INFLUENCE OF PALM OIL FUEL ASH PARTICULATE INCORPORATION ON THE PHYSICO-MECHANICAL PERFORMANCE OF METAKAOLIN-BASED GEOPOLYMER PASTE

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ABSTRACT

Palm oil fuel ash (POFA) has attracted considerable attention as a sustainable supplementary material for geopolymer binders. However, variations in its chemical composition and physical characteristics arising from differences in source and processing conditions can significantly influence geopolymer performance. This study investigated the influence of POFA particulate incorporation on the physical and mechanical performance of metakaolin-based geopolymer paste. Metakaolin was partially replaced with POFA at levels of 0–30 wt.% and activated using sodium hydroxide and silicate solution. The raw materials and geopolymer binders were characterised using X-ray fluorescence (XRF), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), density, water absorption, compressive strength and flexural strength measurements. The results confirmed the successful transformation of kaolin into reactive metakaolin, while POFA exhibited a silica-rich composition and predominantly spherical particles. Increasing POFA incorporation reduced the bulk density from 2.05 to 1.80 g/cm³ and increased water absorption from 9.0 to 11.5%. Moderate POFA incorporation improved mechanical performance, with compressive strength increasing from 19.23 MPa to 23.83 MPa at 20 wt.% POFA, while the highest flexural strength of 5.71 MPa was obtained at 15 wt.% POFA. Beyond these optimum levels, mechanical performance decline. Overall, the study demonstrates that controlled particulate incorporation of POFA provides an effective strategy for improving the physico-mechanical performance of metakaolin-based geopolymer paste, with an optimum incorporation level of 15–20 wt.%. The findings further demonstrate the potential of combining locally available kaolin with agricultural biomass ash to develop sustainable geopolymer binders with enhanced engineering performance.

Keywords: Palm oil fuel ash, Geopolymer, Metakaolin, Sustainable binder, Mechanical properties.

1. Introduction

The construction industry underpins global economic development by providing the infrastructure required for residential, commercial and civil engineering applications. Cement remains the most widely manufactured construction material by mass, with annual production exceeding 30 billion tonnes and continuing to increase in response to rapid population growth, urbanisation and infrastructure development (Scrivener et al., 2018). However, the environmental sustainability of conventional concrete is increasingly challenged by its reliance on ordinary Portland cement (OPC), whose manufacture contributes approximately 7–8% of global carbon dioxide (CO_2) emissions(Andrew, 2017)(Scrivener et al., 2018). These emissions arise primarily from limestone decarbonation and the energy-intensive clinker production process, which also requires substantial consumption of fossil fuels and extraction of virgin raw materials (Habert et al., 2020). Consequently, the development of low-carbon cementitious binders capable of reducing environmental impacts while maintaining adequate engineering performance has become a major research priority.

Among the alternative binders proposed to reduce the environmental footprint of conventional cement, geopolymers have emerged as one of the most promising owing to their ability to utilise aluminosilicate-rich industrial and agricultural by-products while substantially lowering greenhouse gas emissions. Geopolymers are synthesised through the alkaline activation of reactive aluminosilicate precursors,

typically using sodium hydroxide and sodium silicate solutions, to form a three-dimensional aluminosilicate network (Davidovits, 1991)(Provis & Bernal, 2014). In addition to their lower embodied carbon relative to OPC, geopolymer binders exhibit excellent compressive strength, low drying shrinkage, superior resistance to sulphate and acid attack, and excellent thermal stability, making them attractive for sustainable structural and infrastructure applications(Provis, 2018).

Metakaolin is one of the aluminosilicate precursors investigated for geopolymer synthesis, due to its high purity, relatively uniform chemical composition and excellent reactivity resulting from the thermal dehydroxylation of kaolinite(Murray, 2004)(Duxson et al., 2005). Nevertheless, metakaolin-based geopolymers can be brittle and tend to exhibit limited tensile and flexural strengths, making them susceptible to crack initiation and propagation under service loading (Abhay Dhasmana, 2023)(Duxson et al., 2005). Furthermore, although geopolymers generally exhibit lower environmental impacts than OPC, the production of metakaolin and alkaline activators remains energy-intensive, providing further motivation for incorporating renewable agricultural residues capable of partially replacing conventional geopolymer constituents while simultaneously valorising biomass wastes (Heath et al., 2014). Consequently, improving both the structural reliability and sustainability of metakaolin-based geopolymers has become an active area of research. Strategies including fibre reinforcement, nano-modification, hybrid precursor systems and particulate incorporation have been widely investigated to refine the geopolymer matrix and enhance engineering performance (Nidhi et al., 2026)(Kamal et al., 2025)(Wang et al., 2025).

Agricultural residues have increasingly attracted attention because they simultaneously address waste disposal challenges while providing renewable materials with beneficial physical and chemical characteristics for cementitious systems. Among these residues, palm oil fuel ash (POFA) is generated during the combustion of palm kernel shells, fibres and empty fruit bunches for steam and electricity generation in palm oil mills. The continuous expansion of the palm oil industry, particularly in Malaysia, Indonesia, Thailand and Nigeria, has resulted in increasing quantities of POFA, much of which is disposed of in landfills or open dumping sites, creating significant environmental management challenges (Ayub et al., 2021). Owing to its high silica content, relatively low specific gravity, fine particle size and pozzolanic characteristics, POFA has been widely recognised as a valuable supplementary material for sustainable construction, transforming an agricultural by-product into a value-added engineering material (Boateng et al., 2025)(Nadirah et al., 2025).

Previous studies have investigated the utilisation of POFA in both conventional cementitious materials and geopolymer systems (Huseien, 2026)(Nadirah et al., 2025)(Tan & Awang, 2026). Within geopolymer systems, POFA has primarily been employed either as a reactive aluminosilicate precursor or as a partial replacement for fly ash, slag and metakaolin. For example, (Chub-uppakarn et al., 2023) investigated the partial substitution of metakaolin with palm oil fuel ash in geopolymer mortar and reported that replacing 10% of metakaolin with POFA increased the 28-day compressive strength by 22.08%, which was attributed to improved geopolymerisation. More recently, Sakrungrueang et al., (2026) incorporated corncob aggregates into metakaolin-based geopolymers and demonstrated that increasing biomass content reduced bulk density and compressive strength owing to increased pore formation, while simultaneously improving thermal insulation, acoustic performance and moisture buffering capacity. Collectively, these studies demonstrate that both the type and incorporation level of biomass-derived materials exert a significant influence on the physical, mechanical and functional performance of metakaolin-based geopolymers.

Accordingly, this study investigates the influence of palm oil fuel ash particulate incorporation on the physico-mechanical behaviour of metakaolin-based geopolymer composites activated using sodium hydroxide and sodium silicate solutions. POFA was incorporated at varying replacement levels to evaluate its effects on density, water absorption, compressive strength and flexural strength, while X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were employed to characterise the raw materials and provide insight into the material characteristics associated with the observed performance. The findings are expected to improve understanding of the role of agricultural waste particulates in modifying the structure–property

relationships of metakaolin-based geopolymer composites while promoting the sustainable utilisation of palm oil fuel ash.

2. Materials and Experimental Methods

2.1 Materials

Kaolin and palm oil fuel ash (POFA) were used as the starting materials in this study. The kaolin was subsequently calcined to produce metakaolin (MK), which, together with POFA, served as the aluminosilicate precursors for geopolymer synthesis. The kaolin was sourced from Yala Local Government Area, Cross River State, whereas the POFA was obtained from a palm oil processing facility in Akamkpa Local Government Area, Cross River State, both in Nigeria. Analytical-grade sodium hydroxide (SH) pellets (98% purity) and commercial sodium silicate (SS) solution with a silica modulus of 2.5 were procured from a chemical supplier in Cross River State, Nigeria.

2.2 Preparation of Precursors

The as-received POFA was oven-dried at 100°C for 2 h to remove residual moisture and sieved through a 75 µm sieve. The fraction passing the sieve was collected and stored in airtight containers until use. The raw kaolin was calcined in a muffle furnace at 700°C at a rate of 5°C/min to produce MK, following the procedure reported by (Enoh & Ushie, 2023). After calcination, the MK was allowed to cool naturally inside the furnace to room temperature before being stored in sealed airtight containers until use.

2.3 Preparation of Alkaline Activator

A 10 M SH solution was prepared by dissolving the required quantity of analytical-grade SH pellets in distilled water. Owing to the exothermic nature of the dissolution process, the solution was allowed to cool to ambient temperature before use.

The alkaline activator was prepared by mixing the SH solution with SS solution at a sodium silicate-to-sodium hydroxide solution mass ratio (SS/SH) of 1.0. The combined solution was mechanically stirred for approximately 1 min to obtain homogeneity before using immediately. The selected SH concentration and SS/SH ratio were based on the optimum activator composition established in the authors' previous study on metakaolin-based geopolymers (Enoh & Ushie, 2023).

2.4 Mix Design

Metakaolin was partially replaced with POFA at replacement levels of 0, 5, 10, 15, 20, 25 and 30 wt.%. The control mixture, (P0) contained 100 wt.% metakaolin, while the remaining mixtures incorporated progressively increasing proportions of POFA. The total precursor mass was maintained constant at 450g for all mixtures.

Owing to differences in the physical characteristics of metakaolin and POFA, particularly particle morphology and porosity, the total alkaline content was adjusted with increasing POFA content to produce a homogeneous and workable geopolymer paste. The increase in solid-liquid (S/L) ratio with increasing POFA content reflects the lower activator demand required to obtain a workable geopolymer paste. However, the SS/SH ratio was maintained at 1.0 for all mixtures. The detailed mix proportions are presented in (Table 1).

Table 1: Mix proportions of MK-POFA geopolymer pastes

Mix ID	MK (wt.%)	POFA (wt.%)	MK (g)	POFA (g)	SH solution(g)	SS solution (g)	Total activator (g)	S/L ratio
P0	100	0	450.0	0	186.6	186.6	373.2	1.20
P5	95	5	427.5	22.5	182.9	182.9	365.8	1.23
P10	90	10	405.0	45.0	180.0	180.0	360.0	1.25
P15	85	15	382.5	67.5	174.4	174.4	348.8	1.29
P20	80	20	360.0	90.0	171.8	171.8	343.6	1.31
P25	75	25	337.5	112.5	166.8	166.8	333.6	1.35
P30	70	30	315.0	135.0	166.8	166.8	333.6	1.35

MK = metakaolin; POFA = palm oil fuel ash; S/L = solid-to-liquid ratio

2.5 Specimen preparation and curing

The geopolymer specimens were prepared using a conventional two-part activation procedure. Metakaolin and POFA were dry-mixed for 5 min, after which the alkaline activator was gradually added and mixed for a further 10 min to produce homogeneous paste. The fresh paste was cast into moulds and compacted on a vibrating table for approximately 3 min to remove entrapped air. The mixing and vibration durations were adopted from the authors' previous study (Enoh & Ushie, 2023).

Cube specimens (50 × 50 × 50 mm) were prepared for compressive-strength testing, while prism specimens (40 x 40 x 160 mm) were cast for the three-point flexural test.

After casting, the specimens were left in the moulds at ambient temperature for 24 h, demoulded and cured at room temperature (27 ± 2°C) for 28 days. Three replicate specimens were prepared for each mixture, and the average was reported.

2.6 Testing and characterisation methods

2.7.1 Density

The bulk density of the hardened geopolymer specimens was determined in accordance ASTM C642 by calculating the ratio of the specimen mass to its measured volume . Three specimens were tested for each mixture and the average value reported.

2.7.2 Water absorption

Water absorption was determined in accordance with ASTM C642. The specimens were oven-dried to constant mass before immersion in water for 24 h. The percentage water absorption was calculated from the difference between the oven-dry and saturated masses.

2.7.3 Compressive and flexural strength

Compressive strength was determined in accordance with ASTM C109/C109M while flexural strength was determined using the three-point bending method in accordance with ASTM C293/C293M after 28 days of curing. All tests were performed using a universal testing machine. Three specimens were tested for each mixture, and the average value was reported.

2.7.4 X-ray diffraction analysis

X-ray diffraction (XRD) analysis was performed to identify the crystalline phases of the raw materials and selected geopolymer specimens using a diffractometer with Cu-Kα radiation (λ = 1.5406 Å). Powdered samples were scanned over a 2θ range of 5–70°. The diffraction patterns were compared with standard reference data to identify crystalline phases and evaluate changes in the amorphous hump associated with geopolymerisation.

2.7.5 Scanning electron microscopy

The morphology of the raw POFA and metakaolin was examined using a scanning electron microscope (SEM) to evaluate their particle morphology and surface characteristics.

3. Results and Discussion

3.1.1 Chemical Composition of the Raw Materials

The oxide compositions of the raw kaolin, metakaolin and POFA determined by XRF are presented in Table 2. Both the raw kaolin and MK are predominantly composed of silica (SiO_2) and alumina (Al_2O_3), confirming their suitability as aluminosilicate precursors for geopolymer synthesis (Duxson et al., 2007)(Davidovits, 1991)(Provis & Van Deventer, 2009). Following calcination, only slight changes in oxide composition were observed, indicating that calcination primarily induces the dehydroxylation of kaolinite to form reactive amorphous metakaolin without significantly altering its overall chemical composition.

POFA exhibited a distinctly different composition, containing appreciable amounts of silica together with relatively high concentrations of CaO and K_2O. The elevated potassium content is characteristic of biomass ashes owing to the accumulation of alkali metals during plant growth and combustion (Vassilev et al., 2013), while the calcium content may promote the formation of calcium-containing aluminosilicate hydrate (C-(N)-A-S-H) gels alongside the conventional N-A-S-H gel during geopolymerisation (Provis & Bernal, 2014)(Provis, 2018)

Table 2: Oxide composition of the raw materials determined by XRF

Oxide	Raw Kaolin	Metakaolin	POFA
SiO_2	56.67	58.35	35.55
Al_2O_3	36.26	34.79	3.13
Fe_2O_3	1.65	1.53	5.14
CaO	0.60	0.50	19.38
K_2O	1.98	2.02	16.47
SO_3	0.42	-	8.90
TiO_2	0.19	0.08	0.44
Cl	1.45	0.92	1.41

3.1.2 X-ray Diffraction Analysis

Figure 1 presents the XRD patterns of the raw kaolin, metakaolin (MK), and palm oil fuel ash (POFA). The raw kaolin exhibited distinct crystalline reflections, with prominent peaks at approximately 12.3° and 24.8° 2θ corresponding to kaolinite. Additional crystalline phases identified were quartz, albite, muscovite, and hematite, with kaolinite accounting for approximately 59 wt.% of the crystalline phases. Following calcination, the characteristic kaolinite reflections disappeared and were replaced by a broad amorphous hump between approximately 18° and 30° 2θ, confirming the dehydroxylation of kaolinite and its transformation into amorphous metakaolin. Quartz, albite, muscovite, and hematite remained detectable, reflecting the thermal stability of these minerals. The apparent increase in the relative quartz content after calcination resulted from the amorphisation of kaolinite rather than the formation of additional quartz.

The POFA exhibited sharp crystalline reflections superimposed on a broad diffuse background, indicating the presence of both crystalline and amorphous phases. Major reflections at approximately 21° and 26.7° 2θ are consistent with silica-bearing phases such as quartz and cristobalite, which have been widely reported in POFA.

Overall, the XRD results confirm the successful conversion of kaolinite to reactive amorphous metakaolin while retaining the thermally stable accessory minerals. The mixed crystalline–amorphous nature of POFA suggests that its contribution to geopolymerisation depends primarily on the availability of its amorphous

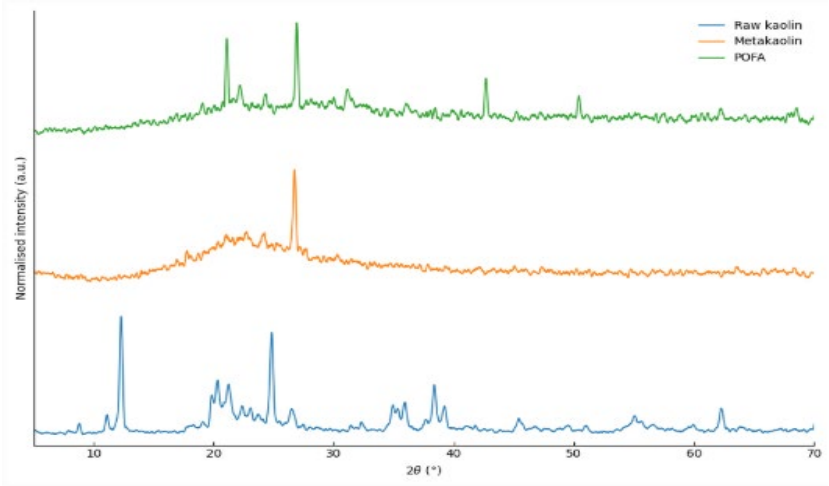

Figure 1: XRD graphs of kaolin, metakaolin and POFA

3.1.3 Fourier-transform infrared spectroscopy

Figure 2 presents the FTIR spectra of the raw kaolin, metakaolin, and POFA. The raw kaolin exhibited characteristic hydroxyl stretching bands at approximately 3675 and 3623 cm^{-1}, together with an Al–OH deformation band at 910 cm^{-1}, confirming the presence of structurally ordered kaolinite. These bands disappeared after calcination, indicating the dehydroxylation of kaolinite and the formation of

amorphous metakaolin. This observation is consistent with the XRD results, which showed the disappearance of the characteristic kaolinite reflections following calcination.
Calcination also altered the main silicate absorption band, shifting it from 1103–1006 cm^{-1} in the raw kaolin to a broader band centred at approximately 1059 cm^{-1} in metakaolin. The broadening of this band reflects the formation of a more disordered aluminosilicate structure. A band at approximately 770 cm^{-1}, attributed to quartz, remained in both materials, indicating that quartz was largely unaffected by calcination.

The FTIR spectrum of POFA was dominated by a broad Si–O–Si stretching band at approximately 1014 cm^{-1}, confirming its silica-rich nature, while weak bands around 3310, 1633, and 1416 cm^{-1} were attributed to adsorbed moisture and minor carbonate species. Overall, the FTIR results confirm the successful conversion of kaolin to reactive metakaolin and the presence of silica-rich functional groups in POFA that are beneficial for geopolymer synthesis.

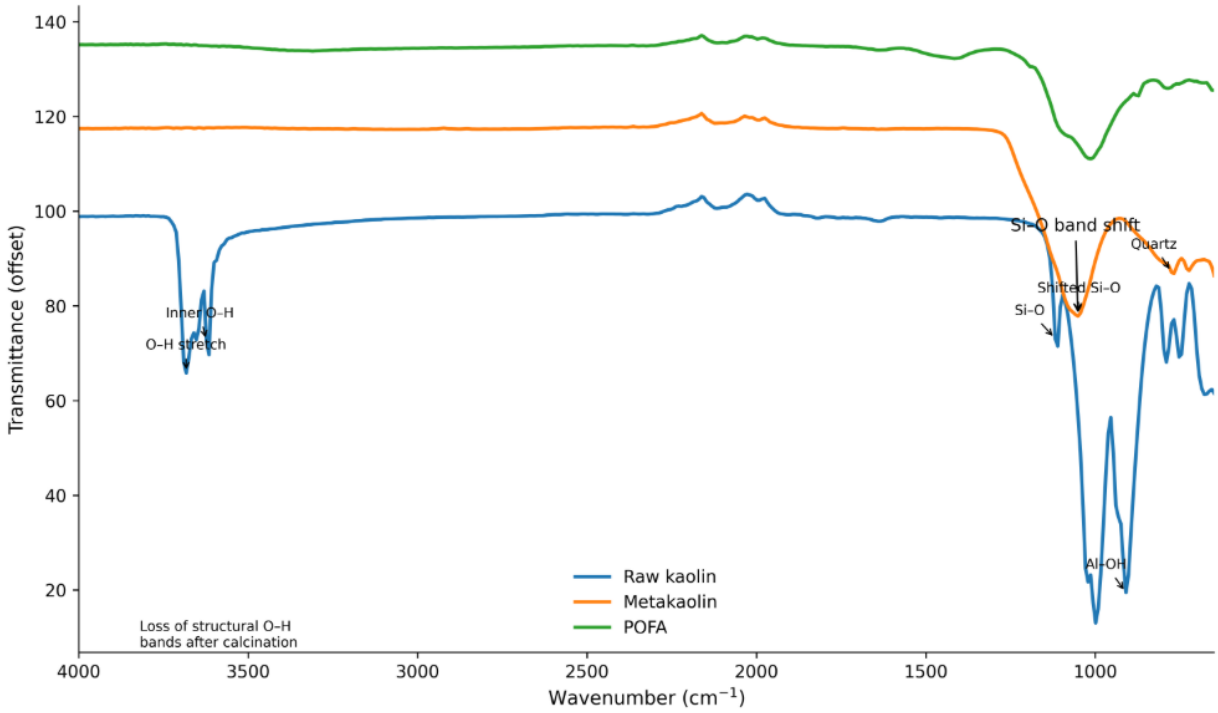

Figure 2: FTIR spectrum for raw kaolin, metakaolin and POFA

3.1.4 Scanning Electron Microscopy (SEM)

Figure 3 presents the SEM micrographs of the metakaolin and POFA. The metakaolin exhibits irregular, angular, and agglomerated particles with rough, fractured surfaces, reflecting the structural disruption of kaolinite during calcination. In contrast, the POFA consists predominantly of spherical to sub-spherical particles with a wide particle-size distribution. Numerous fine particles adhered to the surfaces of the larger particles, which is likely attributable to the absence of post-combustion grinding. The contrasting morphologies suggest differences in particle packing and reactivity, which may influence the geopolymerisation process.

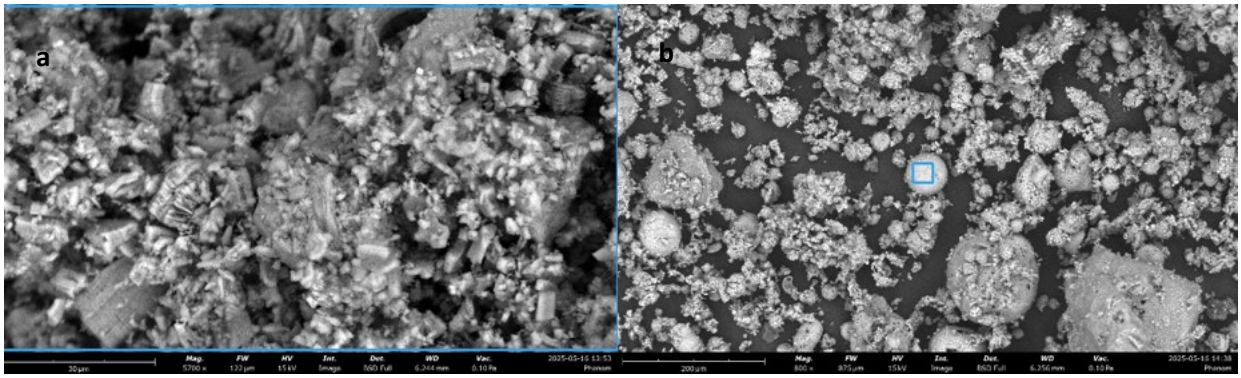

Figure 3: SEM image for (a)metakaolin and (b)palm oil fuel ash

3.1.5 Density and water absorption

Figure 4 illustrates the effect of POFA incorporation on the physical properties of the metakaolin-based geopolymer pastes. Increasing the POFA content from 0 to 30 wt.% reduced the density from 2.05 to 1.80 g/cm³, while the corresponding water absorption increased from 9.0% to 11.5%. The reduction in density is primarily attributed to the lower specific gravity of POFA relative to metakaolin, resulting in a lighter geopolymer matrix. Conversely, the increase in water absorption indicates a progressive rise in accessible porosity with increasing POFA content. Although the spherical morphology of POFA may enhance particle packing, the replacement of reactive metakaolin likely reduced the availability of reactive aluminosilicate species for geopolymerisation, resulting in a less dense matrix with greater interconnected porosity. Consequently, more water was absorbed by the hardened specimens.

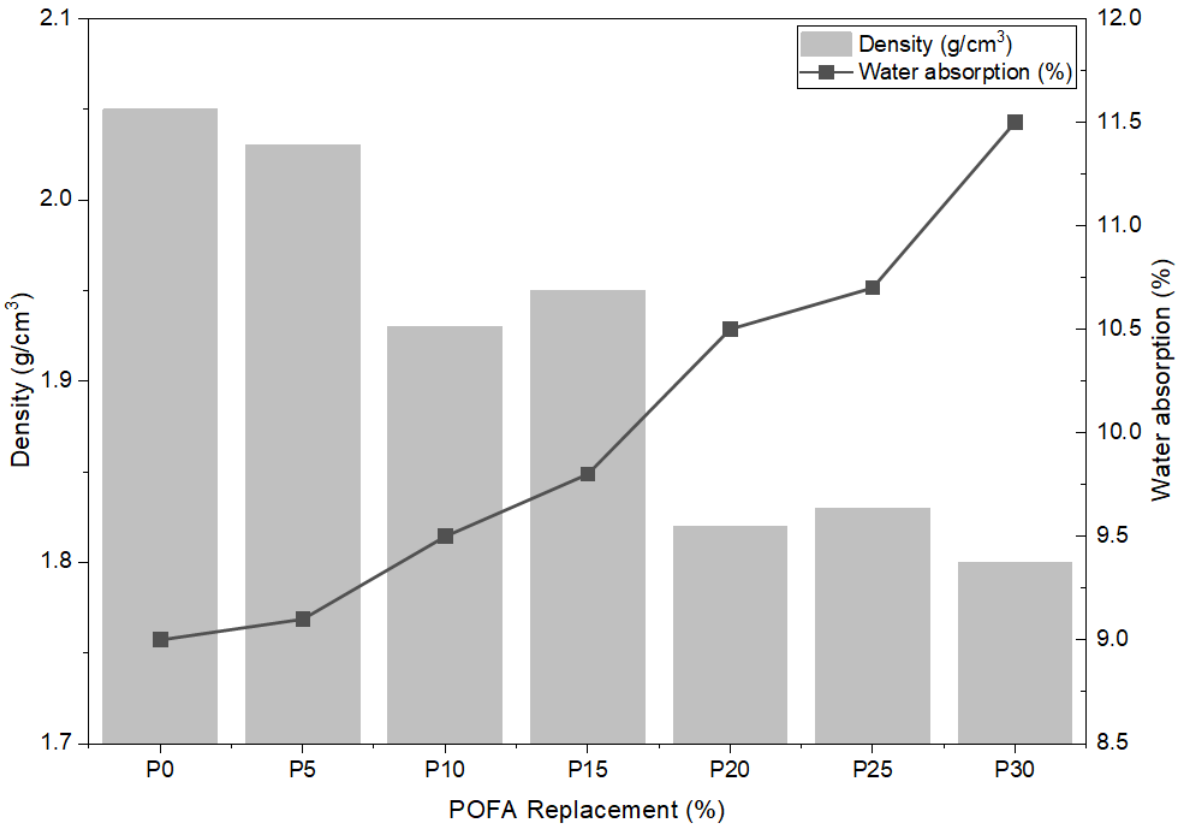

Figure 4: Effect of palm oil fuel ash (POFA) content on the density and water absorption of metakaolin-based geopolymer paste

3.1.6 Compressive and Flexural Strength

Figure 5 presents the compressive and flexural strengths of the metakaolin-based geopolymer composites containing different POFA replacement levels. Both properties improved with increasing

POFA content up to an optimum replacement level, beyond which strength declined. The compressive strength increased from 19.23 MPa for the control to a maximum of 23.83 MPa at 20 wt.% POFA, corresponding to an improvement of approximately 24%. Similarly, the flexural strength increased from 4.52 MPa to 5.71 MPa at 15 wt.% POFA, representing an increase of approximately 26%, before decreasing at higher replacement levels.

The initial improvement in strength can be attributed to the combined filler and chemical effects of POFA. The predominantly spherical morphology of the POFA particles enhanced particle packing, reducing interparticle voids and promoting a denser matrix. In addition, the silica-rich composition of POFA provided an additional source of reactive silicate species, facilitating the formation of geopolymeric reaction products. These observations are consistent with the XRF, FTIR and SEM analyses, which confirmed the presence of abundant silica in the POFA and a morphology favourable for improved particle packing. Similar improvements at moderate replacement levels have been reported for biomass ash-containing geopolymers, where finely divided ashes enhance microstructural densification and contribute to strength development.

Beyond the optimum replacement level, both compressive and flexural strengths declined, although the reduction was more pronounced for compressive strength. At higher POFA contents, the replacement of reactive metakaolin became sufficiently large to reduce the extent of geopolymerisation, resulting in fewer binding gel products and a less cohesive matrix. This interpretation is supported by the accompanying decrease in density and increase in water absorption observed for the higher POFA mixtures, indicating increased pore connectivity and reduced matrix compactness. The earlier peak in flexural strength (15 wt.%) compared with compressive strength (20 wt.%) suggests that flexural performance is more sensitive to microstructural defects and pore development, whereas compressive strength remains comparatively tolerant until higher replacement levels are reached.

Overall, the results indicate that 15–20 wt.% POFA provides the most favourable balance between physical and mechanical performance. Within this range, the beneficial filler effect and additional reactive silica supplied by POFA outweigh the dilution of the metakaolin precursor, whereas excessive replacement compromises the continuity of the geopolymer matrix and leads to strength deterioration.

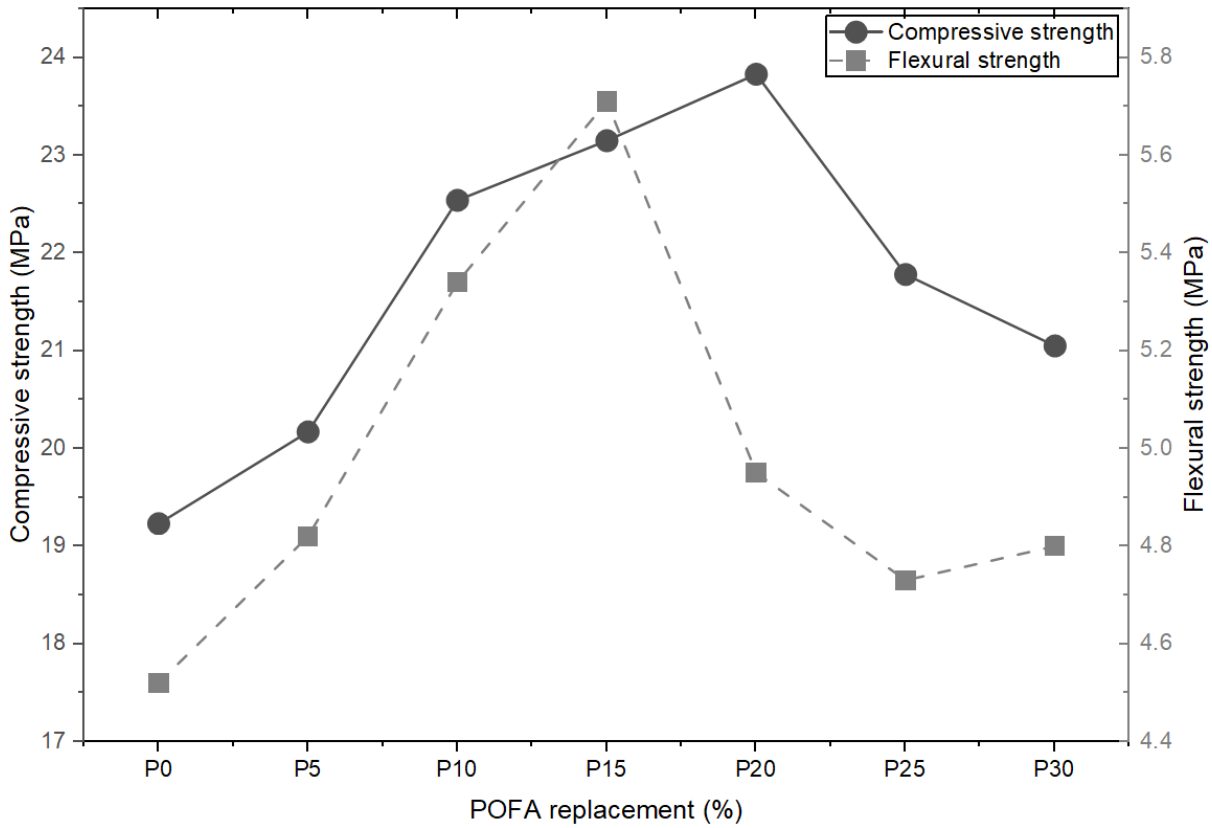

Figure 5: Compressive and flexural strength of the geopolymer pastes

4. Conclusion

This study investigated the influence of palm oil fuel ash (POFA) incorporation on the physical and mechanical properties of metakaolin-based geopolymer paste. The principal findings are summarised as follows:

1. Calcination successfully transformed kaolin into reactive metakaolin, while POFA exhibited a silica-rich composition and predominantly spherical particles, indicating its suitability as a supplementary geopolymer precursor.
2. Increasing POFA content reduced the density from 2.05 to 1.80 g/cm³ and increased water absorption from 9.0 to 11.5%, indicating a gradual increase in matrix porosity with higher replacement levels.
3. Moderate POFA incorporation improved mechanical performance, with the compressive strength increasing by approximately 24% to 23.83 MPa at 20 wt.% POFA, while the flexural strength increased by approximately 26% to 5.71 MPa at 15 wt.% POFA.
4. Further increases in POFA content reduced both compressive and flexural strengths, indicating that excessive replacement of metakaolin adversely affected the geopolymer matrix.
5. An optimum POFA incorporation of 15–20 wt.% provided the best balance between density, water absorption, and mechanical performance.

Overall, the results demonstrate that POFA can be effectively incorporated into metakaolin-based geopolymer paste at moderate replacement levels, providing a sustainable route for the valorisation of palm oil fuel ash while reducing the consumption of virgin aluminosilicate materials. The findings contribute to the development of low-carbon geopolymer binders with improved resource efficiency and enhanced sustainability. Future research should focus on the long-term durability, microstructural evolution, and environmental performance of POFA-modified geopolymers to further establish their suitability for structural and construction applications.

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