UJEAS

UNIZIK JOURNAL OF ENGINEERING
AND
APPLIED SCIENCES

UNIZIK Journal of Engineering and Applied Sciences 6(1), March (2026), 3331-3341
Journal homepage: https://journals.unizik.edu.ng/index.php/ujeas
PRINT ISSN: 2992-4383 || ONLINE ISSN: 2992-4391

Effect of Palm Stalk Ash Reinforcement on the Microstructural, Thermal and Mechanical Properties of Ugwuoba Clay for Refractory Applications

Kenechukwu ThankGod Onyena*, Oluwaseyi Omotayo Taiwo, SamuelMary Onwubiko Obiorah, Nkem Emelike Nwankwo
Department of Metallurgical and materials engineering, faculty of engineering in Nnamdi Azikiwe University Awka
*Corresponding Author's E-mail: Kenechukwuthankgod0@gmail.com

Abstract
The growing demand for sustainable refractory materials has stimulated interest in the utilization of agro-waste additives for enhancing the performance of clay-based ceramics. In this study, Ugwuoba clay reinforced with 20.66 wt.% Palm Stalk Ash (PSA) was investigated to evaluate its suitability for foundry crucible applications. The clay–PSA composite was prepared, shaped, and fired at temperatures ranging from 1100 °C to 1200 °C, and its microstructural, mineralogical, physical, thermal, and mechanical properties were scientifically examined. Microstructural analysis revealed that PSA incorporation modified the clay matrix by introducing a heterogeneous but well-bonded structure, while X-ray diffraction confirmed enhanced formation of thermally stable calcium silicate and silica-rich phases. Apparent porosity and water absorption decreased with increasing firing temperature, accompanied by a corresponding increase in bulk density, indicating improved densification. Linear shrinkage results demonstrated accelerated sintering attributable to the fluxing action of PSA. Thermal conductivity exhibited temperature-dependent behaviour governed by porosity reduction and microstructural consolidation. Thermogravimetric analysis showed minimal mass loss beyond the dehydration and organic decomposition stages, confirming good thermal stability at elevated temperatures. Cold crushing strength increased significantly with firing temperature, reflecting enhanced mechanical integrity and load-bearing capacity. Overall, the results demonstrate that reinforcement of Ugwuoba clay with 20.66 wt.% PSA improves thermal stability, mechanical strength, and densification characteristics, making the composite a promising, sustainable candidate for foundry crucible and other high-temperature refractory applications.

Keywords: Ugwuoba clay, ceramic, foundry, crucible, palm stalk ash

1. Introduction

Refractory materials are specialized ceramics designed to withstand extremely high temperatures, mechanical stresses, and chemically aggressive environments, making them indispensable in metallurgical, glass, and ceramic industries where materials must maintain structural stability under sustained thermal exposure (Egole et al., 2024). Among the various refractory raw materials, aluminosilicate clays are widely utilized due to their natural abundance, cost-effectiveness, good thermal stability, and ease of processing. Nevertheless, naturally occurring clays often possess inherent limitations such as high porosity, brittleness, and relatively low mechanical strength, particularly under repeated thermal cycling, which limits their direct application in demanding high-temperature environments such as furnace linings, crucibles, and kiln furniture where both structural durability and thermal shock resistance are essential. To overcome these shortcomings, several studies have explored the modification of clay matrices through the incorporation of reinforcing additives. Conventional additives such as alumina, graphite, and magnesium oxide have been reported to significantly enhance the mechanical strength, fracture toughness, and thermal stability of clay-based composites used in refractory systems (Nwankwo et al., 2025).

More recently, the growing emphasis on environmental sustainability has driven increased research interest in the use of agricultural waste ashes as functional additives in clay-based materials. Agro-waste ashes such as rice husk ash, palm oil fuel ash, and millet waste are rich in silica and other oxides capable of acting as pozzolanic or fluxing agents, thereby promoting densification, improving thermal insulation, and enhancing the mechanical performance of clay composites while simultaneously enabling the valorization of agricultural residues (Salifou et al., 2024). For instance, millet waste incorporation in earthen block composites has been shown to reduce thermal conductivity and enhance thermal resistance, while rice husk ash has demonstrated effectiveness in improving strength development and durability in clay and cementitious systems due to its high amorphous silica content (Salifou et al., 2024). Similarly, agricultural ashes have been applied in the production of insulating refractory bricks, where their addition to clay matrices has been reported to improve key refractory properties such as density, porosity, and shrinkage behaviour (Joshua et al., 2025). Among these agro-residues, Palm Stalk Ash (PSA), derived from palm agricultural waste, has emerged as a promising additive due to its substantial silica content and the presence of fluxing oxides such as K_2O, CaO, and MgO that can promote sintering reactions and the formation of stable ceramic phases at elevated temperatures.

Previous studies have demonstrated that PSA incorporation can enhance densification, reduce water absorption, and improve compressive strength in ceramic and geopolymer systems (Huseien and Mhaya, 2026). However, despite these promising findings, limited research has specifically examined the reinforcement of indigenous clay deposits such as Ugwuoba clay with PSA for refractory applications, particularly in relation to its microstructural evolution, thermal behaviour, and mechanical performance at high firing temperatures. This represents a critical gap in the literature considering the abundance of both Ugwuoba clay and palm agricultural residues in Nigeria. Therefore, this study investigates the effect of 20.66 wt.% Palm Stalk Ash reinforcement on the microstructural, thermal, and mechanical properties of Ugwuoba clay fired at elevated temperatures, with the innovative objective of developing a sustainable clay–agro-waste composite suitable for high-temperature refractory applications such as furnace crucibles while simultaneously promoting the valorization of palm agricultural waste.

2.0 Materials and methods

2.1 Raw Materials

The primary raw material used in this study was Ugwuoba clay, sourced from Ugwuoba community in Enugu State, Nigeria. The clay deposit is known for its aluminosilicate composition and local availability, making it a potential candidate for refractory applications. The sample preparation and mechanical characterizations where carried out at the Project Development Institute (PRODA), Enugu. The microstructural analysis (XRD and SEM) was carried out at Rolab Research Laboratory, Ibadan while the TGA was carried out at the Bioprocess Lab Unizik, Awka. The as-received clay was air-dried to remove surface moisture, manually crushed using a jaw crusher, and further pulverised using a ball mill. The pulverised clay was then sieved through a 75 μm mesh to obtain a uniform particle size suitable for ceramic processing. Palm Stalk Ash (PSA), used as the reinforcing material, was obtained from dried palm stalk residues collected from a local palm processing site. The stalks were washed to remove dirt and impurities, sun-dried, and combusted in a muffle furnace under controlled conditions at approximately 700 °C to ensure complete combustion of organic matter. The resulting ash was allowed to cool, ground to fine powder, and sieved through a 75 μm mesh to ensure homogeneity and compatibility with the clay matrix.

2.2 Composite Formulation and Sample Preparation

The clay–PSA composite was prepared using an optimised reinforcement content of 20.66 wt.% PSA, determined from preliminary optimisation studies. The required proportions of Ugwuoba clay and PSA were weighed using a digital balance and dry-mixed thoroughly to achieve uniform distribution of PSA particles within the clay matrix. Distilled water was gradually added to the mixture to form a workable paste, which was further homogenised to ensure adequate plasticity and particle bonding. The prepared paste was moulded into standard test specimens using steel moulds under uniaxial compaction to minimise entrapped air and improve green density. The green samples were air-dried for 24 hours and subsequently oven-dried at 110 °C for another 24 hours to remove residual moisture. Dried specimens were fired in an electric furnace at temperatures ranging from 1100 to 1200 °C, with a controlled heating rate and soaking time to allow adequate sintering and phase development. After firing, the samples were furnace-cooled to room temperature prior to characterisation.

2.3 Characterisation Techniques

2.3.1 Microstructural Examination

Fired samples of unreinforced Ugwuoba clay and Ugwuoba clay reinforced with 20.66 wt.% PSA were sectioned using a diamond cutting wheel. The sections were mounted in resin and progressively polished using silicon carbide abrasive papers of increasing grit sizes, followed by fine polishing to obtain smooth, scratch-free surfaces. The prepared surfaces were examined using an optical microscope at suitable magnifications. Micrographs were captured to reveal particle distribution, pore morphology, and the dispersion of PSA within the clay matrix.

2.3.2 X-Ray Diffraction (XRD) Analysis

X-ray diffraction analysis was performed on powdered samples of both unreinforced and PSA-reinforced Ugwuoba clay. The fired specimens were crushed into fine powders using an agate mortar and pestle. The powders were placed on a sample holder and analysed using an X-ray diffractometer equipped with Cu–Kα radiation. Diffraction data were collected over a 2θ range sufficient to identify major crystalline phases. Phase identification was conducted by matching the obtained diffraction peaks with standard reference patterns.

2.3.3 Apparent Porosity, Water Absorption, and Bulk Density

Apparent porosity, water absorption, and bulk density were determined using the Archimedes immersion method. Fired samples were first dried and weighed to obtain the dry weight. The specimens were then immersed in boiling water for a specified duration, cooled, and weighed again to obtain the saturated weight. Subsequently, the samples were weighed while suspended in water to determine the buoyant weight. Apparent porosity, water absorption, and bulk density were calculated using standard ceramic equations based on these measured weights.

2.3.4 Linear Shrinkage Measurement

Linear shrinkage was measured by determining the dimensional change of samples before and after firing. The initial length of each green specimen was measured using a digital vernier caliper prior to firing. After firing and cooling to room temperature, the final length was measured. Linear shrinkage was calculated as the percentage reduction in length relative to the original dimension.

2.3.5 Thermal Conductivity Measurement

Thermal conductivity measurements were carried out on fired samples using a thermal conductivity testing apparatus. Specimens were prepared to standard dimensions to ensure uniform heat flow during testing. Measurements were taken at different temperatures, and thermal conductivity values were recorded based on steady-state heat transfer through the sample. The test setup ensured consistent contact between the sample and heat source to minimise experimental error.

2.3.6 Cold Crushing Strength (CCS)

Cold crushing strength tests were conducted using a universal testing machine. Fired specimens were machined to standard dimensions and placed centrally between the compression platens of the testing machine. A uniaxial compressive load was applied at a constant loading rate until sample failure occurred. The maximum load at failure was recorded, and the cold crushing strength was calculated by dividing the failure load by the cross-sectional area of the specimen.

2.3.7 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis was performed on powdered PSA-reinforced Ugwuoba clay samples using a thermogravimetric analyser. A small, known mass of the sample was placed in a crucible and heated from room temperature to the desired upper temperature limit at a controlled heating rate under a specified atmosphere. The change in sample mass was continuously recorded as a function of temperature to obtain the thermogravimetric curve.

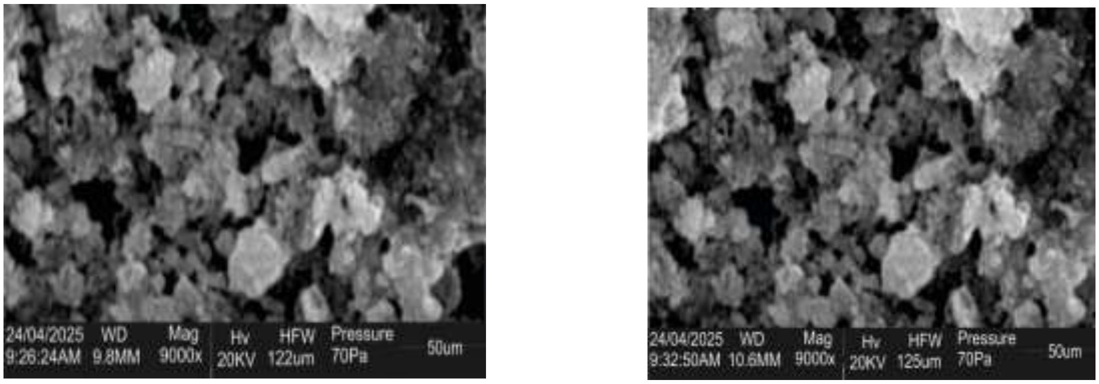
3.0 Result and Discussion

3.1 Microstructural Evolution of Ugwuoba Clay Reinforced with 20.66 wt.% Palm Stalk Ash

The microstructural characteristics of fired unreinforced Ugwuoba clay and Ugwuoba clay reinforced with 20.66 wt.% palm stalk ash (PSA) reveal notable differences in particle arrangement, pore morphology, and matrix heterogeneity. The unreinforced clay exhibits a relatively compact and homogeneous microstructure, characterised by closely packed clay particles with limited visible pore spaces. Such dense microstructural features are typical of aluminosilicate clays subjected to firing, where particle rearrangement and solid-state sintering processes promote grain coalescence and pore elimination as the system seeks to minimise surface energy (Sánchez-Soto et al., 2021). This compact morphology suggests restricted pore connectivity and reduced internal void volume, which is consistent with observations reported for sintered kaolinite-rich clay ceramics fired at elevated temperatures (Freitas-Dutra et al., 2019). Dense particle packing in clay matrices has been widely associated with improved intergranular bonding and reduced permeability, as the collapse or closure of capillary pores occurs during thermal treatment (Utkarsh and Jain, 2024).

In contrast, the PSA-reinforced Ugwuoba clay composite displays a more heterogeneous microstructure, marked by the presence of discrete PSA particles embedded within the clay matrix. The ash particles are distinguishable from the surrounding clay phase, indicating effective physical incorporation of PSA into the matrix during processing. However, their presence disrupts the continuity of the clay network, resulting in the formation of interfacial regions and an increased number of visible pores. Similar microstructural heterogeneity has been reported in clay-based composites reinforced with agro-waste ashes, where the introduction of non-plastic or partially reactive phases modifies particle packing efficiency and sintering behaviour (Salifou et al., 2024). The observed increase in porosity in the PSA-reinforced sample may be attributed to differences in particle size, morphology, and thermal reactivity between the clay minerals and the ash particles. Agro-waste ashes, particularly those rich in silica, often exhibit lower sintering activity at conventional firing temperatures, thereby acting as pore-forming agents within ceramic matrices (Joshua et al., 2025). Comparable microstructural features have been documented in clay composites containing rice husk ash, where ash inclusions generate microvoids and alter pore size distribution due to differential shrinkage and incomplete vitrification (Alhaji et al., 2020 et al., 2022).

Furthermore, the chemical composition of PSA, which typically includes fluxing oxides such as K_2O, CaO, and MgO, can influence localised sintering phenomena. These oxides may promote the formation of transient liquid phases at particle interfaces, facilitating partial densification around ash inclusions while preserving overall microstructural heterogeneity (Huseien and Mhaya, 2026). This combination of localised densification and global porosity has been identified as a characteristic feature of agro-waste-reinforced ceramic composites.



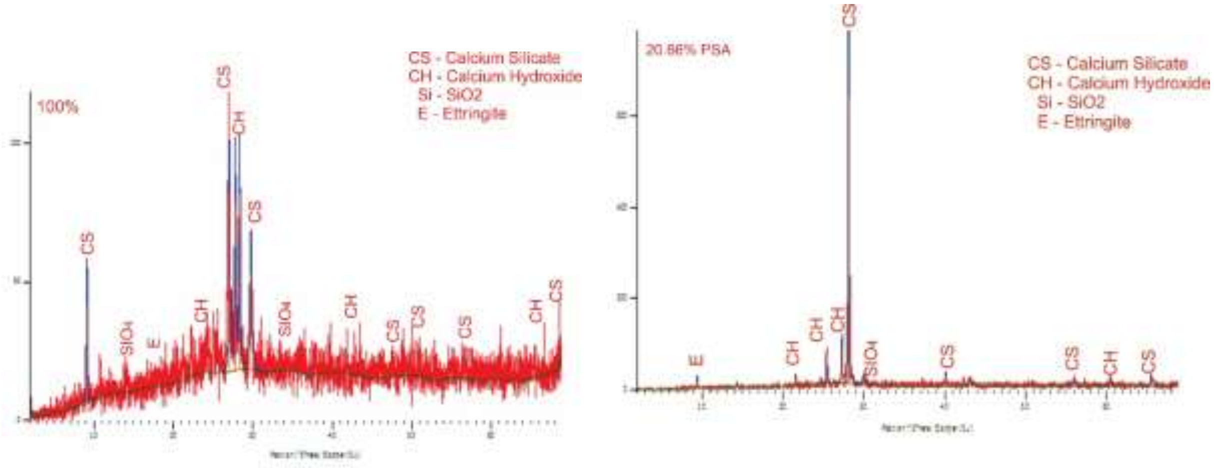
a b.

Figure 1: Microstructure of a. Unreinforced Ugwuoba Clay. b. 20.66wt,% PSA reinforced Ugwuoba Clay.

3.2 Mineralogical Composition of Ugwuoba Clay Reinforced with 20.66 wt.% Palm Stalk Ash.

The X-ray diffraction (XRD) patterns of unreinforced Ugwuoba clay and the composite reinforced with 20.66 wt.% palm stalk ash (PSA) were analysed to determine differences in crystalline phase composition induced by PSA incorporation and firing. As seen in Figure 2a, the pattern of the XRD of the unreinforced clay revealed dominant reflections associated with common clay minerals and silica-rich phases, indicating an aluminosilicate matrix typical of natural clays. Quartz and kaolinitic or other phyllosilicate structures have been widely reported as principal crystalline phases in clay systems, and such features were evident in the Ugwuoba clay, suggesting a composition dominated by stable crystalline silicates (Salifou et al., 2024). Upon reinforcement with PSA (as observed in Figure 2b) and subsequent firing, notable changes in peak intensities and the appearance of additional diffraction features were observed. Also, the peaks shows a more crystalline feature with the intensity of peaks corresponding to calcium silicate and related silicate structures increased in the PSA-reinforced composite. This behaviour is consistent with reports that the addition of calcium-rich ashes or fluxing oxides to clay systems promotes the formation of thermally stable calcium silicate phases during high-temperature treatment (Sdira et al., 2024). The higher intensities of these silicate peaks suggest that PSA, which contains silica and fluxing oxides such as CaO and K_2O, contributed to enhanced phase development and crystallisation of refractory silicate compounds.

XRD studies on agro-waste-reinforced clay composites have also documented similar trends where the introduction of reactive ash phases leads to the emergence of new crystalline peaks or the growth of existing ones. For example, research on clay bricks incorporating palm kernel shell powder revealed attenuation of primary clay peaks and the development or persistence of quartz reflections, indicating changes in crystallinity without the introduction of unrelated phases (Smith et al., 2025). Although no entirely foreign phases were detected in the PSA-reinforced Ugwuoba clay beyond expected silicates, the modified diffraction pattern clearly indicates phase evolution driven by the interaction between clay minerals and PSA constituents. Furthermore, the reduced intensity or slight broadening of some original clay reflections in the PSA composite implies partial structural disruption or transformation of primary clay minerals during firing. This finding is in line with observations in other agro-ash modified clay systems where increasing ash content diminishes the crystallinity of phyllosilicate peaks due to lattice distortion or the formation of amorphous silicate regions (Salifou et al., 2024). These changes in the XRD patterns support the interpretation that PSA facilitates both the retention of major silicate phases and the formation of additional thermally stable phases that contribute to enhanced material performance.



a. b.

Figure 2: XRD of a. Unreinforced Ugwuoba Clay. b. 20.66wt,% PSA reinforced Ugwuoba Clay

3.3 Effect of Apparent Porosity, Water Absorption, and Bulk Density of Ugwuoba Clay Reinforced with 20.66 wt.% PSA.

The effects of firing temperature on apparent porosity, water absorption, and bulk density in Figures 3, 4, and 5 provide crucial insight into the densification behaviour of clay-based composites. Apparent porosity quantifies the percentage of open pores in a fired ceramic body and is inversely related to densification; a decrease in porosity generally corresponds to particle rearrangement, viscous flow, and enhanced sintering at elevated temperatures (Maury-Njoya et al., 2024). In the current study, the apparent porosity of Ugwuoba clay reinforced with 20.66 wt.% PSA as observed in Figure 3 decreased progressively as firing temperature increased, indicating enhanced densification and pore closure at higher temperatures. This trend is consistent with findings in similar clay composites, where increased firing temperatures promote particle fusion and reduce open porosity, leading to a denser microstructure (Maury-Njoya et al., 2024). High porosity values at lower firing temperatures reflect incomplete sintering and limited viscous phase development, while reduced porosity at higher temperatures indicates improved particle bonding and vitrification (Maury-Njoya et al., 2024).

Water absorption measurements further corroborate the porosity trends observed in the composite samples. Water absorption is a direct indication of accessible pore volume and pore connectivity within a ceramic material; higher water absorption values are typically observed in highly porous materials due to greater availability of pathways for water ingress (Obada et al., 2016). In this study, water absorption decreased markedly with increasing firing temperature as presented in Figure 4, aligning with the observed reduction in apparent porosity. This inverse relationship between water absorption and firing temperature has been widely reported in the literature, as elevated temperatures facilitate densification and pore closure, thereby reducing the ability of the material to absorb water (Obada et al., 2016; Bomeni et al., 2025). Similar behaviour has been documented in brick and refractory ceramics, where increasing firing temperature decreases both open porosity and water uptake due to progressive vitrification and the formation of a more consolidated microstructure (Bomeni et al., 2025).

Bulk density, defined as the mass per unit volume of a fired specimen, is expected to increase as porosity decreases because pore elimination leads to a greater proportion of solid material within the bulk volume. In the present study, the bulk density of the PSA-reinforced Ugwuoba clay increased with firing temperature (Figure 5), indicating successful densification and pore collapse during heat treatment. This observation is in agreement with other research on porous ceramic systems, where an increase in firing temperature results in bulk density gains due to decreased pore volume and enhanced matrix bonding (Salifou et al., 2024). The inverse relationship between bulk density and apparent porosity is a well-established phenomenon in ceramic materials; as porosity decreases due to sintering, bulk density increases because of reduced pore space and improved particle packing (Salifou et al., 2024). The combined trends of decreasing apparent porosity and water absorption with increasing bulk density reflect the typical sintering behaviour of clay composites and confirm that PSA addition, coupled with elevated firing temperatures, promotes densification. These changes in physical properties are critical for the development of ceramic materials intended for refractory applications, where low porosity and high density are associated with enhanced mechanical strength and thermal stability.

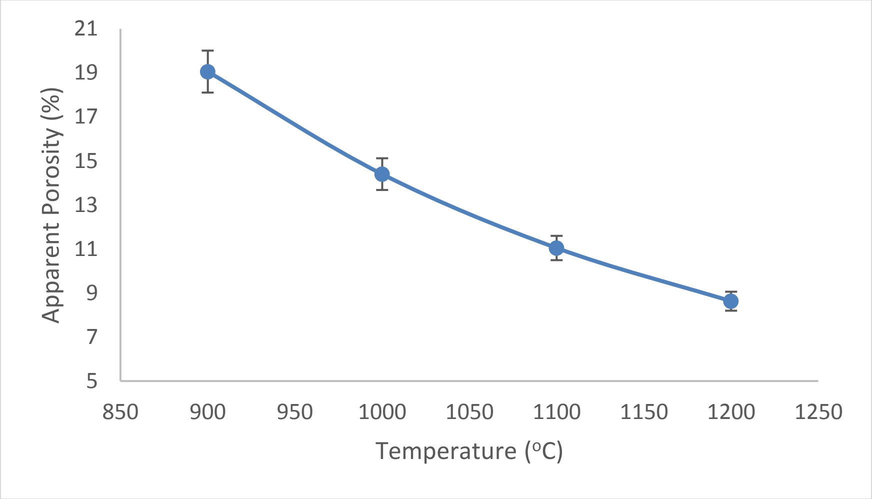


Figure 3. Apparent Porosity of Uguwoba Clay reinforced with 20.66wt.% PSA

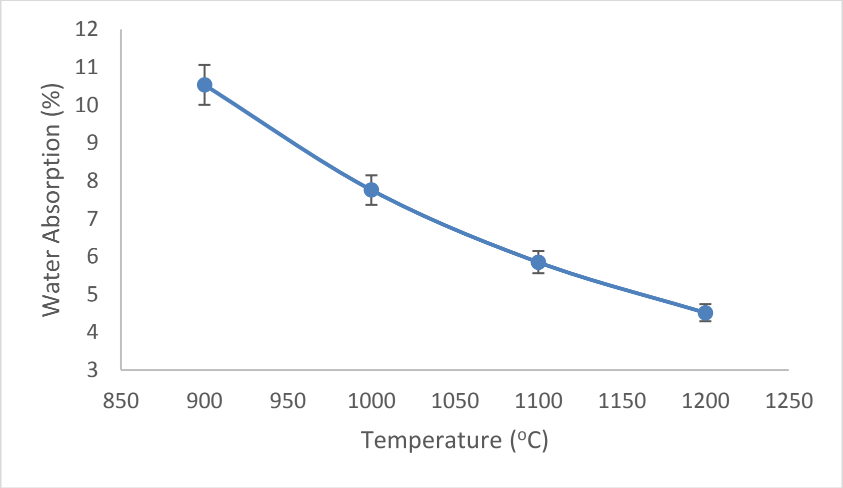


Figure 4. Water Absorption properties of Uguwoba Clay reinforced with 20.66wt.% PSA

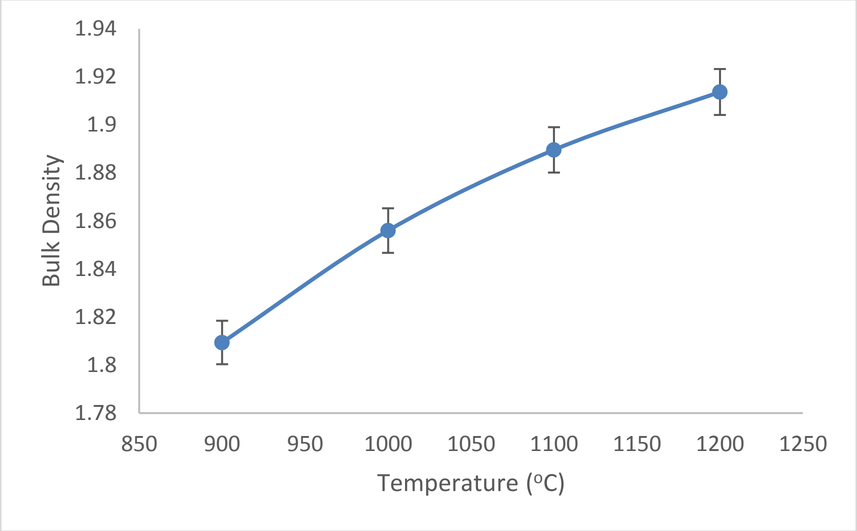


Figure 5. Bulk density properties of Uguwoba clay reinforced with 20.66wt.% PSA

3.4 Linear Shrinkage Behaviour of Ugwuoba Clay Reinforced with 20.66 wt.% PSA

The linear shrinkage of ceramic composites is a direct indicator of sintering behaviour, dimensional stability, and densification during firing. In this study, linear shrinkage of Ugwuoba clay reinforced with 20.66 wt.% PSA increased progressively with increasing firing temperature in Figure 6. This phenomenon is generally associated with enhanced particle rearrangement and viscous flow at elevated temperatures, resulting in a reduction of open pores and contraction of the bulk specimen (Sánchez-Soto et al., 2021). Similar trends have been observed in kaolin clay systems, where increasing firing temperatures enhance densification and closure of microstructural voids, leading to higher shrinkage values (Sánchez-Soto et al., 2021). Earlier stages of firing (below ~1000 °C) typically exhibit limited shrinkage due to incomplete sintering and insufficient liquid phase formation, whereas higher temperatures promote greater viscous flow and tighter particle packing (Sánchez-Soto et al., 2021). In the PSA-reinforced composite, the presence of fluxing oxides from the ash phase likely contributes to the acceleration of sintering, resulting in increased shrinkage compared to unreinforced clay. This behaviour aligns with experimental findings in clay-ash mixtures, where ash additives lower the viscous sintering temperature and influence the onset and rate of shrinkage during firing (Sánchez-Soto et al., 2021). Therefore, the linear shrinkage profile of the PSA-reinforced Ugwuoba clay is reflective of a composite transitioning from solid-state sintering to enhanced densification with increasing thermal input, with implications for dimensional control during processing.

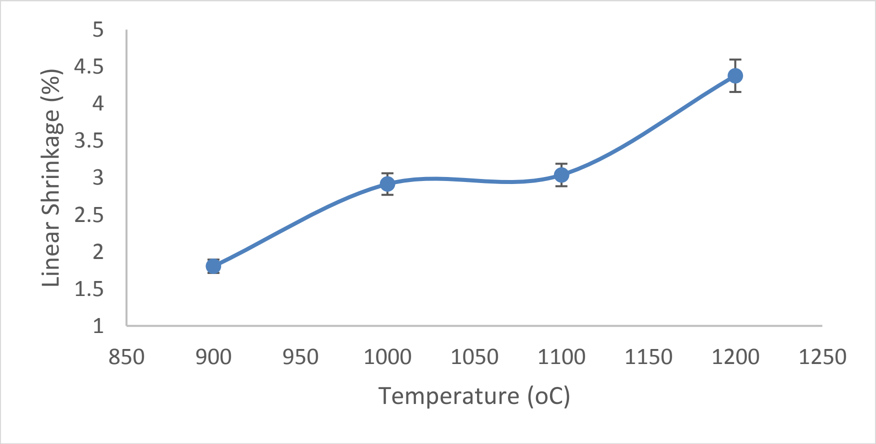


Figure 6. Linear Shrinkage of Uguwoba Clay reinforced with 20.66wt.% PSA

3.5 Thermal Conductivity of Ugwuoba Clay Reinforced with 20.66 wt.% PSA

Thermal conductivity measurements provide insight into how heat is transferred through a material and are strongly influenced by porosity, bulk density, and microstructure. In ceramic bodies, thermal conductivity generally increases with decreasing porosity and increasing density, as the continuity of the solid matrix enhances phonon transport (Sánchez-Soto et al., 2021). The addition of PSA to Ugwuoba clay altered the thermal conductivity behaviour of the composite, as reflected in the measured values across the firing temperature range. In fired clay composites, increased porosity usually corresponds to lower thermal conductivity because entrapped air in pores acts as an insulating medium, reducing heat transfer (Makrygiannis et al., 2025). Studies on clay materials reinforced with various agro-waste and industrial residues confirm that enhanced porosity from such additions often leads to a reduction in thermal conductivity, beneficial in applications requiring thermal insulation (Makrygiannis et al., 2025; Science Publishing Group, 2025). However, where ash additives contribute to partial densification or transport pathways for heat, thermal conductivity can increase at elevated temperatures as the solid network becomes more interconnected (Scientific Reports, 2023; Sánchez-Soto et al., 2021).

For the PSA-reinforced Ugwuoba clay, the observed thermal conductivity trend with temperature as presented in Figure 7 likely reflects the interplay between residual porosity introduced by PSA and the densification effects at high firing temperatures. At lower firing stages with higher open porosity, lower conductivity is expected due to increased thermal resistance from air-filled pores. As firing temperature increases, microstructure consolidation facilitates better heat transfer, thereby increasing thermal conductivity values (Scientific Reports, 2023). This complex behaviour underscores the role of both compositional and microstructural factors in governing the heat conduction properties of ash-reinforced clay composites.

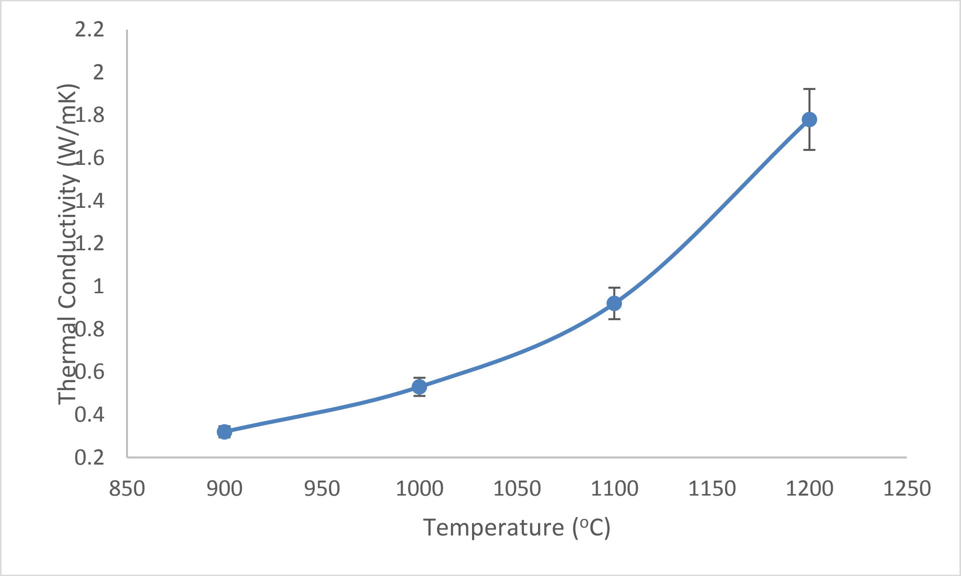


Figure 7. Thermal Conductivity of Uguwoba Clay reinforced with 20.66wt.% PSA

3.6 Cold Crushing Strength of Ugwuoba Clay Reinforced with 20.66 wt.% PSA

Cold crushing strength (CCS) is a critical mechanical property for fired ceramic and refractory composites, reflecting the material's ability to resist compressive loads at ambient conditions. CCS is influenced by the extent of sintering, phase development, and microstructural integrity achieved during thermal treatment. In the Ugwuoba clay composite reinforced with 20.66 wt.% PSA, CCS increased markedly with firing temperature, indicating improved mechanical interlocking of particles and enhanced densification at higher thermal exposures. In numerous studies, an increase in firing temperature has been correlated with rising mechanical strength in clay-based ceramics due to reduced porosity and increased solid-state bonding between grains (Egole et al., 2024). For example, research on blended clay systems shows that higher firing temperatures improve properties such as cold crushing strength as a result of reduced void spaces and stronger interphase bonds (Egole et al., 2024). In PSA-reinforced composites, this trend may be reinforced by the pozzolanic and fluxing behaviour of PSA constituents, which can facilitate the formation of additional silicate or glassy phases that bridge clay particles and resist crack propagation.

The enhanced CCS values observed in this study suggest that the PSA not only contributes to densification but also improves load-bearing capacity, possibly through the formation of thermally stable phases and stronger particle interactions. Such behaviour has been documented in other clay-ash systems where the introduction of ash additives improved compressive or crushing strength by enhancing matrix cohesion and reducing stress concentrators such as open pores (Egole et al., 2024). As a result, the positive correlation between temperature and CCS in the PSA-reinforced Ugwuoba clay supports its potential for high-temperature applications requiring robust mechanical performance.

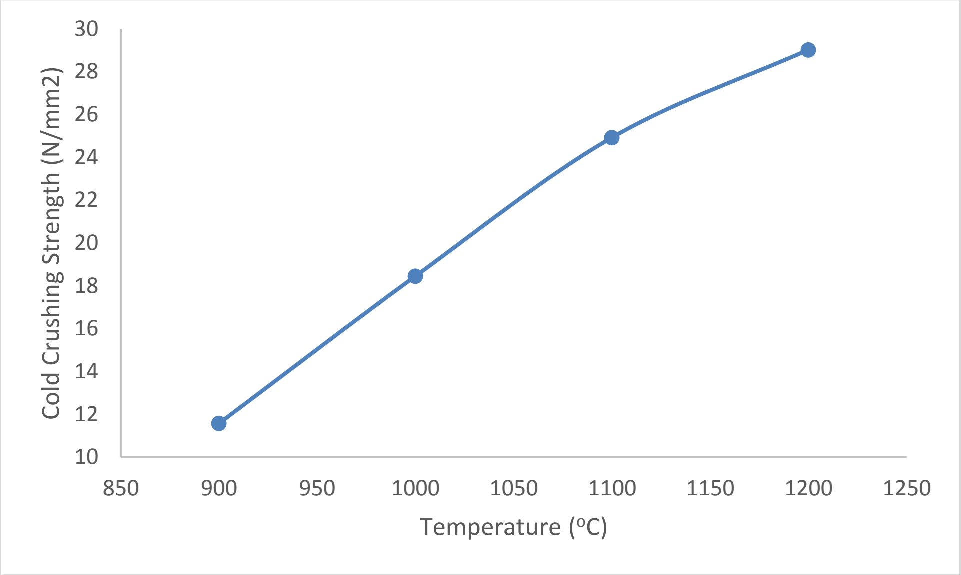


Figure 8. Cold Crushing Strength of Uguwoba Clay reinforced with 20.66wt.% PSA

3.7 Thermogravimetric Behaviour of Ugwuoba Clay Reinforced with 20.66 wt.% PSA

Thermogravimetric analysis (TGA) was performed to assess the thermal stability and decomposition characteristics of the Ugwuoba clay composite reinforced with 20.66 wt.% PSA. Overall mass changes as a function of temperature indicate key transitions in the composite's behaviour under heating. The initial weight loss up to approximately 100 °C is attributed to the removal of physically adsorbed moisture, a common feature of clay-based materials due to their hygroscopic nature (Salifou et al., 2024). Between approximately 100 °C and 600 °C, a more pronounced mass loss was observed, reflecting the decomposition of any residual organic constituents in the PSA and dehydroxylation of clay minerals—consistent with TGA profiles reported for agro-waste-modified clay systems, where organic matter volatilises at elevated temperatures (Salifou et al., 2024).

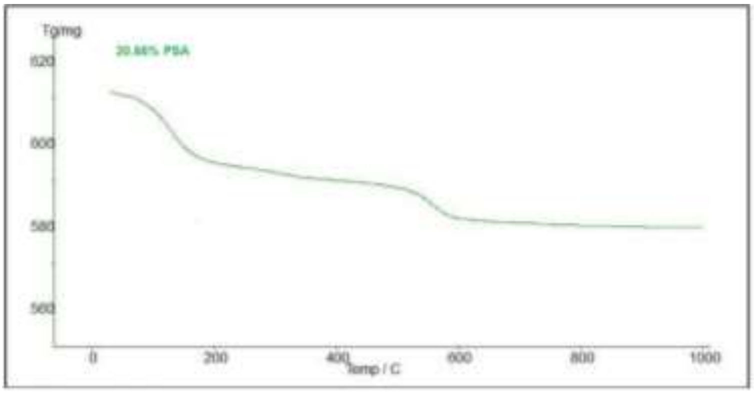


Figure 9. Thermogravimetric behaviour of Uguwoba Clay reinforced with 20.66wt.% PSA

Beyond 600 °C, the TGA curve tends toward a plateau, indicating reduced mass loss and the onset of a thermally stable regime. This behaviour aligns with findings in other refractory and clay systems where high-temperature phase formation and stable crystalline phases suppress further decomposition (Salifou et al., 2024). The presence of PSA, which introduces silica and fluxing oxides into the composite, can also contribute to this enhanced stability by facilitating the formation of thermally resistant silicate phases during firing. Similar observations have been reported in clay composites incorporating palm kernel shell ash, where improved thermal stability at elevated temperatures was linked to the development of stable refractory phases (Sarani et al., 2023). The TGA results therefore demonstrate that the PSA-reinforced Ugwuoba clay exhibits robust thermal behaviour, retaining most of its mass beyond 600 °C and indicating suitability for high-temperature applications requiring thermal resilience.

3.8 Implications for Refractory and Crucible Applications

The combined thermal and mechanical behaviours of the PSA-reinforced Ugwuoba clay composite provide a strong basis for evaluating its potential as a refractory material suitable for foundry crucibles. Refractory materials must exhibit high thermal stability, mechanical strength at elevated temperatures, and resistance to structural degradation under cyclic thermal loads (Nemaleu et al., 2021). The observed TGA profile, which indicates minimal mass loss beyond early dehydration and organic decomposition, suggests that the composite maintains structural integrity under thermal stress—an essential attribute for crucibles subjected to temperatures well above 600 °C during metal melting operations. Furthermore, the progressive increase in cold crushing strength with firing temperature indicates that the composite develops robust mechanical cohesion, which is necessary to resist thermal shock and mechanical loads during crucible use. Other studies on refractory materials incorporating agro-waste residues have similarly highlighted these dual benefits of enhanced strength and thermal stability (Salifou et al., 2024). In particular, the presence of fluxing oxides and silica from PSA supports the formation of thermally stable phases that can withstand high temperatures without rapid degradation, improving refractory performance.

4.0. Conclusion

The study titled “Effect of Palm Stalk Ash Reinforcement on the Microstructural, Thermal and Mechanical Properties of Ugwuoba Clay for Refractory Applications” investigated the potential of incorporating agro-waste–derived Palm Stalk Ash (PSA) into Ugwuoba clay to enhance its suitability for high-temperature refractory uses, particularly in foundry crucibles. The research was guided by the central argument that reinforcing locally available clay with PSA could improve its microstructural stability, densification behaviour, and mechanical performance while promoting sustainable waste utilization. The results demonstrated that the addition of 20.66 wt.% PSA significantly influenced the clay matrix, producing a heterogeneous but strongly bonded microstructure and promoting the formation of thermally stable silica-rich and calcium silicate phases. Increasing firing temperatures from 1100 °C to 1200 °C resulted in reduced porosity and water absorption, increased bulk density, accelerated sintering behaviour, and improved thermal stability, while cold crushing strength increased substantially, indicating enhanced load-bearing capacity and structural integrity. These findings confirm that PSA reinforcement effectively improves the thermal, physical, and mechanical performance of Ugwuoba clay, highlighting its potential as a sustainable and cost-effective material for refractory components such as foundry crucibles. The study contributes to the growing body of research on agro-waste-reinforced ceramic materials and provides a practical pathway for converting agricultural residues into value-added industrial resources. Future studies may further explore varying PSA compositions, different reinforcement percentages, long-term thermal shock resistance, and industrial-scale production to optimize the material’s performance for broader refractory and high-temperature engineering applications.

References

Alhaji, M. M., Alhassan, M., Waheed Adejumo, T., & Ibrahim Dogo, A. (2020). Microstructural investigation and strength properties of clay stabilized with cement, rice husk ash and promoter. Jurnal Teknologi, 82(5). https://doi.org/10.11113/jt.v82.14353.

Bomeni, I. Y., Fotsing Metegam, I. F., Nzeukou Nzeugang, A., Kieufack, G., & Fagel, N. (2025). Geological and geotechnical characterization of clayey materials from Feutap (West Cameroon): suitability in ceramic industry. Discover Geoscience, 3(1). https://doi.org/10.1007/s44288-025-00177-8

Egole, C. P., Nzebuka, G. C., Ufodike, C. O., Medupin, R. O., Ugwuegbu, C. C., Nnodum, N. A., & Ochieze, U. P. (2024). Experimental characterization of two clay deposits blended with feldspar and quartz for building services and refractory applications. Journal of African Earth Sciences, 218, 105373. https://doi.org/10.1016/j.jafrearsci.2024.105373

Freitas-Dutra, L., Freitas, M., Grillet, A.-C., Mendes, N., & Woloszyn, M. (2019). Microstructural Characterization of Porous Clay-Based Ceramic Composites. Materials, 12(6), 946. https://doi.org/10.3390/ma12060946

Huseien, G. F., & Mhaya, A. M. (2026). A Comparative Study of Utilizing Waste Palm Oil Fuel Ash and Tile Ceramics to Enhance Slag–Fly Ash Geopolymer Property-Based Composite. Journal of Composites Science, 10(1), 33. https://doi.org/10.3390/jcs10010033

Joshua, A. G., Moyosiore, A. A., Oni, T. O., & Temitope, S. J. (2025). Influence of agro-waste additives on the microstructural and physical properties of refractory bricks. International Journal of Research and Innovation in Applied Science, 10(4), 112–121.

Makrygiannis, I., Karalis, K., & Tzampoglou, P. (2025). Enhancing the thermal insulation properties of clay materials using coffee grounds and expanded perlite waste: A sustainable approach to masonry applications. Ceramics, 8(2), 30. https://doi.org/10.3390/ceramics8020030

Maury-Njoya, I. Q., Lecomte-Nana, G. L., Barry, K., Njoya, D., El Hafiane, Y., & Peyratout, C. (2024). An Overview on the Manufacture and Properties of Clay-Based Porous Ceramics for Water Filtration. Ceramics, 8(1), 3. https://doi.org/10.3390/ceramics8010003

Nemaleu, J. G. N. D., Kaze, R. C., Kamseu, E., & Sglavo, V. (2021). Controlling the thermal stability of kyanite-based refractory geopolymers. Materials, 14(11), 2903.

Obada, D., Dodoo-Arhin, D., Muhammad Dauda, Fatai Anafi, Ahmed, A., Ajayi, O., & Ibraheem Samotu. (2016). Physical and Mechanical Properties of Porous Kaolin Based Ceramics at Different Sintering Temperatures. West Indian Journal of Engineering. https://journals.sta.uwi.edu/ojs/index.php/wije/article/view/7723.

Salifou, G., Harouna, S., Makinta, B., & Madougou, S. (2024). Thermal Performance of Clay and Millet Waste Compressed Earth Blocks Stabilized with Cement. International Journal of Materials Science and Applications, 13(4), 71–80. https://doi.org/10.11648/j.ijmsa.20241304.12

Sánchez-Soto, P. J., García-Garzón, V., Martínez-Martínez, S., Pérez-Villarejo, L., Sánchez-Garrido, J. A., & Garzón, E. (2025). Influence of features and firing temperature on the ceramic properties and phase evolution of raw kaolins. Construction and Building Materials, 466, 140215. https://doi.org/10.1016/j.conbuildmat.2025.140215

Sánchez-Soto, P. J., Garzón, E., Pérez-Villarejo, L., & Eliche-Quesada, D. (2021). Sintering behaviour of a clay containing pyrophyllite, sericite and kaolinite as ceramic raw materials: Looking for the optimum firing conditions. Boletín de La Sociedad Española de Cerámica Y Vidrio, 62(1), 26–39. https://doi.org/10.1016/j.bsecv.2021.09.001

Sarani N.A., Kadir A.A., Hashim M.A.A, Hassan, Hamid, A., Huda, N., Nur, & Johan, S. M. (2023). Physical-mechanical properties and thermogravimetric analysis of fired clay brick incorporating palm kernel shell for alternative raw materials. Construction and Building Materials, 376, 131032–131032. https://doi.org/10.1016/j.conbuildmat.2023.131032

Sdira, H., Farissi, L., Arbaoui, M. A., Waqif, M., Arbaoui, A., Saadi, L., Tlemçani, M., & Bouich, A. (2024). Enhancing energetic efficiency: Structural and thermal properties of fly ash-clay ceramics. JP Journal of Heat and Mass Transfer, 37(3), 349–364.

Utkarsh, & Jain, P. K. (2024). Enhancing the properties of swelling soils with lime, fly ash, and expanded polystyrene -A review. Heliyon, 10(12), e32908. https://doi.org/10.1016/j.heliyon.2024.e32908