UJEAS

UNIZIK JOURNAL OF ENGINEERING AND APPLIED SCIENCES

UNIZIK Journal of Engineering and Applied Sciences 5(3), September (2025), 2689-2699
Journal homepage: https://journals.unizik.edu.ng/index.php/ujeas
PRINT ISSN: 2992-4383 || ONLINE ISSN: 2992-4391

Study on the Improvement of Physical and Mechanical Properties of Ugwuoba Clay Using Palm Stalk Ash

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Abstract
This study investigates the enhancement of the microstructural, physical and mechanical properties of Ugwuoba clay through the incorporation of palm stalk ash (PSA) as a reinforcing additive. Ugwuoba clay reinforced with 10wt.%, 20wt.%, 30wt.%, and 40wt.% of PSA were prepared and tested for flexural strength, refractoriness, thermal shock resistance, and other physico-mechanical properties. Results indicate that the inclusion of PSA has a significant influence on the microstructure of the Ugwuoba clay. Additionally, the addition of PSA enhances the flexural strength but reduces both refractoriness and thermal shock resistance with an increasing weight fraction of PSA. Also, the physic-mechanical properties (linear shrinkage, bulk density, modulus of rupture, apparent porosity, and water absorption) of one of the reinforced samples (30wt.% PSA reinforced Ugwuoba clay composite) at increasing sintering temperature between 900°C to 1300°C was compared to those of the unreinforced Ugwuoba clay. Results show significant influence on the physico-mechanical properties with increasing sintering temperature. The study demonstrates that PSA can effectively enhance the mechanical properties of Ugwuoba clay with minimal reduction in its thermal properties. However, this approach provides an insight into sustainable utilisation of agricultural waste and contributes to value-added material development, while further studies can be carried out to improve on the thermal properties affected. These findings highlight PSA's potential as a sustainable reinforcement in clay-based ceramics for moderate thermal applications such as kiln linings, insulating bricks, and structural ceramics.

Keywords: Ugwuoba clay, palm stalk ash, clay composites, physicomechanical properties.

1. Introduction

Clay materials are extensively utilized in the production of refractories, ceramics, and other structural components due to their plasticity and thermal resistance. However, native clays often lack the mechanical robustness and thermal shock resistance required for advanced applications (Ilyina et al., 2023). These native clays have deposits in different part of the country including Ugwuoba clay, Kankara clay, Ise-Ekiti clay, Dukku, Yauri, and others (Salihu and Suleiman, 2018; Jide, 2014) Among these, Ugwuoba clay which is the focus of this study is notable for its suitability for refractory bricks production (Ozoekwem et al., 2024). The Ugwuoba clay deposit, located in Enugu State, southeastern Nigeria, is a widely available kaolinitic clay known for its accessibility and abundance with promising chemical characteristics but moderate physical properties (Ifeanyichukwu et al., 2024). However, like many naturally occurring clays, it has limitations in its as-mined state, especially in meeting the demanding requirements for high-temperature applications such as foundry crucibles (Dawodu et al., 2012). These limitations include inadequate thermal resistance, low refractoriness, and poor mechanical strength when subjected to repeated thermal cycling (Dawodu et al., 2012). Thus, there is a growing need to improve the properties of Ugwuoba clay to expand its industrial applications.

Interestingly, recent years have witnessed composite technology as a viable means of enhancing the properties of engineering materials including ceramic-based materials. Reinforcements (particles) derived from agricultural wastes such as rice husk ash, palm kernel shell ash, and coconut shell ash have shown promising results in improving thermal, mechanical, and microstructural characteristics of clay-based composites (Wang et al., 2024). These agro-based particles are rich in silica and alumina, which are key components in refractory materials. In the use of PSA as an agro-based reinforcements, some studies have shown its significant influence on various matrices used. Srisang and Srisang (2020) reported that PSA inclusion in a clay based composite influenced the bulk density and acid corrosion thereby making it suitable for tiles production. Chikwado et al., (2020) studied a PSA reinforced polyethylene composite and reported an increase in tensile strength at 50%, thereby increasing the brittleness of the material and reducing the ductility. The authors report showed that a proper mixture of palm trunk ash and low-density polyethene composite is good engineering material for reinforcement loading. They also reported a decrease in flexural strength with increasing PSA to 50% level also decreases the flexural strength of the composite. In another study by Chinnu et al., (2022) to investigate and compare the influence of PSA in concrete with some non-agro-waste ashes. The water absorption of the PSA reinforced concrete is at close range with the non-agro-wastes while 10wt.% PSA was reported to give the optimum combination of properties. These summarises the effects of PSA as a reinforcement in differen matrix materials.

It should be noted that the integration of agro waste-derived particles into clay matrices and of course others matrices, not only improves material performance but also aligns with sustainable development goals through waste valorisation and environmental conservation (Rozhkova et al., 2025). Therefore, this study seeks to enhance the physical and mechanical properties of Ugwuoba clay by incorporating PSA particles synthesised from selected agro-wastes, with the aim of producing high-performance foundry crucibles..

2.0 Materials and methods

2.1 Material Collection and Processing

For this study, Ugwuoba clay samples were collected using ASTM D4220/D4220M-14 guidelines from Ugwuoba clay deposits in Afikpo, Oji-River Local Government Area of Enugu State. PSA, which is the filler material, was sourced from Ugwuoba Garriki, Oji-River L.G.A., Enugu State. The collected Ugwuoba clay sample was soaked, washed, and filtered using a sieve of 150μm to obtain fine particle sizes. The sieved samples were sun-dried and subsequently oven-dried. The PSA was also sun-dried, crushed and ground before being loaded into the muffle furnace. The furnace was set at 650oC for 3 hours to allow for proper calcination into amorphous ash. Subsequently, the clay composite samples were mixed to varying proportions of clay to PSA in 90:10, 80:20, 70:30, and 60:40.

2.2 Material Tests

2.2.1 Physical Properties Test:

The bulk density, apparent porosity, and water absorption of the samples were determined by the ASTM C20 standard. In this test, the dried samples were weighed to determine the dry weight (D_w). The samples were then suspended in water and boiled for 2 hours. After this, the heat is turned off and the samples are suspended for a minimum of 12 hours in water. While being suspended in water, the samples are weighed to determine their suspended weight (S_w). Finally, samples were removed from the water and cleaned with a moistened cotton cloth to remove all drops of water from the surface and to determine the saturated weight (ST_W).

Given that V = $ST_w - S_w$ (1)

Bulk Density: $\frac{D_w}{V}$ (2)

Apparent Porosity (%) = $\left[\left(ST_w - \frac{D_w}{V}\right) X\ 100\right]$ (3)

Water Absorption (%) = $\left[\left(\frac{ST_w - D_w}{D_w}\right)\right] X\ 100$ (4)

2.2.2 Microstructural Test

The microstructure of the samples was examined by the use of SEM (Scanning Electron Microscopy). In carrying it out, the working distance was 10.4 mm, and the accelerating voltage used was 20 kV, which is a standard setting that enables deep penetration of the electron beam for high-resolution imaging. The horizontal field width (HFW) is 126 microns, and the pressure was maintained at 70 Pa.

To determine the elemental oxide composition of the Ugwuoba clay samples, X-ray Fluorescence (XRF) analysis was employed due to its accuracy, speed, and non-destructive nature. The raw clay samples were first sun-dried to remove moisture and then oven-dried at 105°C for 24 hours to eliminate any residual water. Same thing was done to the PSA samples. The clay samples were mixed with PSA samples using percentage weight fractions in the ratios 60:40, 70:30, 80:20, and 90:10. The dried composite samples were then crushed and ground using an agate mortar and pestle to obtain a fine powder (<75 μm), ensuring homogeneity. Approximately 5 grams of the powdered sample were mixed with a few drops of boric acid as a binder to facilitate pellet formation. The mixture was then pressed into pellets using a hydraulic press under a pressure of 20–25 tons to form a uniform and compact disc suitable for XRF analysis. The pellets were subjected to X-ray Fluorescence Spectrometry (XRF) using a calibrated XRF spectrometer. The XRF machine was operated under standard conditions, and each sample was scanned multiple times to ensure reproducibility and accuracy. The XRF software automatically quantified the oxide concentrations based on the intensity of fluorescence emitted by each element. Results were expressed in weight percentage (wt%) and validated using known reference standards to ensure analytical reliability.

2.2.3 Mechanical Characteristics

Flexural strength (FS) was measured using a three-point bending test. The specimens for the test where cut into bar-shapes measuring 120 mm × 20 mm × 15 mm. Refractoriness was assessed via pyrometric cone equivalent (PCE) analysis, and thermal shock resistance (TSR) was evaluated by subjecting samples to rapid thermal cycling according to ASTM C 484 standard. Thermal shock resistance was carried out using the prism spalling test method according to ASTM 484 in which the spalling resistance was measured by the number of thermal cycles (heating, cooling and testing for failure).

Linear Shrinkage: The rectangular bar test pieces of dimension 8.0cm x 4.1cm x 2.1cm were molded, and a mark 5cm apart was made at their centres. The green samples were allowed to dry at room temperature, while the others were dried at room temperature and then fried to the corresponding temperatures intended for them in a kiln. All shaped samples were air-dried for 24 hours and then oven-dried at 110 °C for 12 hours to remove residual moisture. The dried samples were then sintered in a muffle furnace at temperatures of 900 °C, 1000 °C, 1100 °C, and 1200 °C to observe progressive thermal and mechanical property changes. The sintering was carried out in an oxidizing atmosphere (air) to simulate typical ceramic processing environments. A dwell time of 2 hours at each sintering temperature was maintained to ensure complete densification and reaction between the clay and PSA phases. Heating and cooling rates were maintained at 5 °C/min to avoid thermal shock. The linear shrinkage was determined using the equation.

The shrinkage was then calculated

$$\text{Linear Shrinkage} = \left(\frac{L_d - L_f}{L_d}\right) \quad (5)$$

Where;

Ld = Length of the sample after drying (before firing)

Lf = Length of the sample after firing (sintering).

3.0 Results and Discussion

The chemical composition of Ugwoba clay, as presented in Table 1a, provides insight into its suitability for various industrial applications such as ceramics, refractories, and foundry operations. The chemical analysis of Ugwoba clay shows it to be a silica-alumina-rich material with low concentrations of deleterious oxides. Its moderate Al_2O_3 content and relatively low Fe_2O_3, CaO, and MgO values make it suitable for refractory, ceramic, and foundry applications. The major and minor oxides present, alongside the loss on ignition (LOI), indicate its mineralogical profile, potential plasticity, refractoriness, and overall chemical behavior during thermal processing. Silicon dioxide is the predominant oxide in Ugwoba clay, comprising about 64.87% of the material. High silica content is typical of kaolinitic and illitic clays and contributes to the clay's refractoriness and thermal stability. The high silica content suggests good potential for ceramic and refractory applications, provided the fluxing agents are controlled (Ifeanyichukwu et al., 2024). Alumina is the second most abundant oxide in the clay, which is crucial for imparting refractoriness, mechanical strength, and chemical resistance. The relatively high Al_2O_3 content suggests the presence of kaolinite or other aluminosilicates and makes the clay suitable for refractory linings and structural ceramics (Maj and Matus, 2023). The SiO_2/Al_2O_3 ratio (~3.15) indicates that the clay has a moderate alumina-silica balance, characteristic of kaolinitic clays with good refractory potential. Iron oxide content of 4.75% is relatively moderate. While iron oxide can act as a fluxing agent and reduce the sintering temperature, it also imparts a reddish coloration to the fired clay and can negatively affect refractoriness at high concentrations (Gikunju et al., 2024). Other minor components such as calcium oxides and magnesium oxides acts as minor fluxing agents and also suggest minimal risks of excessive shrinkage as against if they are in large quantities (Gikunju et al., 2024). .

Table 1b shows the chemical composition of PSA reveals its potential as a promising additive in construction materials, particularly for pozzolanic, ceramic, or composite applications. The oxide constituents indicate useful fluxing properties, moderate pozzolanicity, and a capacity to reduce sintering temperatures when blended with other materials such as clay. PSA contains a high level of silica, making it a good candidate for pozzolanic reactions and ceramic use. Additionally, the high silica can assist in forming glassy phases in ceramics, enhancing vitrification and surface finish. The table shows that the PSA has a very high K_2O content, making PSA an effective fluxing agent. Potassium lowers the melting point of silica and alumina phases, promoting sintering at lower temperatures in ceramic applications. Combined alkali (K_2O and Na_2O) content is high. This is advantageous in ceramics, where fluxing is required, but in cement, it requires careful control to avoid deleterious reactions, especially in the presence of reactive aggregates. The presence of Al_2O_3 contributes to the formation of calcium-alumino-silicate hydrates (C-A-S-H) and improves thermal and mechanical resistance. This makes PSA valuable in ceramic applications, particularly when blended with high-silica or high-alumina materials. A low LOI improves compatibility with cementitious systems and minimises potential negative impacts on hydration or mechanical strength.

3.1 Microstructural Analysis

Figure 1 below provided SEM micrograph of Ugwuoba clay. The micrographs reveals a heterogeneous mixture of particles with irregular, angular, and sub-rounded morphologies. The presence of such shapes suggests a limited degree of post-depositional weathering or mechanical grinding, preserving the natural mineral structure (Wang et al., 2024). The angular grains indicate the potential for better mechanical interlocking in composites, which can enhance load-bearing capabilities and flexural strength (Wang et al., 2024). Some dark regions in the micrograph denote micro-pores or inter-particle voids. These are critical for thermal insulation applications but can reduce mechanical integrity if excessive (Makrygiannis, et al., 2025). Controlling porosity through additives like agro-waste ashes can tailor the properties of Ugwuoba clay-based products.

Table 1: Elemental Composition a. Ugwoba clay composition b. PSA composition.

Ugwoba clay composition	
oxide name	Composition (%)
SiO2	64.87
CaO	0.22
Al2O3	20.56
Fe2O3	4.75
MgO	0.75
Na2O	0.15
K2O	1.64
SO3	0.04
LOI	5.99

Palm stalk ash Composition	
oxide name	Composition (%)
SiO2	51.84
CaO	11.13
Al2O3	5.62
Fe2O3	2.32
MgO	2.24
Na2O	2.22
K2O	21.65
SO3	-
LOI	3.18

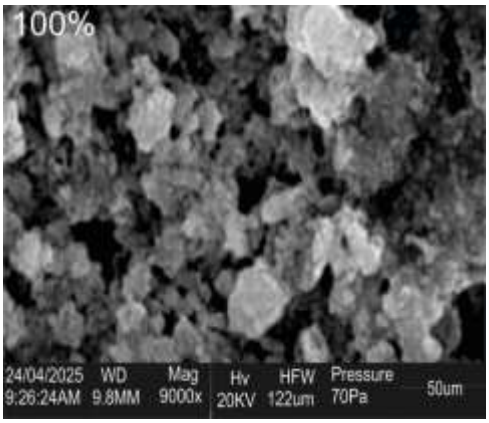

Figure 1: SEM of Ugwuoba Clay

Figure 2 shows the micrograph of 30% wt.PSA reinforced Ugwuoba clay. In terms of microstructural characteristics, the image reveals a collection of particles with irregular, angular shapes. These particles vary in size. The variation in particle size indicates a broad particle size distribution, which is typical of natural or processed ashes like palm shell ash. The particles are closely packed together, with relatively few visible gaps, suggesting a densely compacted morphology. However, some dark regions in the image hint at the presence of interparticle voids or pores. The surfaces of the particles appear rough and textured, which could enhance interfacial bonding in a composite system by promoting mechanical interlocking with a surrounding matrix. Overall, the micrograph of the 30% wt.PSA reinforced Ugwuoba clay suggests a microstructure composed of angular and irregularly shaped filler particles with good packing density and surface roughness. These features can influence the mechanical, thermal, and interfacial properties of composites, particularly in applications where PSA is used to reinforce polymeric or ceramic matrices (Makrygiannis et al., 2025).

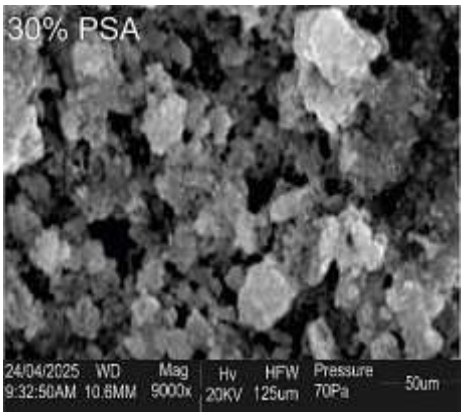

Figure 2: SEM of 30wt.% PSA-Ugwoba Clay Composite

3.2 Flexural Strength

Figure 3 shows the flexural strength (MPa) of Ugwuoba clay reinforced with varying percentages of PSA, ranging from 0 to 40 wt.%. The trend clearly demonstrates a progressive increase in flexural strength as PSA content increases. Specifically, the flexural strength of the unreinforced (pure) clay sample begins at approximately 32 MPa. As PSA is added incrementally—10%, 20%, 30%, and finally 40%—the flexural strength correspondingly rises to about 34 MPa, 36.5 MPa, 39 MPa, and ultimately reaches approximately 41.5 MPa at 40% PSA. This steady enhancement in flexural strength might be attributed to several beneficial effects of PSA on the clay matrix. Palm stalk ash, being rich in oxides such as silica (SiO_2), alumina (Al_2O_3), and potassium oxide (K_2O), contributes to pozzolanic reactions during the sintering process, which enhance the bonding between clay particles. These oxides can also lead to the formation of secondary mineral phases such as potassium alumino-silicates, which serve to reinforce the microstructure of the composite (Huang et al., 2024). Additionally, the presence of PSA occupies voids within the clay matrices, leading to improved packing density and a reduction in internal stress concentrations. This densification and microstructural refinement are evident in similar studies where PSA-containing ceramics showed fewer micro-cracks and enhanced load-bearing capability (Makrygiannis et al., 2025). Therefore, the progressive increase in flexural strength can be directly linked to the synergistic effect of PSA's pozzolanic activity, mineral phase contribution, and pore-filling ability.

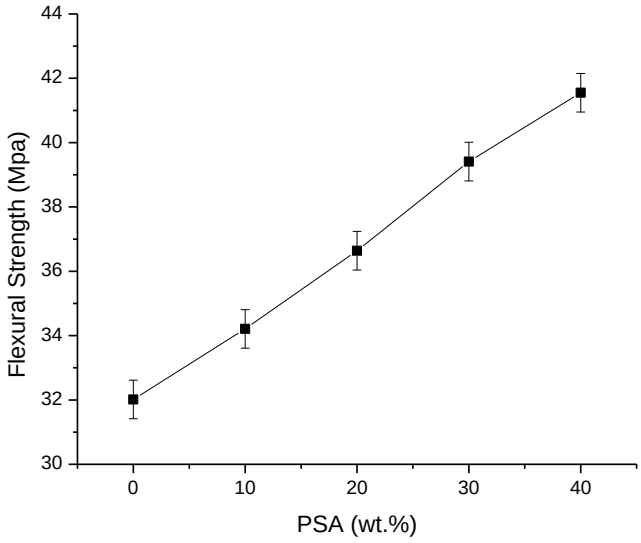

Figure 3: Flexural Strength of Ugwuoba clay reinforced with PSA

3.3 Thermal shock resistance of Ugwuoba clay reinforced with PSA

Figure 4 presents the deteriorating thermal shock resistance of Ugwuoba clay as palm stalk ash (PSA) content increases from 0 to 40 wt.%. Thermal shock resistance, a critical property for refractory applications, measures a material's ability to withstand repeated heating and cooling cycles without cracking. At 0% PSA, pure Ugwuoba clay exhibits a relatively high thermal shock resistance of about 29 cycles. This high performance indicates a stable and cohesive microstructure that resists damage under thermal cycling. However, as PSA is gradually introduced, thermal shock resistance declines steadily. At 10 wt.% PSA, it reduces slightly to 28 cycles. By 20 wt.%, the material tolerates only around 26 cycles, and this decline becomes more pronounced beyond 30 wt.%, where the resistance drops to about 22 cycles. At 40 wt.% PSA, thermal shock resistance is significantly reduced to around 20 cycles. This trend suggests that while Ugwuoba clay initially benefits from minor PSA inclusion in terms of other properties, its ability to resist thermal stress is compromised at higher PSA levels. The underlying cause of this reduction lies in the microstructural effects induced by PSA. Palm stalk ash contains high levels of alkali oxides and organic matter, which can volatilize during sintering, creating pores and micro-cracks that act as stress concentration points under thermal cycling (Hidayat et al., 2025). Furthermore, the mismatch in thermal expansion coefficients between the clay and ash particles causes internal stresses during heating and cooling, which weakens the material's structural integrity (Gikunju et al., 2024). Also, as PSA content increases, it interferes with the sintering behaviour of the clay, resulting in reduced vitrification and a less cohesive matrix. The lack of sufficient bonding between particles makes the material more susceptible to spalling and crack propagation when exposed to rapid temperature changes (Makrygiannis et al., 2025).

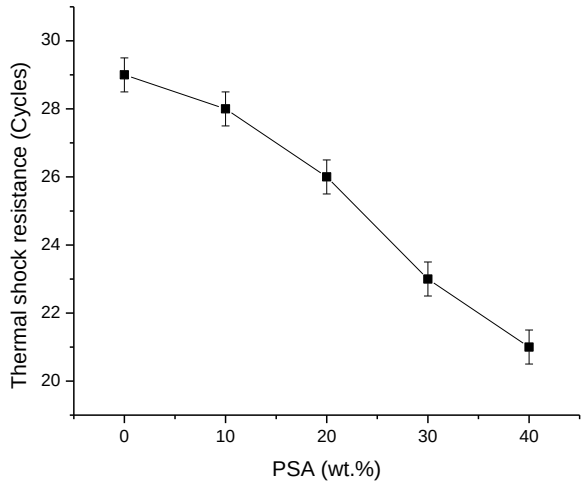

Figure 4: Thermal Shock Resistance of Ugwuoba Clay reinforced with PSA

3.4 Refractoriness

Figure 5 depicts the influence of PSA on the refractoriness of Ugwuoba clay. Refractoriness, which refers to a material's ability to withstand high temperatures without softening or deforming, is a key property for materials used in high-temperature environments such as furnaces, kilns, and refractory linings. From the graph, it is evident that pure Ugwuoba clay (0% PSA) exhibits the highest refractoriness, maintaining thermal stability up to approximately 1600°C. This high thermal endurance is characteristic of clays rich in alumina and silica, which are known to form strong refractory phases (Siddika et al., 2021). However, as PSA is incrementally added to the clay in amounts of 10%, 20%, 30%, and 40% by weight, the refractoriness decreases steadily. At 10% PSA, the refractoriness drops to around 1550°C, further declining to 1490°C at 20%, 1390°C at 30%, and finally reaching 1350°C at 40% PSA. This progressive decline in refractoriness can be attributed to the chemical characteristics of PSA. PSA is composed of substantial quantities of alkali oxides such as K_2O, CaO, and MgO, which act as fluxing agents. These oxides lower the melting point of the ceramic matrix by forming low-melting eutectic compounds, thereby reducing the material's resistance to heat (Siddika et al., 2021). Additionally, the PSA reduces the relative proportions of alumina and silica

in the composite, thus diminishing its inherent refractory nature (Ihuoma et al., 2022). Moreover, PSA often contains organic residues that decompose during firing, producing gases that result in increased porosity and microstructural defects. These defects create weak points within the material, accelerating failure under high-temperature conditions (Chompoorat et al., 2022). As a result, the structure becomes less resistant to softening and thermal deformation.

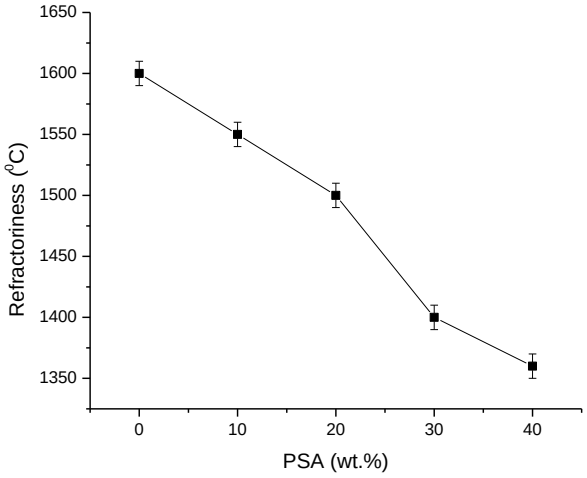

Figure 5: Refractoriness of of Ugwuoba Clay reinforced with PSA

3.5 Physico-mechanical properties

To evaluate the physico-mechanical properties of reinforced Ugwuoba clay, 30wt.% PSA reinforced Ugwuoba clay was selected and its properties compared with that of the pure ugwoba clay across varying sintering temperatures (900°C–1200°C) as shown in Table 2a and 2b below. The comparative analysis reveals significant differences in thermal, structural, and mechanical behaviours. These changes are a direct result of the introduction of PSA, which acts as a fluxing agent due to its rich content in alkali and alkaline earth oxides (e.g., K_2O, CaO, MgO). The linear shrinkage of pure Ugwuoba clay is consistently higher at all firing temperatures compared to the 70:30 clay-PSA blend. For example, at 1200°C, pure clay exhibits a shrinkage of 6.10%, while the 70:30 sample shows only 4.38%. The reduced shrinkage in the PSA-reinforced clay indicates dimensional stability, likely due to the fibrous and silica-rich structure of PSA, which inhibits particle rearrangement and excessive densification during sintering (Onah et al., 2022).

Both materials show a decrease in porosity and water absorption with increasing temperature, reflecting densification. However, pure clay retains slightly higher porosity and water absorption across the temperatures. For instance, at 1200°C, pure clay has a porosity of 21.86% and water absorption of 12.27%, while the 70:30 sample records 8.63% and 4.51%, respectively. This improvement in the 70:30 composite is due to PSA acting as a pore filler, reducing interstitial voids and enhancing compaction (Onah et al., 2022). Densities increase for both materials with temperature. However, the bulk density of the 70:30 PSA sample surpasses that of pure clay at higher temperatures (e.g., 1.91 g/cm³ vs. 1.78 g/cm³ at 1200°C). This reflects a more compact microstructure, which enhances strength and reduces permeability, attributed to the glass-forming oxides in PSA that promote vitrification (Ihuoma et al., 2021).

The modulus of rupture (MOR) of the PSA-reinforced sample is markedly superior. At 1200°C, the 70:30 sample reaches 39.41 MPa compared to 32.02 MPa in pure clay. The ash likely enhances interparticle bonding and contributes to phase formations like mullite, improving the sample's resistance to bending stress (Almeida et al., 2022). The inclusion of 30% PSA in Ugwuoba clay improves mechanical strength, densification, and structural stability, while slightly compromising high-temperature performance.

Table 2a: Physico-mechanical Properties of Ugwuoba Clay

100% Ugwuoba clay					
Temp(^oC)	Linear shrinkage	Modulus of Rupture(MPa)	Apparent porosity (%)	Water absorption (%)	Bulk density (g/cm^3)
900	3.240222	20.91458	25.75617	14.78272	1.742438
1000	4.540546	29.14691	23.82606	13.52836	1.761739
1100	4.994341	31.78672	22.49943	12.67573	1.775006
1200	6.100218	32.01597	21.86308	12.2737	1.781369

Table 2b: Physico-mechanical Properties of 70 % Clay 30% PSA Sample.

70 % Clay 30% PSA Sample					
Temp (^oC)	Linear shrinkage (%)	Modulus of Rupture (MPa)	Apparent porosity (%)	Water absorption (%)	Bulk density (g/cm^3)
900	1.804859	23.37924	19.05574	10.5313	1.809443
1000	2.914798	33.00863	14.39876	7.758836	1.856012
1100	3.037251	38.31485	11.04359	5.844607	1.889564
1200	4.376984	39.40741	8.627314	4.508914	1.913727

4.0 Conclusion

This study concludes that reinforcing Ugwuoba clay with PSA significantly influences its mechanical performance, particularly its flexural strength, with 40 wt.% PSA content giving the best flexural strength. The improvement can be attributed to the filler effect and the fluxing oxides present in PSA, which promote densification and strength development during sintering. Microstructural properties was also influenced effectively by its addition. Finally, physico-mechanical properties were significantly influenced with the addition of PSA and at increasing sintering temperature. However, the inclusion of PSA results in a notable decline in thermal properties such as refractoriness and thermal shock resistance, due to the formation of low-melting phases introduced by alkali and alkaline earth oxides. Therefore, while PSA serves as a viable and sustainable reinforcement for clay-based ceramics, its application must be tailored to the specific thermal and mechanical requirements of the intended use. This research underscores the potential of agricultural waste materials like PSA for sustainable, low-cost, and eco-friendly development of refractory and structural ceramic products.

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