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Optimization of geopolymer synthesis using coal fly ash for enhancement in properties as sustainable and durable construction material

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ABSTRACT

Coal-based power plants produce a huge quantity of fly ash, which requires a huge amount of revenue. The same practice is conducted at the SEPCO coal power plant in Karachi; this study was conducted to address the disposal issue productively and to determine the potential of coal fly ash for the development of sustainable, efficient construction materials. In this study, work was conducted on the effect of alkali activator concentration on fly ash-based geopolymer, examining the morphological properties of the geopolymer material at the micro level using X-ray diffraction, scanning electron microscopy, Fourier-transform infrared spectroscopy, and Brunauer-Emmett-Teller analysis to develop a durable and environmentally friendly material for the construction industry. The obtained material formed was a porous, amorphous-structured material with regular micro- and macro-pores, resulting in reduced density and potentially enhanced properties. EDX analysis revealed lower incorporation of sodium (Na), silicon (Si), and aluminum (Al), indicating a limited dissolution of fly ash and poor formation of the geopolymeric gel as well as higher levels of unreacted oxides, such as calcium (Ca), iron (Fe), and residual quartz reflects an underdeveloped geopolymer matrix due to lower concentration of alkaline activator solution. In contrast, the 8 M geopolymer shows significantly higher concentrations of Na, Si, and Al, which are key elements in the formation of N-A-S-H gel networks. The improved Na/Al and Si/Al ratios in the 8 M sample point to more complete dissolution of the raw material and better integration into the geopolymer framework. FTIR analysis indicated the chemical environment of GP, with broad peaks in the 3336 cm^{-1} range suggestive of hydroxyl groups as well as new peaks for carbonate and bicarbonate groups. These changes indicate improved surface properties and reactivity, making GP more suitable for various applications. BET isotherm analysis confirmed the presence of mesoporous material in GP, with an average pore size of 60 µm, porosity of 30.8%, and a BET surface area of 25 m^2/g, respectively. This research underscores the potential of CFA-derived geopolymers as a sustainable and high-performance alternative for construction materials.

Keywords: geopolymer, construction material, fly ash, bricks, alkaline solution.

INTRODUCTION

The construction sector has traditionally used Portland cement as a major binder in concrete and a key factor in constructing infrastructure. Global cement production has extensive negative environmental consequences, as it contributes to around 8% of total CO_2 emissions. This has led to alternative materials being explored with a focus on sustainability. The concerns about carbon footprint within the construction industry have led to the research and development of geopolymers as

a novel and sustainable alternative to traditional cement. Geopolymers are formed by thermally activating an alkaline solution, such as NaOH and KOH, with alumino-silicate materials. Emerging from this activation, a strong three-dimensional polymer network is formed, which is stronger, more durable, and more fire-resistant than conventional cement. The production of traditional clay-fired bricks and concrete blocks often requires significant energy and is associated with serious environmental issues, including resource depletion and high CO_2 emissions. Due to the presence of rich silica and alumina in fly ash, it serves as an alternate precursor for various applications including geopolymer synthesis as an environmentally friendly and durable material. In contrast, geopolymer bricks and blocks, which are made from industrial waste materials like coal fly ash, offer a chance to lessen the environmental impact of the building sector while providing strong, affordable, and high-performing substitutes for traditional materials. Geopolymer blocks and bricks have dual advantages, such as waste handling, less energy production, and less CO_2 production compared to conventionally fired clay bricks. Geopolymer binder exhibits exceptional mechanical properties, high fire resistance, and resistance to chemical attack, making it an ideal material for construction applications. This study focused on creating a sustainable and eco-friendly alternative to traditional cementitious bricks by developing a fly ash-based geopolymer. The geopolymer properties were enhanced by increasing the concentration of the alkaline activator used in the synthesis. The material was thoroughly characterized using SEM, EDX, FTIR, and BET analyses to evaluate its morphology, chemical composition, functional groups, and pore structure. This geopolymer shows potential as either a direct alternative or to improve the physical and mechanical properties of conventional bricks.

MATERIALS AND METHODS

Preparation of geopolymer from different coal fly ash types

Fly ash was collected locally from a coal-based power plant. First, a mixture of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solutions was prepared with a 1:1 ratio to obtain the alkali-activating solution, 10 mL of 4 M and 8 M NaOH solutions were mixed with 10 mL of sodium silicate solution. Then, the fly ash was gradually combined with the alkali solution while continuously stirring to form a homogeneous slurry. The mixture was then gently stirred for 30 minutes to ensure even distribution of the foaming agent, resulting in the formation of a foamed geopolymer paste. Subsequently, the foamed mixture was poured into molds and allowed to set. In the last material, the material was heated at a temperature of 80 °C for 4 h in a muffle furnace to form a fly ash source geopolymer (Kalombe et al., 2020) (Figure 1).

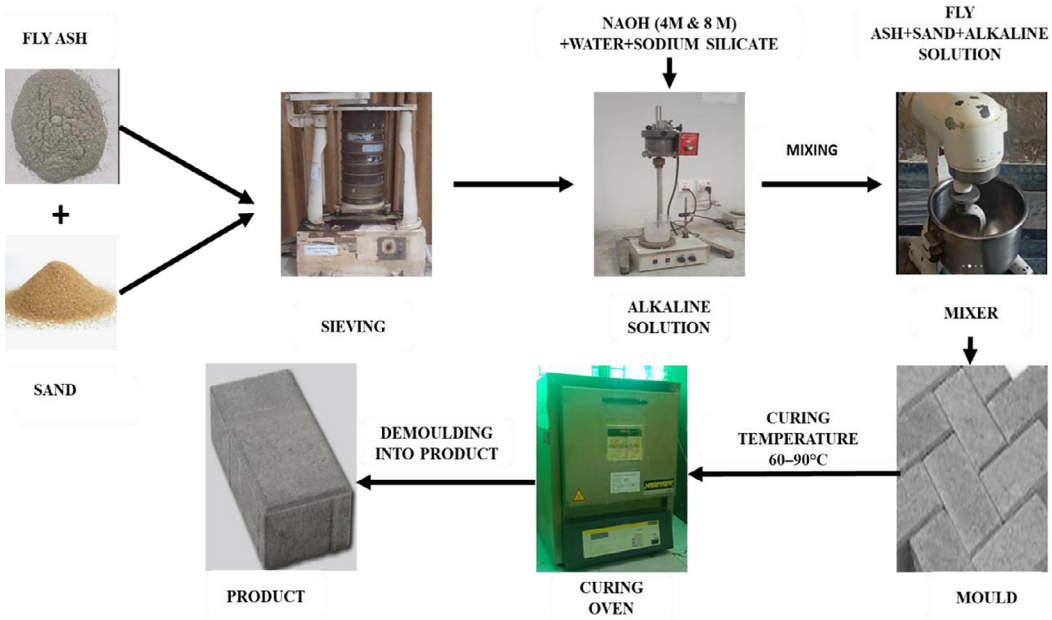

Figure 1. Synthesis of geopolymer

Morphological and chemical characterization

The morphological and chemical behavior of geopolymer samples were accurately analyzed by using advanced methods. Scanning electron microscopy (SEM) and energy dispersive x-ray (EDX) analysis were performed using the JEOL Japan JSM-6490 LV facility, which provides high-resolution images of the sample structure and elemental content. Fourier transform infrared (FTIR) spectra were obtained using a Shimadzu Prestige 21. Surface characteristics were also examined with nitrogen adsorption-desorption isotherms, which were calculated from the Quanta Chrome Novawin data. The apparent average pore diameter was also calculated by carefully examining SEM images and quantifying them using the Web Plot Digitizer software, where accurate measurements were taken. Specific surface area and porosity of the samples were also calculated using the BET (Brunauer-Emmett-Teller) equations, providing significant information regarding the surface characteristics of the material.

Testing of geopolymer bricks

The geopolymer and sand mixture in a 1:2 ratio was poured into the mold and compressed to obtain a compact sample. The size of the bricks was measured based on the British standard, BS 3921:1985, which states that 1 unit of brick should have at least 215 mm long, 102.5 mm wide, and 65 mm high. After the compression, the samples were demolded and placed in an oven for curing at 60 °C for 24 hours before testing.

Compressive strength test

The compressive strength test for brick was carried out according to ASTM C67-1116. The compressive strength was determined after 24 hours. The reported compressive strength values were an average of the results obtained for the five samples produced for each ratio.

Table 1. Physicochemical characterization of coal fly ash

No.	Component	Chemical composition (Wt%)
1	SiO_2	44.52
2	Al_2O_3	22.48
3	Fe_2O_3	11.58
4	CaO	9.84
5	MgO	3.92
6	C	2.54
7	K_2O	1.73

Water absorption test

Water absorption is an important property that influences the durability of bricks. Water absorption test was conducted according to ASTM C140-07a after 24 h immersed in water using 3 specimens for each ratio.

RESULTS AND DISCUSSION

Physicochemical properties of coal fly ash

Fly ash resulting from coal combustion in power generation industries was studied for chemical composition and physicochemical behavior to analyze its suitability for the production of geopolymer. The fly ash was analyzed chemically to determine the composition, where silicon dioxide (SiO_2) was the predominant constituent at 44.52%, followed by aluminum oxide (Al_2O_3) at 22.48% and iron oxide (Fe_2O_3) at 11.58%. Some other important constituents were calcium oxide (CaO), 9.84%; magnesium oxide (MgO), 3.92%; carbon (C), 2.54%; potassium oxide (K_2O), 1.73%; and sulfur trioxide (SO_3), 1.35%, as evident from Table 1. Other elements made up the rest of 2.05%. The table above shows that the selected fly ash contains an appreciable quantity of silica and alumina required for the synthesis of geopolymer.

Effect on the morphology of the geopolymer

SEM analysis indicates, as shown in Figure2, considerable morphological differences in geopolymers produced with 4 M and 8 M NaOH activator solutions. The 4 M sample has a porous, irregularly shaped morphology with identifiable unreacted fly ash particles distributed throughout the matrix. Porosity and unreacted particles are signs of an underreacted geopolymerization reaction, resulting in a stronger and less cohesive product. In contrast, the 8 M geopolymer has a denser and more compact microstructure. The surface is smoother and more homogenous with fewer pores and near-complete coverage of fly ash particles by geopolymer gel. The denser morphology suggests that the higher content of hydroxide ions in the 8 M solution can facilitate the dissolution of

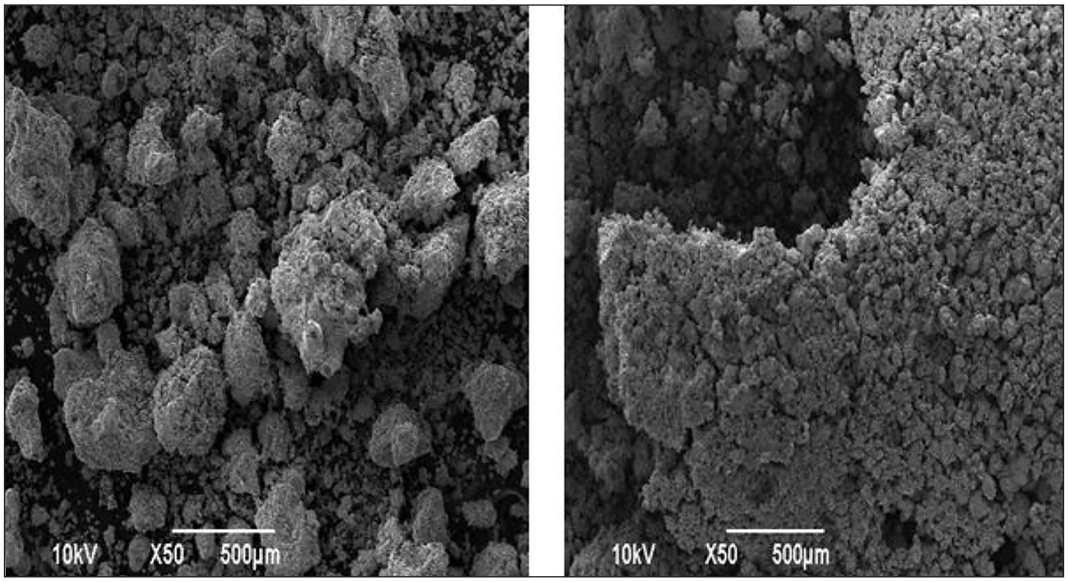

Figure 2. EDX analysis of geopolymer using different concentrations of alkaline activator (a) 4 M (b) 8 M

aluminosilicates effectively and therefore forms a stronger network of gel and superior mechanical properties. There are fewer pores with uneven and irregular shapes. The structure seems more crystalline than amorphous{Aziz, 2023 #45}{Aziz, 2023 #46} (Aziz et al., 2023). EDX analysis provides elemental composition data that helps evaluate the extent of geopolymerization in fly ash-based geopolymer. In the 4 M NaOH-activated sample, EDX typically shows lower incorporation of sodium (Na), silicon (Si), and aluminum (Al), indicating a limited dissolution of fly ash and poor formation of the geopolymeric gel. The presence of relatively higher levels of unreacted oxides, such as calcium (Ca), iron (Fe), and residual quartz reflects an underdeveloped geopolymer matrix. In contrast, the 8 M geopolymer shows significantly higher concentrations of Na, Si, and Al, which are key elements in the formation of N-A-S-H gel networks. The improved Na/Al and Si/Al ratios in the 8 M sample point to more complete dissolution of the raw material and better integration into the geopolymer framework. This enhanced elemental distribution confirms a higher degree of reaction and supports the findings from SEM, XRD, and FTIR, indicating that 8 M NaOH facilitates the development of a chemically homogeneous and structurally stable geopolymer (Figure 3).

FTIR analysis

Figure 4 shows the functional groups present from two different alkali activator concentrations on geopolymer samples. Strong broad bands indicated in FTIR for 4M concentration water molecules are weakly bound or adsorbed on the surface or trapped inside the cavities in geopolymer. A weak peak at 1411 cm^{-1} is attributed to CO bending vibration, indicating the sodium carbonate form of carbonation in sodium hydroxide-rich geopolymer. In addition to this, the peak at 1109 cm^{-1} is due to polycondensation with Si-O and AI-O bonds, and bands at 70 cm^{-1} show the Si-O-Si/Si-O-Al and Al-O/Si-O bending vibrations. FTIR analysis of 8M alkali activator concentration produces a geopolymer revealing significant chemical and structural changes, offering improved surface properties and reactivity.

The broad peaks in the range 3000–3200 cm^{-1} are indicative of hydroxyl groups (–OH), suggesting the presence of free or bonded hydroxyls within the geopolymer matrix. Hydrogen peroxide treatment results in the formation of additional hydroxyl groups, thereby increasing the hydroxyl content and enhancing chemical interactions. The peaks around 900–1100 cm^{-1} correspond to the asymmetric stretching vibrations of silicon-oxygen (Si–O–Si) and aluminum-oxygen-silicon (Al–O–Si) bonds, which are crucial components of the geopolymer framework, indicating the retained structure of the material. Additional peaks appearing around 1410–1500 cm^{-1} and 870 cm^{-1} can be attributed to carbonate and bicarbonate groups formed during the decomposition of hydrogen peroxide. These byproducts slightly alter the chemical environment within the geopolymer matrix, impacting its acidity or alkalinity. The overall chemical stability of the treated geopolymer can

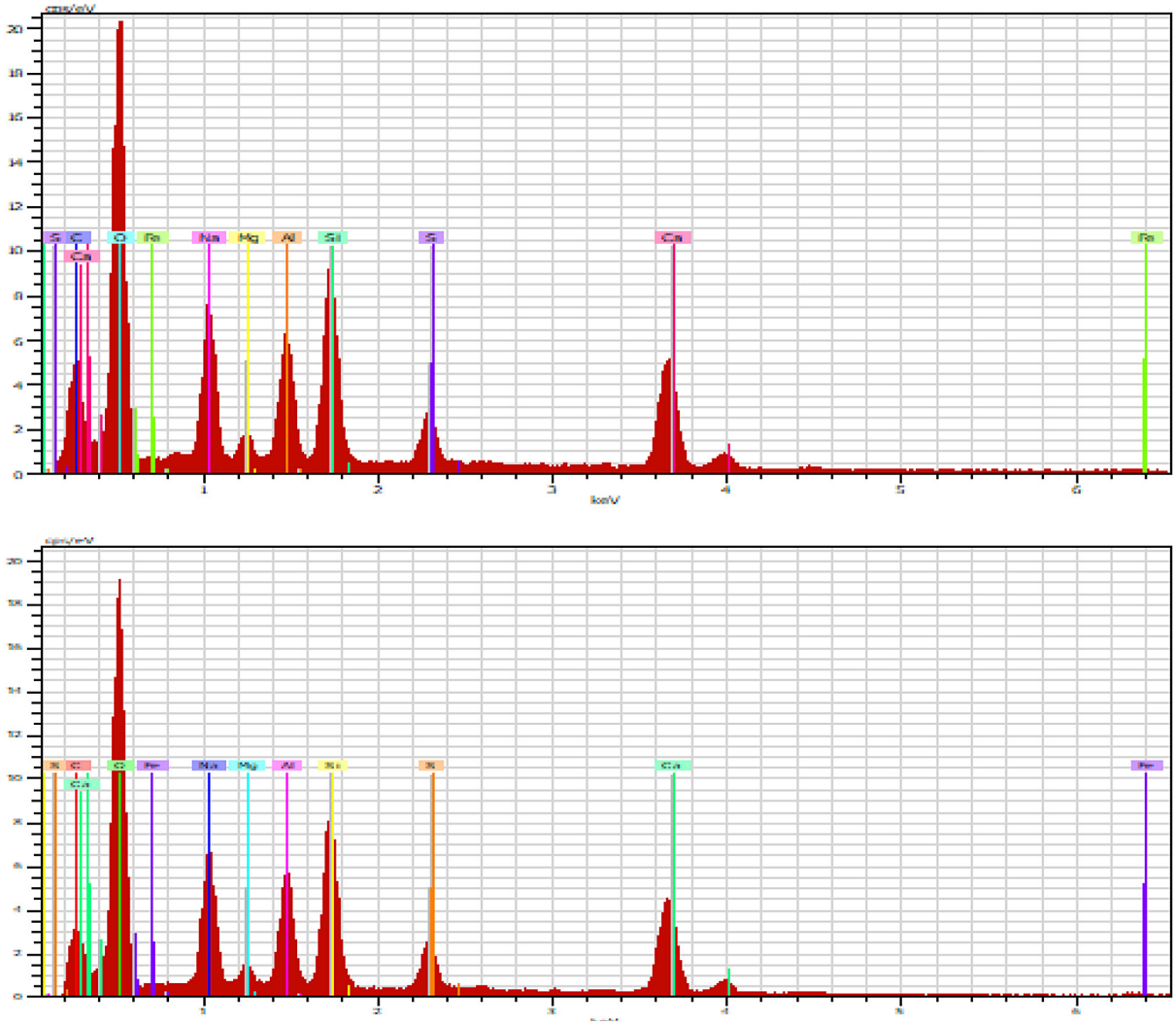

Figure 3. EDX analysis of geopolymer using different concentrations of alkaline activator (a) 4 M (b) 8 M

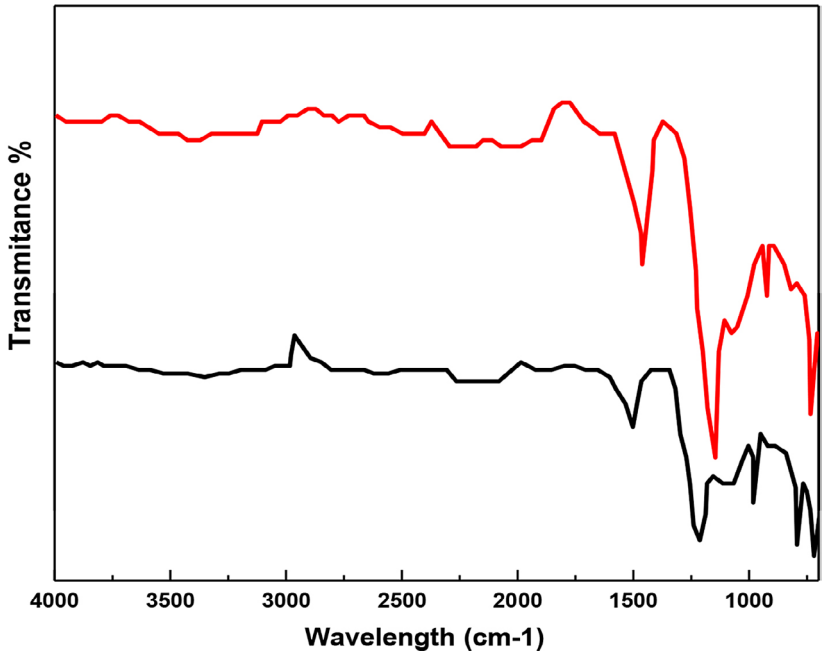

Figure 4. FTIR of the geopolymer formed at various alkali concentrations

be inferred from the broadening or shifting of peaks around key functional groups, reflecting a more dynamic bonding environment. The changes in surface chemistry, as evidenced by variations in FTIR spectra, indicate improved adsorption properties and reaction kinetics. By comparing the FTIR spectra of the geopolymers produced with 4M and 8M alkali activator concentrations, it is possible to gain deeper insights into the chemical and structural modifications induced by increasing alkali activator concentration. Such results in enhanced mechanical strength, improved thermal and electrical conductivity, as well as increased chemical resistance. Geopolymer is suitable for various advanced applications, including lightweight construction materials, thermal insulation components, and chemical-resistant structures (Nikoloutsopoulos et al., 2021).

XRD analysis

The X-Ray Diffraction pattern of the geopolymer is shown in Figure 5. The paste was prepared with 8 molar NaOH at 105 °C. The XRD pattern of the geopolymer paste (Figure 5) indicates that it is predominantly composed of quartz (Q), calcite (Ca), albeit (Al), anorthite (An), clinochlore (Cl), and illite (I). The presence of calcite, or calcium carbonate, in XRD indicates the reactivity of calcium hydroxide with carbon dioxide in air, resulting in the formation of calcium carbonate that is more stable in water-rich environments. However, the addition of calcium will form the C-S-H gel, but due to its amorphous structure, no peak was seen on the XRD pattern. The addition of alkaline solutions during geopolymerization results in a meaningful conversion of crystalline phases into amorphous phases. At the same time, the SEM image also shows evidence of an amorphous microstructure due to an increase in alkali activator concentration.

BET analysis

Figure 6 shows the BET Isotherm of 8 M alkali activator concentration made geopolymer. As it can be seen, the steep increase in the adsorption curve at higher relative pressures (near $P/P_0 \approx 1$) and the distinct hysteresis loop between the adsorption and desorption curves suggest the presence of mesopores and macropores.

The total pore volume is estimated from the quantity of nitrogen gas adsorbed at high relative pressure ($P/P_0 \approx 1$). From the graph, the total amount of gas adsorbed is around 28.70 cm^3/g, indicating substantial porosity introduced by increasing alkali activator concentration. Porosity is determined by the ratio of pore volume to the total volume of the material. According to the given adsorption data, the material exhibits significant internal pore structure (Fan et al., 2021). Moreover, the analysis of the BET data revealed that GP-HP had an average pore size of 70 µm, porosity of 34.8% and BET pore surface area of 30 m^2/g, respectively.

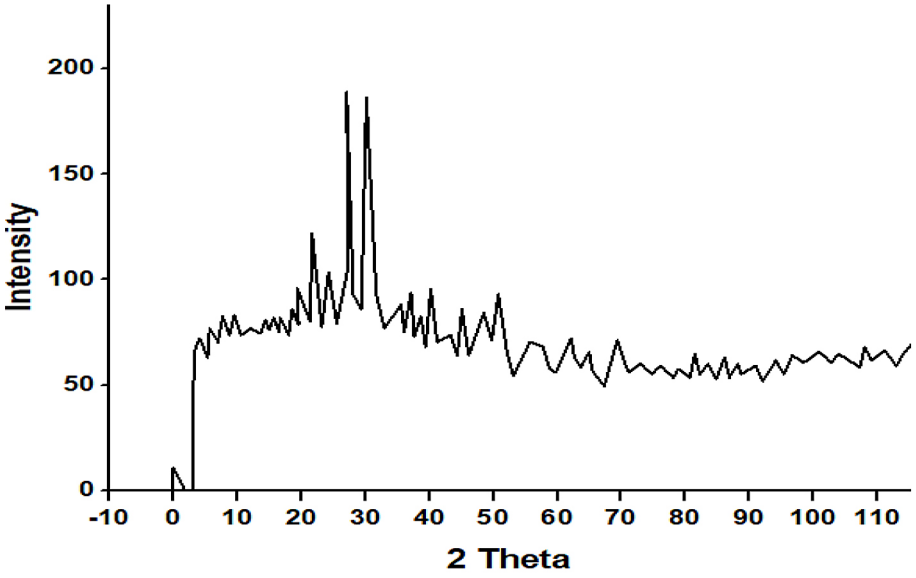

Figure 5. XRD patterns of the geopolymer

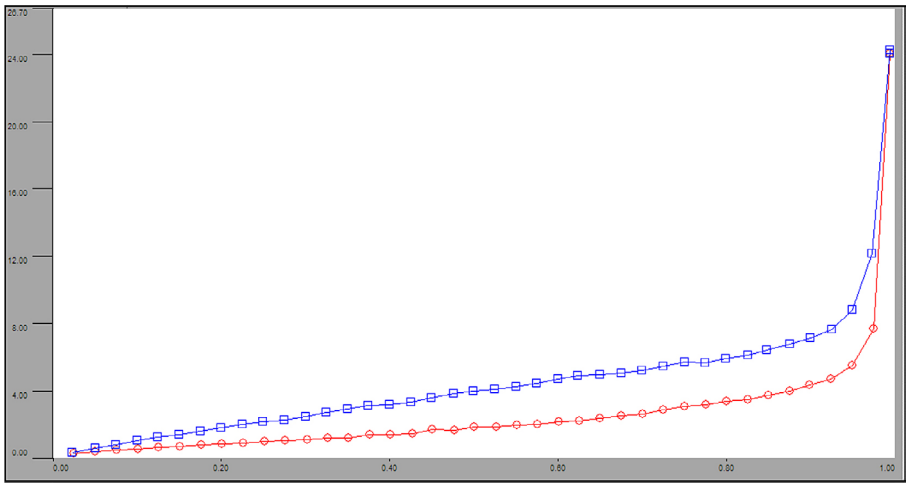

Figure 6. BET Isotherm of the geopolymer

Brick production and testing of properties

Fly ash-based geopolymer bricks are produced by activating Class F fly ash with an alkaline solution, typically a mix of sodium hydroxide and sodium silicate. The materials are thoroughly mixed to form a homogeneous paste, which is then molded and compacted into brick shapes. Curing is typically performed at room temperature or under heat (60–90 °C), depending on the level of strength development required. Experiments by Hardjito and Rangan (2005) and Palomo et al. (1999) have demonstrated that these bricks are robust in terms of compressive strength and durability, providing a sustainable solution to traditional fired clay bricks. Tests such as compressive strength, tensile strength, and flexural strength were performed on the geopolymer. For a comparative study, commercially available country bricks and fly ash bricks of the same size were also evaluated. The test samples were placed in a chamber and steam-cured at 600 °C for 24 h.

Compressive strength

The bar chart depicts the development of fly ash-based geopolymer brick compressive strength for curing temperatures of 7, 14, and 28 days. There is a clear upward trend where the compressive strength increases gradually from about 51 MPa at 7 days to about 55 MPa at 14 days, to about 63 MPa at 28 days. This continuous increase in strength with the passage of time is a common feature of geopolymer materials, especially those prepared from Class F fly ash and alkaline activators, like sodium hydroxide and sodium silicate. Comparable strength development trends have also been observed in other research. For instance, in the research conducted by Hardjito and Rangan (2005), fly ash-based geopolymer concrete showed a continuous improvement in compressive strength to 28 days, particularly when cured at room temperature or mild heating. The main chemical reaction that produces a

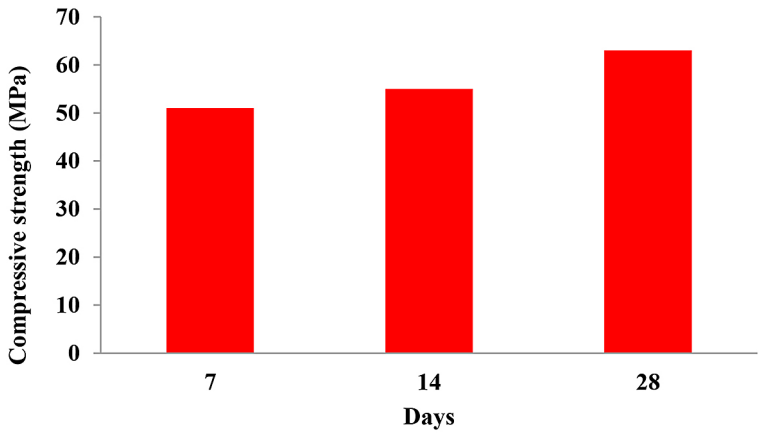

Figure 7. Effect of time on compressive strength

3D polymer chain structure is the response of the aluminosilicate present in fly ash to an alkaline solution. The structure becomes more dense and cross-linked over time, which improves mechanical performance (Hardjito et al., 2005). In addition, the research by Olivia and Nikraz (2012) revealed that compressive strength significantly increased with time when fly ash-based geopolymers were cured under ambient conditions. Their work supports the observation in the given Figure 7, where strength improves due to the continued dissolution of Si and Al species and the subsequent formation of geopolymeric gel phases like N-A-S-H (sodium alumino-silicate hydrate). This gradual formation and densification of gel phases contribute to the strength gain over extended curing periods (Olivia and Nikraz et al., 2012){Assi, 2016 #33}{Assi, 2016 #33}. {Assi, 2016 #33}

Flexural strength

The bar graph compares the flexural strength of three different brick types: geopolymer bricks, ordinary fired clay bricks, and fly ash bricks. The results show that geopolymer bricks exhibit the highest flexural strength, 31 MPa, followed by ordinary bricks, 23 MPa, and fly ash bricks, ~18 MPa. These networks dramatically increase the interparticle adhesion and bending load resistance. Moreover, the research conducted by Sukmak et al. (2013) confirms this finding, indicating that geopolymer bricks surpass ordinary and fly ash bricks in terms of flexural strength owing to the lower porosity and improved matrix integrity. Activated fly ash-based bricks dissolve in compressive and flexural strength, making them less robust compared to ordinary bricks and geopolymer bricks. However, their strength increases due to the advanced geopolymerization process, enabling a sustainable and high-performance material for construction purposes.

Water absorption

The following result, shown in Figure 8, highlights the dense microstructure and reduced porosity of geopolymer materials. The research by Temuujin et al. (2009) supports this observation, explaining that the geopolymerization process forms a compact aluminosilicate gel, which minimizes void spaces and capillary channels that typically allow water ingress. Further, studies by Olivia and Nikraz (2012) reported that geopolymer mortars and bricks exhibit superior impermeability and water resistance due to tightly bonded gel phases. In contrast, conventional fired bricks and fly ash bricks (with cementitious binders) tend to have higher porosity due to incomplete sintering or hydration, which results in higher water absorption{Sukmak, 2013 #32}{Sukmak, 2013 #32}{Sukmak, 2013 #1391}{Suhartana, 2022 #452}{Sukmak, 2013 #1391}{Sukmak, 2013 #1391}{Sukmak, 2013 #32}. The high absorption in fly ash bricks also indicates insufficient binder densification compared to alkali-activated systems (Olivia and Nikraz et al.). Therefore, the remarkably low water absorption in geopolymer bricks, as shown in the graph, affirms their suitability for applications where moisture resistance and durability are crucial. This property also contributes to longer life and reduced maintenance in structures (Figure 9).

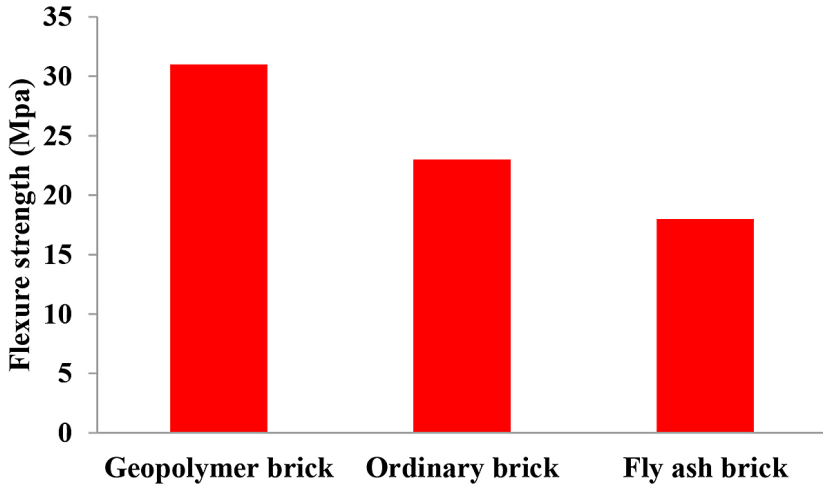

Figure 8. Flexural strength of different bricks

Discussion

The study presented aimed to evaluate the effects of alkaline activator in the preparation of geopolymers, particularly focusing on the morphological, compositional, and structural changes observed in 4M molar GP compared to 8M GP. As depicted in Figure 1, the 4M geopolymer (GP) displayed a compact and dense structure with a predominantly whitish appearance, indicative of its crystalline nature. The microstructures in GP were compact, with fewer, irregularly shaped pores. Such morphology suggests limited porosity and density typical of unmodified geopolymers. On the other hand, 8 M geopolymer showed a less dense and more homogeneous foam-like structure. Regular micro and macro pores were observed, contributing to the overall reduced density of higher alkaline concentrated GP. The introduction of a higher alkaline activator concentration effectively transformed the micro-porous structure of the untreated geopolymer into a highly porous matrix with a distinctly amorphous structure. This structural transformation implies enhanced properties, such as increased surface area and potential applications in lightweight construction materials and thermal insulation (Anand et al., 2025). Figure 2 shows the EDX composition of two different alkali activator concentrations on geopolymer samples. As it can be seen, the EDX spectra in the 4 M concentration peaks are sharper for the primary elements than at 8 M concentration, where peaks appear broader. This can be attributed to higher concentrated amounts of primary elements within a compact structure. The weight % of elements such as aluminum, silicon, iron, and oxygen appears to decrease as a result of increasing concentration from 4 M to 8 M. This indicates the major compositional structure of the whitish crystalline thick portion shown in the SEM image. On the other hand, the weight % of the elements such as sodium, magnesium, sulfur, calcium, and carbon tends to increase with increasing concentration. This shows the major constitutional structure of the treated sample. Higher concentration of carbon may have resulted in a higher number of micro-pores with an evenly distributed, uniform matrix. Moreover, it can enhance the chemical and thermal stability of the geopolymer, making it more resistant to acidic and high-temperature environment. Figure 3 shows the functional groups of two different alkali activator concentrations on geopolymer samples. In the 4M sample, the broad bands are strong due to weakly bound water molecules either adsorbed on the surface or trapped in the large cavities within the geopolymer rings. The weak peak at 1411 cm^{-1} is attributed to CO bending vibration, indicating the formation of sodium carbonate from the reaction of sodium hydroxide-rich geopolymer carbonation. On the other hand, FTIR analysis of 8 M alkali activator concentration produces. The geopolymer exhibits significant chemical and structural changes, resulting in improved surface properties and enhanced reactivity. By comparing the FTIR spectra of geopolymers produced with 4 M and 8 M alkali activator concentrations, it is possible to gain deeper insights into the chemical and structural modifications induced by increasing alkali activator concentration. Such changes result in enhanced mechanical strength, improved

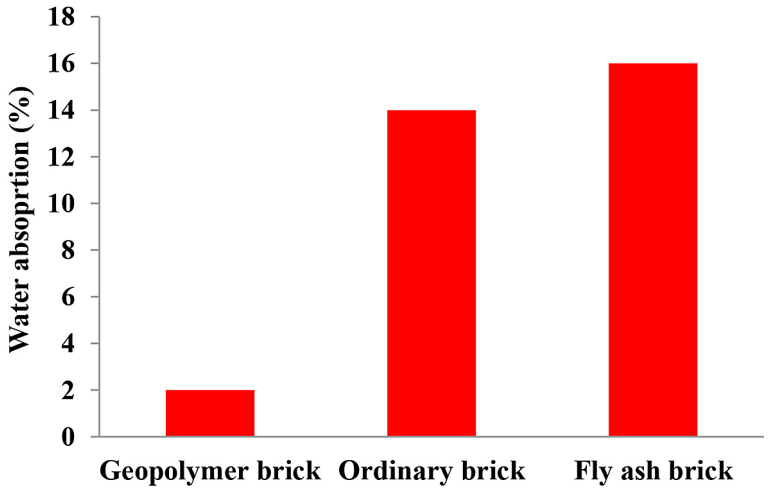

Figure 9. Water absorption of different bricks

thermal and electrical conductivity, and increased chemical resistance. The geopolymer is suitable for various advanced applications, including lightweight construction materials, thermal insulation components, and chemical-resistant structures. BET isotherm analysis, presented in Figure 5, further facilitated the understanding of pore characteristics in hydrogen peroxide-treated geopolymers. The steep increase in the adsorption curve at higher relative pressures ($P/P_0 \approx 1$) and the distinct hysteresis loop between the adsorption and desorption curves of 8M alkali activator concentration synthesized GP suggested the presence of mesopores to macropores. The total pore volume, derived from the amount of nitrogen gas adsorbed at a high relative pressure, was around 28.70 cm^3/g. The increased porosity significantly correlated with the reduced density and enhanced surface area of 8 M alkaline activator concentration. The analysis of the BET data revealed that the alkaline-activated 8M concentration geopolymer possessed an average pore size of 70 μm, a porosity of 34.8%, and a BET pore surface area of 30 m^2/g, respectively. This comprehensive study underscores the transformative effects of alkaline activators on the synthesis of geopolymer. The introduction of an increase in concentration of alkaline activators notably alters the morphology, composition, and structure of the geopolymer, leading to a more porous, amorphous material with enhanced surface properties, improved chemical and thermal stability, as well as increased adsorption capacity.

CONCLUSIONS

The influence of alkaline activator concentrations (4 M and 8 M NaOH solutions) on the structure of fly ash geopolymer synthesized for the production of materials to be applied as construction materials or for the formation of bricks was investigated. Results show that the alkaline activator concentration plays a significant role in geopolymerization and the ultimate properties of the material. At 4 M, the geopolymer indicated incomplete dissolution of the fly ash particles, leading to a lower level of geopolymerization. This was attributed to a more inhomogeneous microstructure with remnants of unreacted fly ash, a lower level of gel formation, and reduced mechanical strength. The limited availability of hydroxide ions at this concentration hindered the effective dissolution of aluminosilicate structures, resulting in a less dense and porous matrix. In comparison, the 8 M NaOH solution caused better dissolution enhancement of fly ash, resulting in higher geopolymer gel formation. The higher availability of OH^- ions led to a higher release of silicate and aluminate ions, resulting in a more uniform and closely packed microstructure. This resulted in higher structural strength, higher compressive strength, and higher amorphous geopolymer gel content, as confirmed by XRD and SEM characterizations. Overall, an increase in the alkaline activator concentration from 4 M to 8 M enhances the geopolymerization reaction as well as yields improved structural and mechanical properties. However, over-concentration (beyond 8 M, not explored within this study) may induce alkali saturation or efflorescence and requires levels of concentration that are optimized based on application requirements. On the basis of the findings, the fly ash geopolymers initiated with 8 M NaOH have sufficient mechanical strength and durability to qualify them for application in the manufacture of bricks. The produced geopolymer exhibits a dense microstructure and enhanced binding characteristics, showing excellent resistance to weathering. Therefore, this product presents a sustainable and environmentally friendly option to conventional clay bricks.

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