortar. The mixing of geopolymer paste was carried out mechanically inside the Hobart mixer following the modified method of section [34]. Fly ash was thoroughly mixed for two minutes for homogeneity at a low speed of 62 rpm. This was followed with the introduction of sodium hydroxide into the bowl, and mixing was continued for another four minutes. The machine was stopped for 30 seconds for the purpose of scraping the side of the bowl, and the process of mixing continued and completed for another two minutes at a high speed of 120 rpm. The geopolymer mortar was prepared with fly ash to sand ratio of 1.5. After mixing the fly ash at a low speed of 62 rpm for two minutes, sodium hydroxide was added and the speed was increased to 120 rpm for the period of four minutes. Sand was introduced into the bowl gradually, and mixing continued for another four minutes. The mixture proportion is shown in Table 1, and the mixing process is depicted in Figure 2.

(1) Initial and Final Setting Times of Geopolymer Paste. Manually operated Vicat apparatus according to [35] was used for the determination of the time of setting of the geopolymer binder. The paste with normal consistency from the mixer was formed into balls and placed inside the conical ring of Vicat apparatus and allowed to rest inside the moist cabinet without disturbance as shown in Figure 3. One milliliter needle is used to measure the initial setting time at every 15 minutes intervals. The initial setting time was calculated as the time difference between the time fly ash and the alkaline activator came together and when a penetration of 25 mm or less was observed. The final time of setting of the geopolymer binder was taken when the needle failed to sink into the paste or make complete circular impression on it.

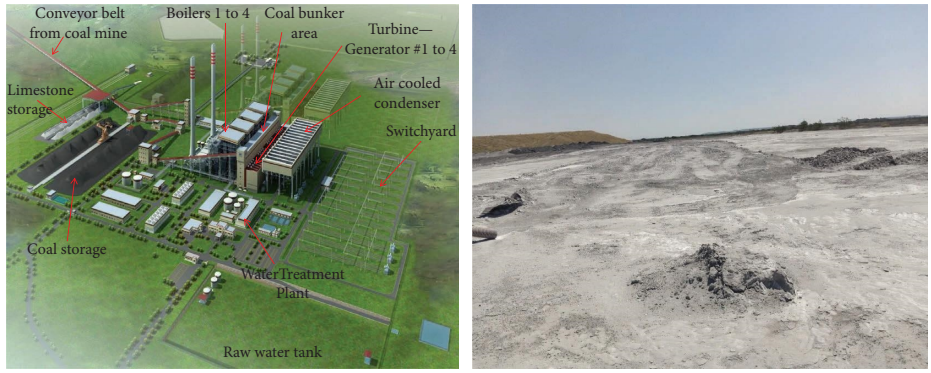


Figure 1: Morupule power Plant B bird's eye view and ash lagoon.

Table 1: Mixture compositions of fly ash paste and mortar.

Mix no.	Fly ash (g)	NaOH (g)	NaOH conc.	Sand (g)
S1	1000	750	8	666
S2			10	
S3			12	
S4			14	
S5	1000	620	8	666
S6			10	
S7			12	
S8			14	
S9	1000	550	8	666
S10			10	
S11			12	
S12			14	

Figure 2: Mixing process of fly ash paste and mortar.

This is taken to be the time difference between the first contact of fly ash and activator and when there is no complete mark.

The Vicat time of setting is calculated as

$$\left[\left(\frac{(H-G)}{(E-F)}\right) * (E-25) + G\right], \quad (2)$$

where G is the time in minutes of last penetration greater than 25 mm; H is the time in minutes of first penetration less than 25 mm; E is the penetration reading at time G; F is the penetration reading at time H.

Figure 3: Determination of setting times of the geopolymer binder.

(2) Flow-Ability of the Geopolymer Mortar. The flow of the geopolymer was carried out according to [36]. The flow table and mould are in conformity with [37] and are shown in Figure 4. The mould was filled with mortar into about 25 mm high and was tamped 20 times with tamper for even distribution and subsequently filled the mould completely. Each test of the flow was carried out three times, and the readings were taken from four different marks on the flow table. Expressing the flow as a percentage of the original base diameter of the mould, we have

$$\text{Flow} = \frac{F-E}{E} * 100, \quad (3)$$

where F is the average reading of the flow in mm and E is the inner base diameter.

(3) Compressive Strength of Geopolymer Mortar. After the flow test had been carried out, the samples were taken to the vibrating machine and mechanically vibrated for the purpose of removing the air voids. The test specimens used are 40 mm × 40 mm × 160 mm prisms according to [38] and are shown in Figure 5. Thereafter, the samples were covered up with polythene to prevent evaporation, and three hours rest period was ensured before samples being taken to oven for thermal curing at 70°C for a period of 24 hours. This was followed with demoulding after 24 hours, and the samples were neatly cut into 40 mm × 40 mm cube. The compressive

Figure 4: Determination of flow of geopolymer mortar.

Figure 5: Determination of compressive strength of geopolymer samples.

strength of the geopolymer binder was determined with a universal testing machine of 2000 kN capacity at the age of 3, 7, 21, and 28 days. The load rate of 2400 N/s was used throughout the test period and the strength was calculated as

$$\text{Compressive Strength} = \frac{\text{Maximum load at fracture } (N)}{\text{Area} = 1600\,\text{mm}^2}. \quad (4)$$

3. Results and Discussion

3.1. Physio-Chemical and Microstructural Characteristics of the Fly Ash

3.1.1. Physical Characteristics of the Fly Ash. The physical characteristics of the Botswana Power Corporation, BPC Plant B fly ash are given in Table 2 with the specific gravity of 2.52 g/cm^3 which is slightly lower than 3.15 for that of Portland cement. Also, the specific surface area was 0.74 m^2/g. The specific surface area gives an indication about the fineness of the fly ash. The mean particle size was 21.24 μm. When the processed fly ash was compared with the as-received from the plant (33.35 μm), the mean particle was greatly reduced by 36% and the specific surface area was increased by 25%. The particle size distribution is given in Figure 6. These parameters play a significant role in the reaction and strength development of the geopolymer paste and mortar. The colour of the ash is blackish grey with fine texture when felt with hands.

3.1.2. Scanning Electron Microscopy (SEM) of the Fly Ash. The SEM micrograph shown in Figure 7 revealed that the fly ash is partially spherical in shape with a lot of flaky and elongated particles. This phenomenon can be attributed to the fact that cenospheres are not fully formed which can be attributed to the lower boiler temperature of (800–900°c), and again, addition of limestone might have resulted into disintegration of the shapes. In other fly ashes of the coal class, full spheres are always the case. There is a noticed coating on the ash, and this could be said to be anhydrite and other minerals as given by XRD analysis.

3.1.3. Chemical and Microstructural Analysis of the Fly Ash. The chemical oxides in the fly ash are tabulated in Table 3, and according to [39], the ash can be classified as class C due

Table 2: Physical characteristics of the Morupule fly ash.

Specific gravity (g/cm^3)	D_{10} particle size (μm)	Median particle size (μm)	D_{90} particle size (μm)	Specific surface area (m^2/g)	Colour
2.52	3.81	21.24	51.43	0.74	Blackish grey

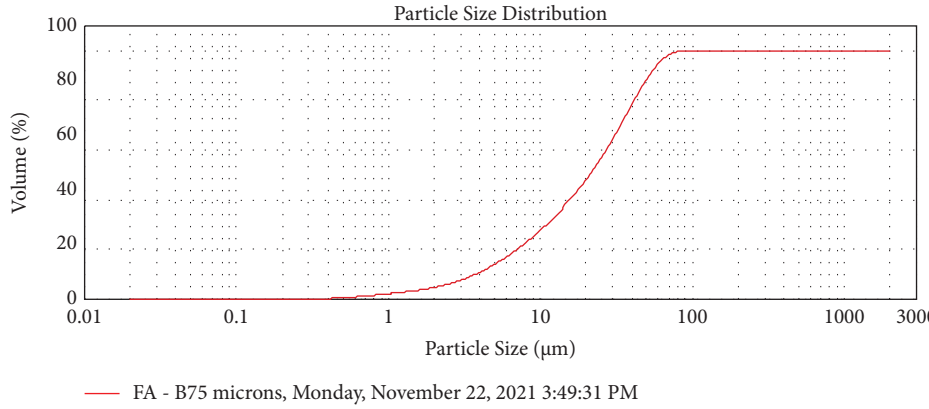


Figure 6: Particle size distribution of fly ash.

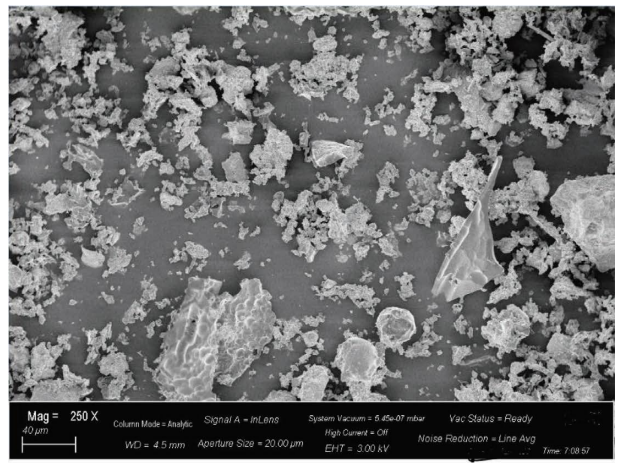


Figure 7: SEM micrograph of the fly ash.

Table 3: Elemental composition of the Morupule fly ash.

Fly ash	Al_2O_3	CaO	Fe_2O_3	K_2O	MnO	Na_2O	SiO_2	SO_3	P_2O_5	TiO_2	LOI
Percentage	28.01	11.36	10.44	0.61	0.12	0.38	37.24	7.44	0.20	2.79	3.58

to the fact that addition of alumina, silica, and iron oxides is 76%, the calcium oxide is 11.36%, and LOI is less than the specified maximum value by the code. The four oxides in high content are in the order of decrease as SiO_2, Al_2O_3, CaO, and Fe_2O_3 which are essential oxides needed for early and late strength development in the cement chemistry. The addition of the two alkalis, K_2O and Na_2O, is less than one percent of the whole oxides.

Again, as a result of sieving through the 75 μm, the coarser part of the fly ash got excluded and this greatly improved the percentage of anhydrite, hematite, quartz low, and aluminum silicon oxide. The peak of the XRD graph occurred between the 26 and 28 position as shown in Figure 8.

3.2. Effect of Alkaline Activator to Fly Ash Ratio on the Compressive Strength of Geopolymer Mortar. Sodium hydroxide was the only alkaline activator used in this study. No sodium silicate, no superplasticizer, and no extra water were added during the mix. This was done in order to know the potential capability of NaOH in leaching oxides in the fly ash that possess the same characteristics in terms of XRF, SEM, and XRD. In Figures 9(a)–9(c), it could be seen that the ratio of alkaline activator to fly ash is crucial to the strength development, and regardless of the concentration of the sodium hydroxide, there was a steady strength development across the age; however, the optimum strength of the geopolymer mortar is greatly influenced by the ratio of alkaline activator to fly ash ratio.

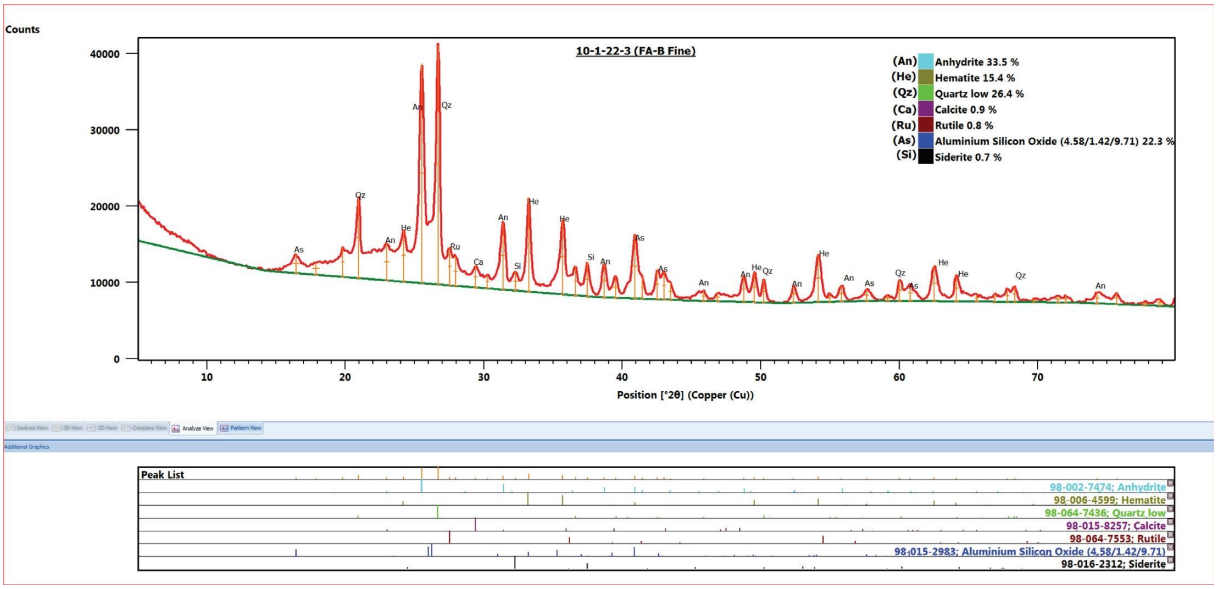
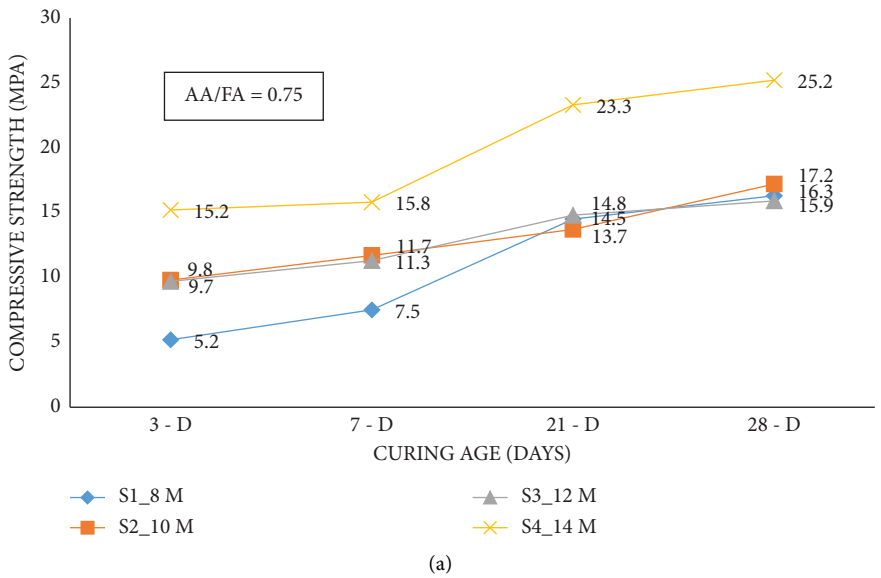
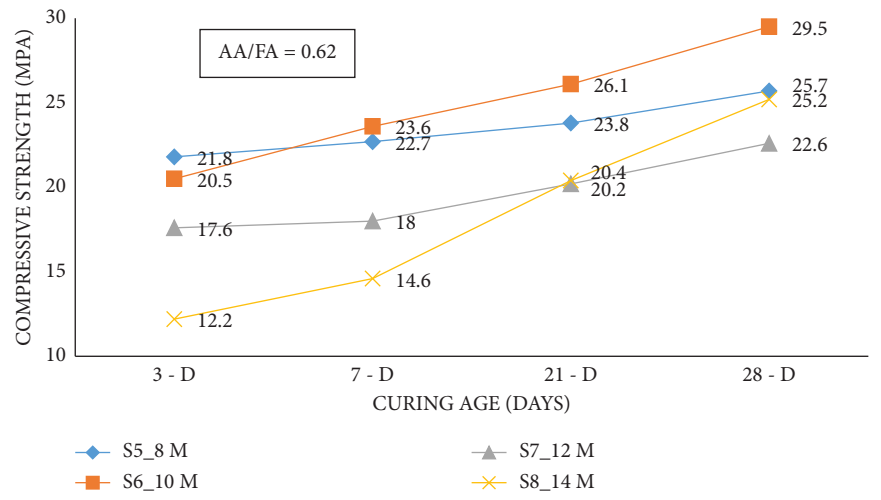


Figure 8: Fly ash Plant B XRD diffractograph.

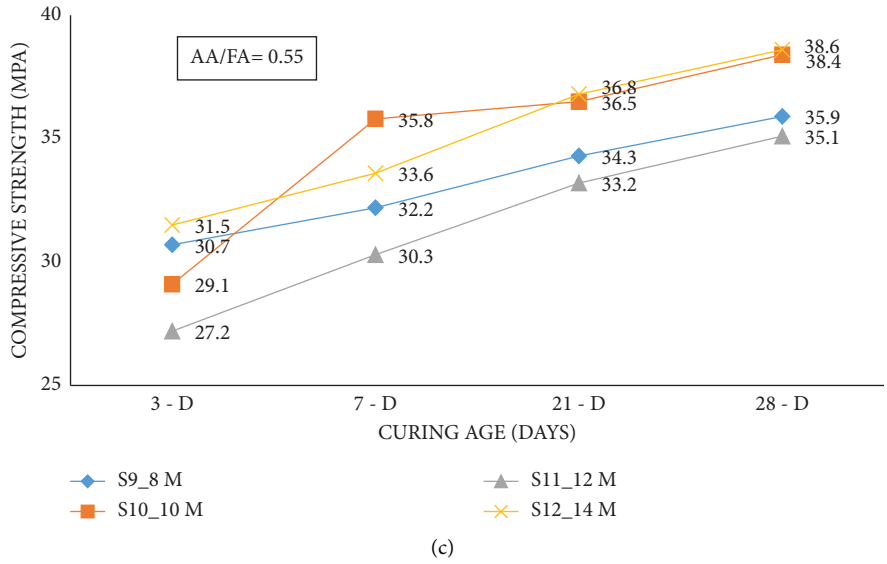


(a)



(b)

Figure 9: Continued.



(c)

Figure 9: Effect of alkaline activator to fly ash ratio on the compressive strength of geopolymer mortar.

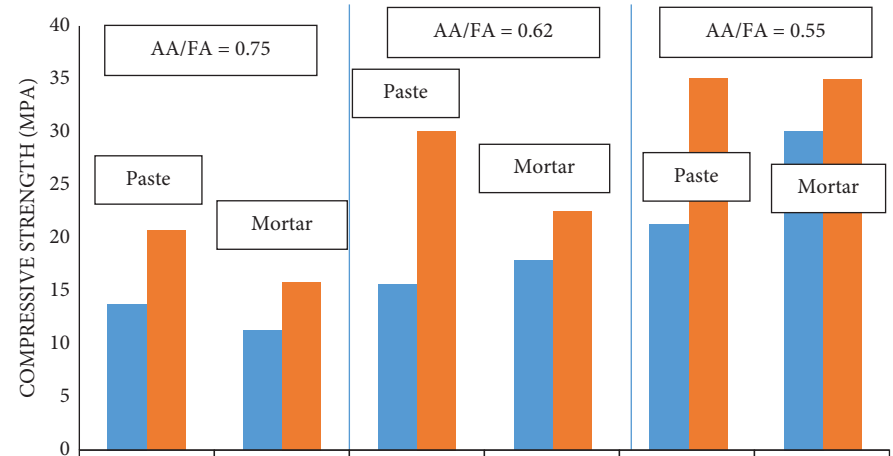
In this study, the fly ash characteristics and other variables were kept constant; however, the increase in the alkaline activator/fly ash ratio from 0.55 to 0.75 led to the reduction in the strength across the age and across the concentration. Also, the lower ratio of AL/FA below 0.55 resulted in poor result. This occurrence has been attributed to insufficient activator necessary for the dissolution of the fly ash particles which subsequently resulted into higher proportions of unreacted fly ash [40]. It could be said that for optimum compressive strength to take place, the alkaline activator to fly ash ratio should be kept at 0.55 using 10 M as a baseline. The decrease in strength has been elucidated to the excessive activator in the mix which prevented geopolymerization to occur [41, 42].

3.3. Effect of Sodium Hydroxide Concentration on the Compressive Strength of Geopolymer Mortar. The role of concentration of sodium hydroxide in strength development is reported in Figures 9(a)–9(c) for the three AL/FA ratios considered. The concentration level was increased from molarity of 8 to 14. For AL/FA ratio of 0.55, there was a mild increase in strength across the curing ages as the concentration was increased. The percentage increase in strength between the three days and twenty-eight days strength are approximately 15%, 24%, 23%, and 18%, respectively, for 8, 10, 12, and 14 moles. With the increase of AL/FA from 0.55 to 0.62, the percentage increase is as follows: 15%, 31%, 22%, and 52%, respectively, for 8, 10, 12, and 14 moles. Further increase in AL/FA to 0.75 experienced more increase in strength development to the tune of percentage increase of 68%, 43%, 40%, and 40%, respectively, for 8, 10, 12, and 14 moles.

With the increase in the alkaline activator to fly ash ratio, the compressive strength of the AL/FA is still much higher. Concentration has little significance on the compressive strength of AL/FA of 0.55, and higher AL/FA ratio requires activator of high concentration to leach out good quantity of the oxides for geopolymerization to take place. Minor increase in strength was reported by when concentration was increased from 12 to 16 moles, and [43] reported that lower molarity of sodium hydroxide gave poor compressive strength, but a remarkable compressive strength was recorded at 8.01 M and above. In contrast, when the molarity was above 11.01 M, a reduction in strength occurred and the reason given was that viscosity of the mix was higher and this created compaction challenge.

3.4. Effect of Curing Age on Strength Development. All the samples are cured thermally for a period of 24 hours at a temperature of 70°C. The samples were later withdrawn and kept at ambient temperature until the crushing days. Again, from Figures 9(a)–9(c), there is an increase in strength across the curing age irrespective of the ratio of AL/FA and concentration of the activator. For all the mixes, the quantity of sand was kept constant, but the ratio of alkaline activator to fly ash was varied as 0.75, 0.62, and 0.55, respectively. There was a progressive increase in the compressive strengths across the curing age; however, there is not much significant appreciation between the 7-day strength and 28-day due to the fact that most samples had achieved over 70% of 28- day strength at the age of seven; this justifies geopolymer mortar to be an early strength construction material, and this might have occurred due to leaching of silica oxide from the fly ash, polymerization, and heat curing.

At alkaline activator to fly ash ratio, AA/FA of 0.75 in Figure 9(a), the maximum compressive strength was 25.2 MPa which occurred when sodium hydroxide concentration was 14 M. With AA/FA of 0.62 in Figure 9(b), the maximum compressive strength was 29.5 MPa and this happened when sodium hydroxide was 10 M, while the maximum compressive strength was 38.6 MPa at 14 M NaOH when AA/FA was 0.55, as shown in Figure 9(c).



	P1_12 M	S3_12 M	P2_12 M	S7_12 M	P3_12 M	S11_12 M
7 D	13.8	11.3	15.6	18	21.4	30.3
28 D	20.7	15.9	30.2	22.6	35.2	35.1

Figure 10: Comparison of compressive strength of geopolymer mortar and paste.

Further test on the compressive strength of geopolymer mortar was compared with geopolymer paste for a representative sample from each classified AA/FA ratio of 0.75, 0.62, and 0.55 at constant 12 M in Figure 10; the values of compressive strength geopolymer mortar were lower at ratio 0.75 and 0.62 compared to geopolymer paste at 7 and 28 days; however, there was a drastic increase in the strength from 21.4 (paste) to 35.2 MPa (mortar) and from 30.3 (paste) to 35.1 MPa (mortar) when the AA/FA was fixed at 0.55. This confirms that the larger proportion of alkaline in the mix weakened the strength development. The same occurrence was earlier reported by [25], and the reason attributed to this reduction by [18] was that strength of mortar largely depends on the geopolymer gel and bonding between the paste and sand.

The failure mode of the crushed samples is shown in Figure 11, and this revealed that the geopolymer mortar and the binder underwent true failure and were satisfactory which was in line with [38]

3.5. Flow-Ability of the Geopolymer Mortar. In Figures 12(a)–12(d), as the ratio of AA/FA increases from 0.55 to 0.75, there was an increase in the flow measurements with a slight reduction as the concentration of NaOH increases. It was observed that as the concentration of NaOH increases, the mixture becomes thicker; however, no additional water was added in order to know the effectiveness of NaOH in leaching the silica oxide. The flow measurement was classified into three as high, medium, and moderate for the three AA/FA classifications. The range of the measurements was between 105 and 143% as shown in Figure 12.

3.6. Initial and Final Setting Times of Geopolymer Paste. The result of initial and final setting times carried out on geopolymer paste yielded values of 280–350 minutes for initial and 950–1170 minutes for final times, respectively. It

Figure 11: Failure mode of geopolymer samples.

was noticed that increase in the molarity of NaOH reduces both initial and final setting times. Also, mixes with higher AA/FA of 0.75 had longer initial and final setting times compared to AA/FA of 0.62 and 0.55. For the actual construction, setting times of cement play a significant role and possibility of reducing longer final setting times through addition of high calcium oxide material such as slag that can assist in quick hardening of the geopolymer paste.

3.7. Microstructural Analysis of the Geopolymer Mortar and Paste. Remains of crushed samples were used for carrying out both Scanning Electronic Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) after 28 days of the test. A cohesive geopolymer matrix was noticed in both Figures 13 and 14, respectively. The higher strength in mortar mix S10 (Figure 13) is may be a result of the dense structure and a strong interlocking interface between the fly ash and aggregates which was coated by sodium hydroxide. The original fly ash was a mixture of spherical and flaked shaped particles, some of which was noticed as unreacted in Figure 14, and this could be the reason for reduced compressive strength. A closer look into Figures 13(b) and 14(b) shows that there is some presence of pores and voids despite the surfaces of the fly ash being well covered with the activator, and this ultimately reduces the maximum strength.

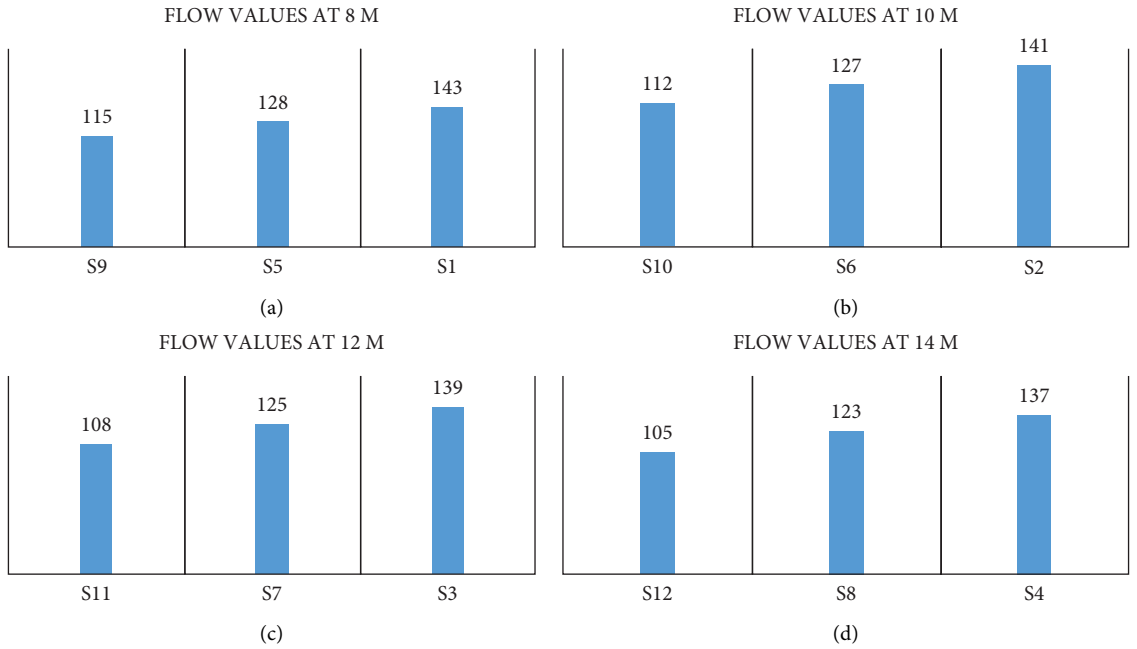
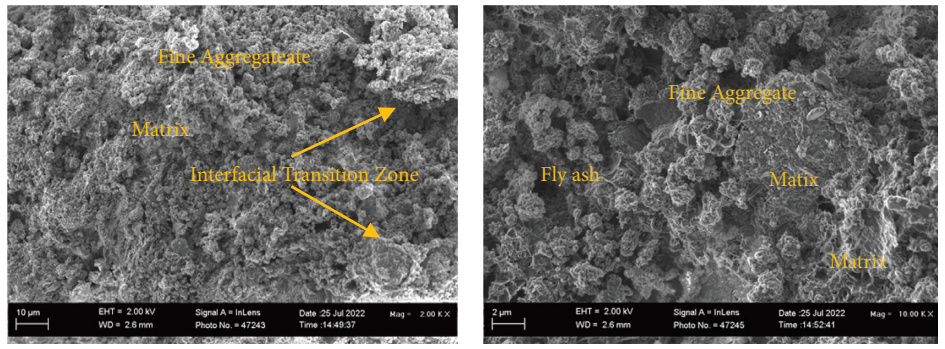


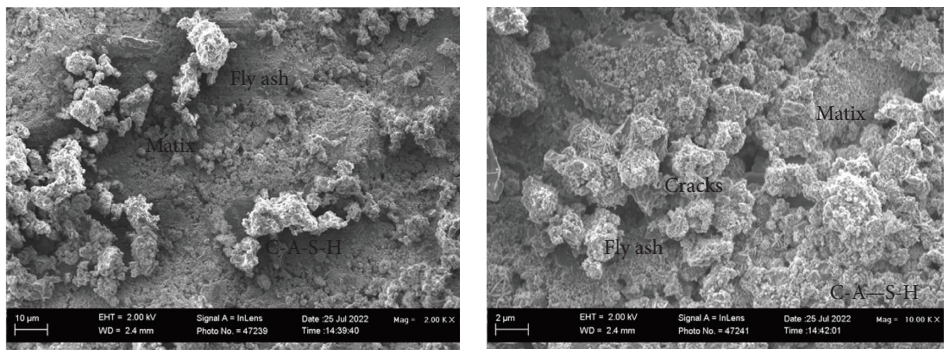
Figure 12: Flow values of the geopolymer mortar.



(a) (b)

Figure 13: SEM images of mortar mix S10.

Figures 15 and 16 reveal EDS mortar samples S10 and S6, and it could be inferred that leaching, dissolution, reaction, polymerization, and condensation had taken place due to the presence of reaction products that were spread over the surfaces of the fly ash which was absent in the original fly ash (Figure 7). The microstructural analysis gives the atomic weights of elements present in the matrix with Si, Al taking the lead and coming directly from the fly ash, and there is a presence of Ca in the qualitative analysis. The mix S10 with higher Si/Al of 1.31 and Na/Al of 0.55 had higher compressive strength and lesser pores in the images compared to mix S6 with Si/Al of 1.14 and Na/Al of 0.63. In addition, the reaction products of the geopolymer mortar in Figures 15 and 16 show the formation of sodium aluminate silicate hydrate (N-A-S-H) gel, and this had earlier been reported by [20]. In the presence of calcium in the analysis, there is a tendency for the formation of C-A-S-H gel. There was not much departure in the ratio of Si/Al and Na/Al percentages, and this also explains the reason for closeness in the compressive strength of geopolymer mortar in this study.



(a) (b)

Figure 14: SEM images of mortar mix S6.

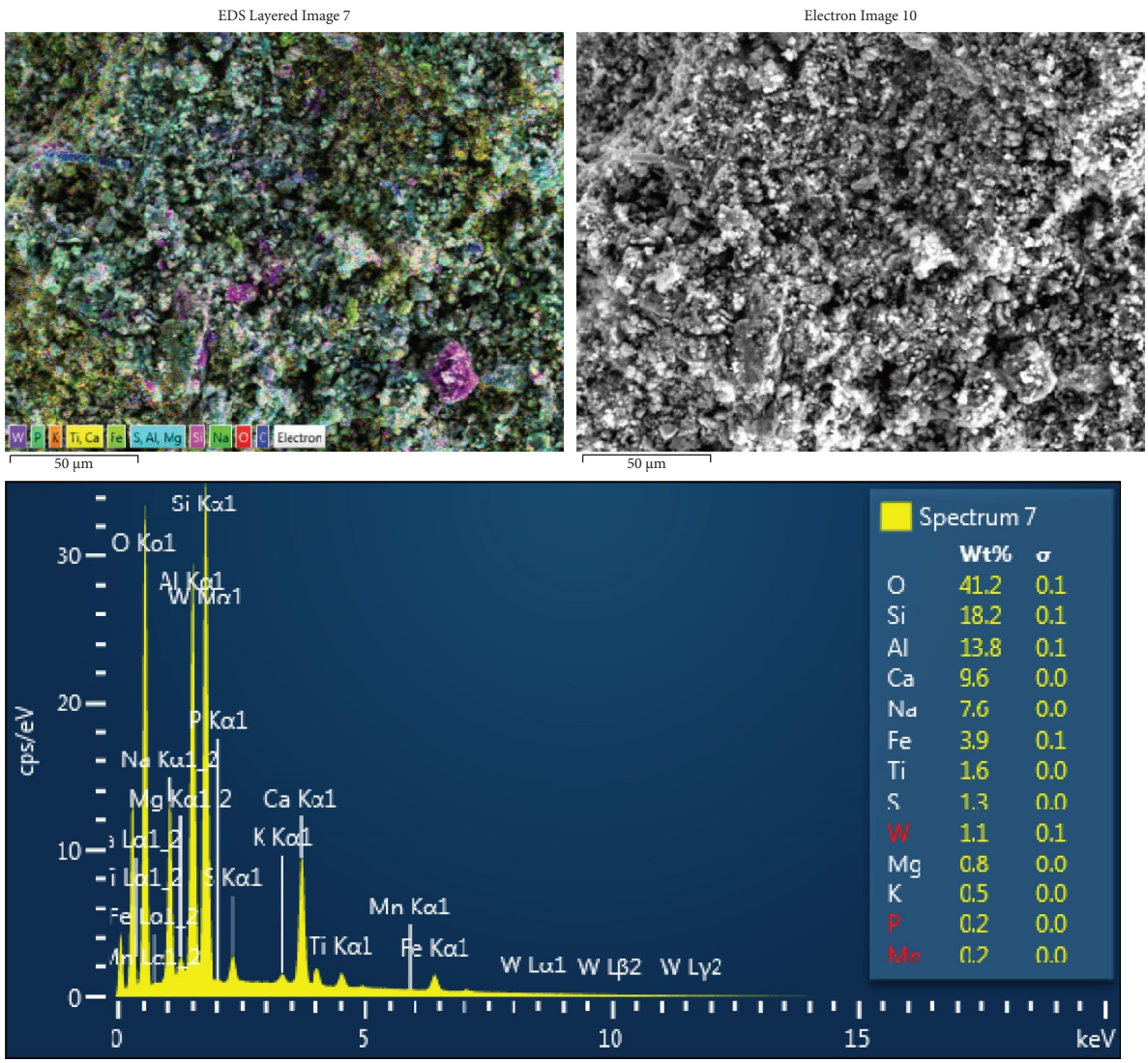


Figure 15: EDS image of mortar mix S10.

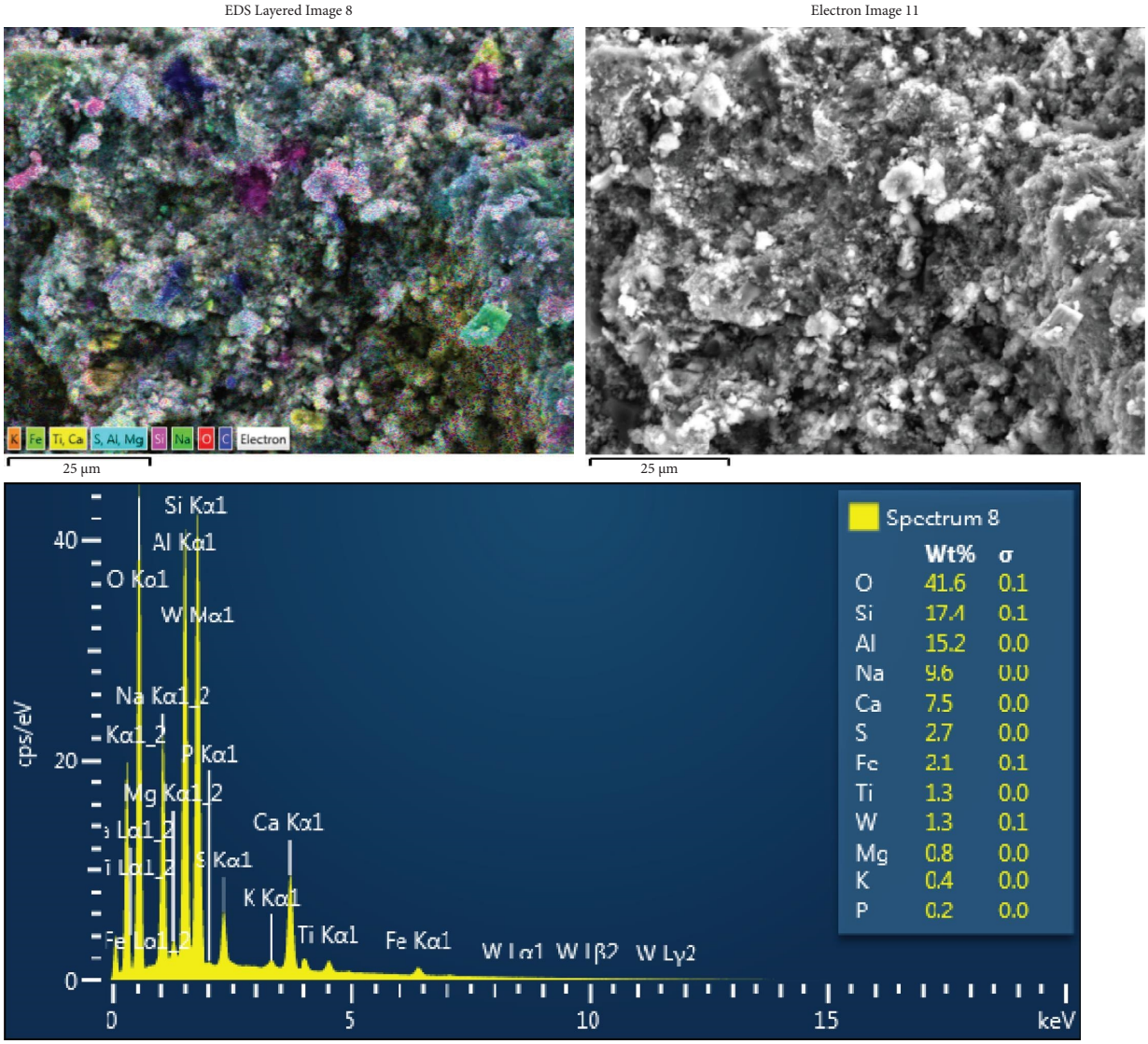


Figure 16: Energy dispersive spectroscopy image of mortar mix S6.

4. Conclusion

A laboratory investigation on dissolution of silica and alumina oxides from Morupule fly ash has been examined. The as-received fly ash was milled and sieved with 75 micrometers sieve, and physical, chemical, and microstructural analysis was carried out. For all the samples, the mixing time was the same and heat curing of 70°C was used. The following conclusions were made from this study:

(1) The fly ash possessed adequate and sufficient oxides with suitable mineral compositions necessary for geopolmerization to take place. With the algebraic sum of silica, aluminum, and iron oxides of over 50%, the ash is classified as Class C pozzolana according to the relevant standard.

(2) The compressive strength of the geopolymer binder depends on the alkaline activator/fly ash ratio and concentration of sodium hydroxide. With the sodium hydroxide to fly ash ratio of 0.55, 0.62, and 0.75, the compressive strengths were found to be from 14.90 to 38.60 MPa at 28 days. For the alkaline activator/fly ash ratio of 0.75, an increase in concentration of NaOH played a significant role in the strength development, while a mild strength increase from 3 days to 28 days was noticed when concentration was increased from 8 M to 14 M for sodium hydroxide to fly ash ratio of 0.62 and 0.55, respectively.

(3) The workability of the geopolymer mortar determined gives the range of 105–143% and initial setting times of 280–350 minutes and final setting times of 950–1170 minutes.

(4) The values of compressive strength indicated that the binder can be used for normal constructions and precast operations.

The results of the microstructural analysis also corroborated that the reaction had taken place, and the product is a viable construction material. This study has provided the first hand information that the waste coming from the Botswana Power Corporation, BPC can be turned into resource recovery.

Data Availability

No data were used to support the findings of this study.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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