



CHARACTERIZATION OF GULA CLAY FOR REFRACTORY APPLICATIONS

M. M. Dukku¹, M. A. Bawa², A. Tokan², M. H Muhammad²

¹Department of Mechanical Engineering, Nigerian Army University Biu, Nigeria

²Department of Mechanical Engineering, Abubakar Tafawa Balewa University, Bauchi, Nigeria

*Corresponding author (Email: mdukk@gmail.com)

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Abstract: This study evaluates the physical, thermal, chemical, mineralogical, and microstructural characteristics of raw clay sampled from Gula town in the Biu Local Government Area of Borno State, Nigeria, to determine its suitability as a local alternative for industrial refractory applications. The clay sample was processed and subjected to standardized testing profiles including apparent porosity, bulk density, moisture content, firing shrinkage, loss on ignition (LOI), thermal shock resistance, and cold crushing strength (CCS). Chemical and mineralogical compositions were determined using X-ray fluorescence (XRF), X-ray diffraction (XRD), and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM/EDS). The results showed that Gula clay satisfies the specifications for a standard fireclay. The evaluation physical and mechanical properties revealed an apparent porosity of 23.02%, a bulk density of 1.88 g/cm³, a moisture content of 4.06%, and a low firing shrinkage of 7.2%, which minimizes the risk of structural warping. Mechanically, the clay exhibited a high CCS of 26.52 MPa and an excellent thermal shock resistance of 26 cycles, confirming its ability to withstand acute thermal stresses in high-temperature environments. XRF analysis indicated major oxide concentrations of SiO₂ (55.24%) and Al₂O₃ (22.72%), while qualitative XRD analysis confirmed quartz (55.14.0%) and orthoclase feldspar (34.0%) as the dominant primary mineral phases, alongside minor fractions of goethite and anorthite. SEM/EDS microstructural analysis revealed highly dispersed, non-spherical particles with tight intergranular bonding and Mg was detected at approximately 4.0 wt.% in the analyzed EDS region, although Mg was not significant in the bulk XRF composition. Collectively, these results establish that Gula clay possesses the required thermomechanical and chemical benchmarks to serve as a viable, low-cost local substitute for imported siliceous fireclay refractory materials in industrial refractory applications.

Key words: Fireclay, Characterization, Gula Clay, Refractory and Properties

1. Introduction

Clay minerals are naturally occurring materials consisting primarily of tiny crystalline minerals which occur as a result of the chemical weathering of granite and feldspathic (Omidiji *et al.*, 2023). They are very small, colloidal-sized crystals and the individual crystals are in the form of tiny plate-like flakes consisting of many crystal sheets which have a repeating atomic structure and the different types of clay minerals are determined basically by the structure

(Moussa *et al.*, 2022; Uwaezuoke *et al.*, 2022; Olusola, 2026). Clay minerals are classified into groups on the basis of variations of chemical composition and atomic structure as kaolinite, illite, vermiculite, smectite and chlorite groups (Samad *et al.*, 2021). It is a hydrous aluminum-silicate that exhibits plasticity properties when mixed with water but becomes hard again upon drying and firing. Clays are a natural aggregate of minerals classified into four main groups: kaolinite, montmorillonite-smectite, illite, and chlorite depending

on the mineral that is dominant in the clay (Michael *et al.*, 2022).

Nigeria is blessed with huge deposits of clays across all its six geo-political zones. These deposits are estimated to be around three billion metric tons of kaolinite clay and an estimate of more than 700 million metric tons of bentonite clay (Ogunro *et al.*, 2023). Proper exploitation of these vast resources for economic benefit relies on the characterization of the various deposits to determine their suitability in potential application either in raw or beneficiated form (Adegbola *et al.*, 2024).

Some studies have been carried out on some deposits for specific areas of application (Aramide *et al.*, 2014). The physical, chemical and mineralogical characteristics of these clay deposits with unique properties related to their characterization and quality important for the technical performance of local products (Abubakar *et al.*, 2025).

Many researchers have investigated the various properties of clays in Nigeria from different regions for industrial use. For example, (Adegbola *et al.*, 2024) reported that Lukosi clays has properties within the appropriate standard values, which qualifies it as a refractory, ceramic, and high-melting clay. Similarly, (Shuaib-Babata *et al.*, 2018) studied the suitability of Baruten Clays in Kwara State, Nigeria and reported that the clay could suitably replace the imported clays in some refractory applications having exhibited good physical and thermal properties. Clay deposits in Northeastern Nigeria, especially in the south axis of Borno State, represent a largely unexploited resource for the innovative processing of low-cost refractory materials that might be a replacement of imported materials.

This study aims to characterize Gula clay as a potential raw material for refractory applications. The study evaluates its physical, chemical, mineralogical, thermal, mechanical and microstructural characteristics using analytical and performance-based approaches. This will establish relationships between refractory performance and the various oxides and mineral phases identified, thereby providing a basis for understanding the behaviour of the clay. The investigation also provides experimental evidence for assessing whether Gula clay can serve as a locally available alternative to imported siliceous fireclay materials. This approach is significant because the identification and utilization of suitable indigenous refractory resources could

contribute to the development of locally produced refractory materials and reduce reliance on imported raw materials. The study is designed not merely to determine whether Gula clay satisfies selected refractory specifications, but to establish a comprehensive property structure assessment of the clay. The result will provide a basis for evaluating its potential application in refractory products, including refractory bricks and linings used in high-temperature industrial environments. The characterization of Gula clay contributes to the broader body of knowledge on Nigerian refractory clays and provides baseline scientific data for the future development and optimization of locally sourced refractory materials.

2. Materials and Methods

2.1 Materials

The clay used in this study was collected from Gula town in Biu emirate of Biu Local Government in Borno State Nigeria (latitude 7°30'–7°35'N, longitude 5°45'–5°50'E). A representative bulk sample of 5 kg was extracted, hermetically sealed in polyethylene packaging to preserve run-of-mine moisture, and transferred to the laboratory.

2.2 Methods

The samples were air-dried, and crushed to powder form with a pestle and mortar to achieve homogeneity of the particle size. The ground samples were sieved with a 75µm mesh according to the ASTM D422/D6913 standards. The sample oven dried at a temperature of 110 °C for at least 24 hours to ensure that all moisture content is removed completely.

2.2.1 Apparent porosity

Apparent porosity Gula clay was determined using a prepared test sample of size 30 mm diameter by 4 mm thickness. The compacted test sample was heated in a furnace at 1100°C and then allowed to cool. It was brought out of the furnace, weighed and recorded as weight D. The clay sample was suspended in distilled water for 30 minutes and weighed as weight W and recorded. The clay sample was removed from water and the water on the surface of the specimen was cleaned off using filter paper and weighed as soaked weight S and recorded (Amkpa *et al.*, 2017). Apparent porosity (AP) was calculated using Equation (1)

$$AP = \frac{W - D}{W - S} \times 100 \quad (1)$$

2.2.2 Moisture Content

The determination of moisture content in the clay sample follows the ASTM C72-76 standard, which gave the procedure for the analysis of water content of refractory materials. The wet mass of the sample is recorded M_w and dried in an oven at 110 °C for about three hours. The final weight of the sample is recorded M_d , and the moisture content is computed as described by (Eni *et al.*, 2025) using Equation (2).

$$\begin{aligned} \text{Moisture Content} &= \frac{M_w - M_d}{M_w} \\ &\times 100 \end{aligned} \quad (2)$$

2.2.3 Bulk Density

The prepared samples were air-dried for 12 hours and then oven dried at 110°C and cooled. The samples were then fired at a temperature of 1100°C in a furnace and allowed to cool in a desiccator. The dried weight of the sample was recorded as W_1 . The clay sample was suspended in distil water for 30 minutes and the soaked weighs was measured and recorded as V_1 . The sample was then suspended in a beaker using a sling and the respective suspended weight was measured and recorded (V_2) as highlighted by (Olajide *et al.*, 2015). The bulk density of the sample was determined using equation (3) according to ASTM C20.

$$\text{Bulk density} = \frac{W_1}{V_1 - V_2} \quad (3)$$

2.2.4 Firing Shrinkage

The sample was prepared to standard brick of size 20 mm × 50 mm × 100 mm and marked along a length to maintain the same position after heating. The sample was then dried in still air for 24 hours and later dried at temperature of 110°C in oven for 6 hours, allowed to cool at room temperature (L_1). The sample is then fired in a muffle furnace at temperature of 1100°C. The dried/fired length of the sample was recorded (L_2) and the firing shrinkage values of the sample was determined using the relationship in equation (4) following the ASTM (C326, 2018) standard as described by (Attah and Oden, 2010; Jongs *et al.*, 2018).

$$\text{Firing Shrinkage} = \frac{L_1 - L_2}{L_1} \times 100 \quad (4)$$

2.2.5 Cold Crushing Strength

The Cold crushing strength (CCS) of the clay was performed by preparing test sample to standard brick of

size 20 mm × 50 mm × 100 mm. This cubic shaped size was used to test for cold crushing strength using the universal testing machine. The sample was fired at a temperature of 1100°C while holding it for 1-2 hours and cooled slowly in the furnace. The application of a uniform load on it was followed. The CCS was determined according to ASTM C133-97 (Bhardwaj *et al.*, 2017). The weight pressure at which crack occurred on the sample was observed and the cold crushing strength was calculated using Equation (5) as described by (Czechowski *et al.*, 2015).

$$\begin{aligned} CCS &= \frac{\text{maximum load (kN)}}{\text{cross-sectional area (m}^2\text{)}} \\ &= \frac{LD}{SSA} \end{aligned} \quad (5)$$

2.2.6 Loss on Ignition

Loss on ignition was determined based on the Lechler and Desiletes method. The sample was oven-dried at 110°C. 1g of each of the sample of the clay was preheated, cooled, and pre-weighed (W_2) and heated to 1000°C in a furnace for an hour. The crucibles and contents were removed, cooled in a desiccator to room temperature, and weighed again (W_2). The loss in weight was calculated as mentioned by (Rat *et al.*, 2023). The loss on ignition was calculated using equation (6).

$$\begin{aligned} \text{Loss on Ignition (LOI)} &= \frac{w_1 + w_2}{w_1} \\ &\times 100 \end{aligned} \quad (6)$$

2.2.7 Thermal Shock Resistance

Thermal Shock Resistance was determined by placing the clay sample in a furnace and maintaining a temperature of 1200°C for about 10 minutes. The sample was then removed and allowed to cool in a desiccator for 10 minutes. The sample was then put back into the furnace while maintaining the temperature at 1200°C for another 10 minutes. This process was repeated until cracks were observed on the sample. The number of heating and cooling cycles of the sample was recorded according to ASTM C1171-16(2022). The values obtained at different instances were recorded (Griffin and Miller, 2008; Rongos and Aneziris, 2012).

2.2.8 Chemical Composition

Elemental composition of the clay sample was determined using XRF spectrometry. Powdered sample was fused with lithium tetraborate (1:10 ratio) to form glass beads and analyzed under vacuum

conditions. Major and minor oxides such as SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , K_2O , and TiO_2 were identified. The XRF analysis provided bulk chemical composition data used to deduce purity, elemental composition, and potential suitability for industrial refractory applications (Ugwuoke and Amalu, 2017; Singh *et al.*, 2022).

2.2.9 Mineralogical Composition

Mineralogical identification was performed using a PANalytical B. V., Axion spectrometer using X-ray powder diffraction technique. Two theta range: 0 – 160 degrees (WAXS), Voltage: 20 – 60kv; X-ray wavelength: 1.54 λ . The sample conditions were recorded from 10° to 70° 2 θ with a step size of 0.02° at 1s per step as described by (Aliyu *et al.*, 2013).

2.2.10 Scanning Electron Microscopy

Microstructural and surface morphological analyses were performed using the JEOL 840; The SEM photograph was obtained from the clay sample was captured under low-vacuum (or variable pressure) conditions. Micrographs were recorded at magnifications of 8000x, 9000x and 10,000x at working distances of 10.4mm, 10.2mm and 8.4mm respectively and accelerating voltage of 20KV, while EDS provided point and area chemical analyses. The technique revealed particle morphology, agglomeration, and pore structure, showing correlation with mechanical properties (Silva *et al.*, 2021).

3 Result and Discussion

3.1 Apparent porosity

The results of the apparent porosity of the clay sample examined showed that it exhibits a low porosity of 23.02%, this is an indication that the clay is suitable for use as a refractory material having fallen within the standard limit of 20-30% porosity for fireclay according the ASTM C20 (Ranti *et al.*, 2015).

3.2 Moisture Content

The clay exhibits a good moisture content, having a moisture content of 4.06% which is within the recommended standard values. The result indicates Gula clay contains water adequate for proper mixing and better molding capacity into the desired shapes (Serawat *et al.*, 2020).

3.3 Bulk Density

The result of the bulk density test carried out on the clay showed that the clay possesses a bulk density of 1.88

g/cm^3 . It is important to note that bulk density as a clay property is a major criterion in material selection for refractory production. The bulk density of the clay samples fell within the standard values of 1.7-2.3 g/cm^3 for fireclay refractory brick (Laita *et al.*, 2021).

3.4 Firing Shrinkage

The results of the investigation on the firing shrinkage of the clay sample showed that it exhibits a low shrinkage of 7.2%. This value conforms with the standard recommended firing shrinkages of 4-10% for refractory clays as high shrinkage values may result in warping and cracking of the clay and this may cause loss of heat in the finished products (Mokwa *et al.*, 2019).

3.5 Cold Crushing Strength

The reason of undertaking this test is to determine the ability of the clay to withstand stresses in service. The Cold Crushing Strength test conducted on the clay sample indicated that Gula clay has a high Cold crushing strength (CCS) of 26.52Mpa. This value of CCS falls within the recommended standard of 15 – 59 MPa as indicated by (Ugwuoke and Amalu, 2017) in their work on oberefractory clays.

3.6 Loss on Ignition

Loss on ignition is the combustion of volatile matter present in the clay which are often required to be low. The results of the test on Gula clay samples showed a LOI of 5% which fall within the recommended standard values of loss on ignition of refractory clays as limit for low melting clay (3-15%) and (4-12%) for high melting clay. This indicates the suitability of the clay samples to be used for application in metallurgical industries as reported by (Chima *et al.*, 2017).

3.7 Thermal Shock Resistance

The Gula clay demonstrated exceptional thermal shock stability, surviving 26 cycles before

failure. This performance compares favourably with the standard benchmark of 20–30 cycles expected of fireclays (Eni *et al.*, 2025), satisfy ASTM C1171, validating the material's capacity to withstand severe cyclical thermal gradients without failing (Oke and Nenuwa, 2023).

3.8 Chemical Composition results

The chemical composition of the Gula clay sample is presented in Table 1 as determined using XRF spectrometer. The results showed high concentration of SiO_2 and Al_2O_3 in the clay. High SiO_2 content is associated with crystalline phase of quartz, and when combined with alumina to form aluminosilicate clays. Alumina content is usually associated with the presence of orthoclase phase confirming the clays suitability in refractory applications (Nayak and Singh, 2007). This can be confirmed by XRD analysis.

Table 1: Chemical Composition of Gula Clay

Clay Oxides	Percentage % wt	Clay Oxides	Percentage % wt
SiO_2	55.24	CaO	2.91
Al_2O_3	22.72	TiO_2	2.76

Fe_2O_3	5.20	MnO	0.19
K_2O	6.54	SrO	0.13

3.9 Mineralogical Composition of Gula Clay

Result on the XRD analysis as shown in Figure 1 shows a large amount of quartz with some alumina present, and the qualitative XRD results in Table 2 corroborates this assertion. The major peaks in the XRD results indicates high presence of quartz (SiO_2) phase, and feldspars in the form of orthoclase ($\text{K Al Si}_3 \text{O}_8$), Goethite ($\text{Fe}_2 \text{O}_3 \cdot \text{H}_2 \text{O}$) and Anorthite also present in the clay sample. Relatively high intensity of quartz peaks indicates significant presence of free silica indicating suitability in refractory applications (Olaremu, 2015).

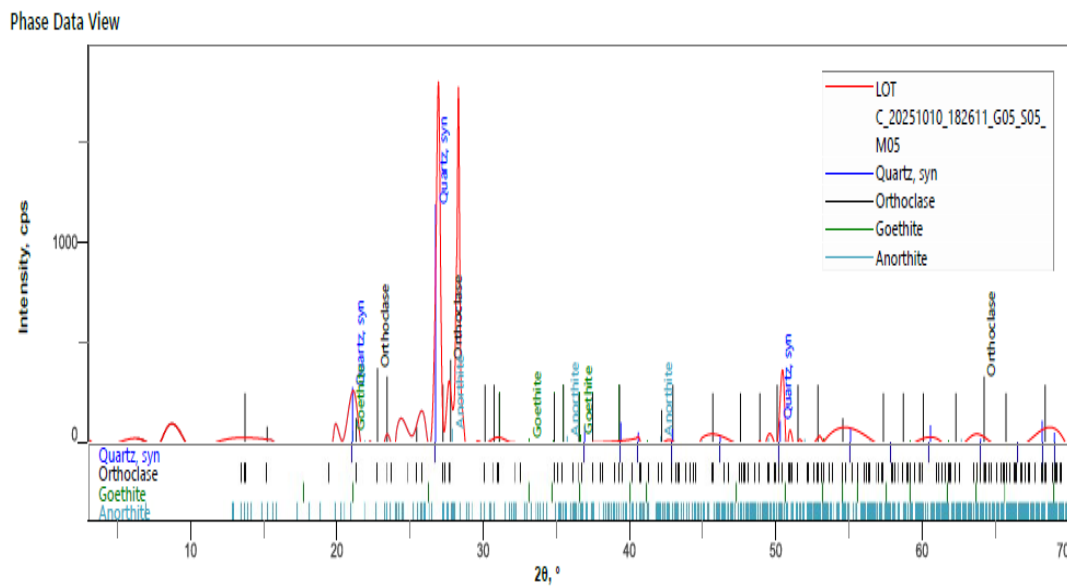


Figure 1: XRD Analysis of Gula Clay

Table 2: Qualitative analysis of Gula Clay Mineral

No.	Phase Name	Formula	Weight (wt. %)
1	Quartz,	SiO_2	55.14
2	Orthoclase	$\text{K Al Si}_3 \text{O}_8$	34.0
3	Goethite	$\text{Fe}_2 \text{O}_3 \cdot \text{H}_2 \text{O}$	5.2
4	Anorthite	$\text{Ca Al}_2 (\text{Si O}_4)_2$	2.0

3.10 Scanning electron microscopy (SEM) The particle morphology of the clay was examined using SEM at the secondary electron mode. The corresponding images are given in Fig. 2a, 2b, and 2c respectively. The tight intergranular bonding indicates adequate firing and should support good cold crushing strength. The clay particles exhibit

an irregular shape in the SEM image with silicate flakes of kaolinite and quartz for all the clay samples (Szczerba, 2010; Majdoubi et al., 2021). The presence of quartz was revealed by rounded

platelets with brighter luminescing and this confirms the results of XRD.

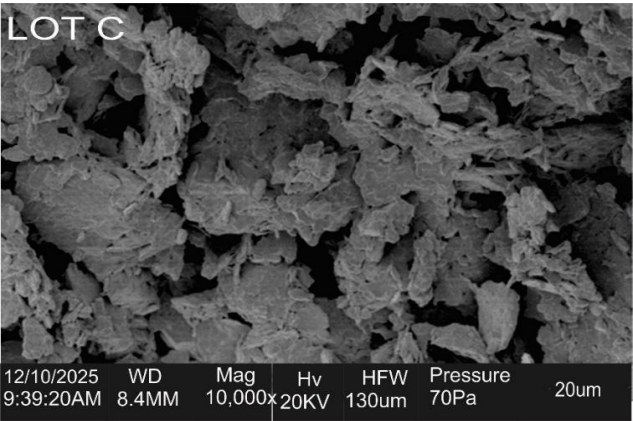


Figure 2a: SEM Images of Gula Clay at 9000x, Magnification

The SEM images of the clay (Figure 2), revealed that the structural orientation of the clay exhibits highly dispersed particles of various sizes. The elemental composition of the clays obtained from the electron

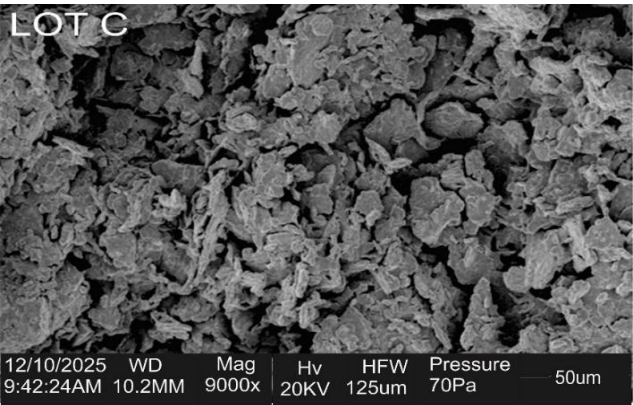


Figure 2b: SEM Images of Gula Clay at 10000x Magnification

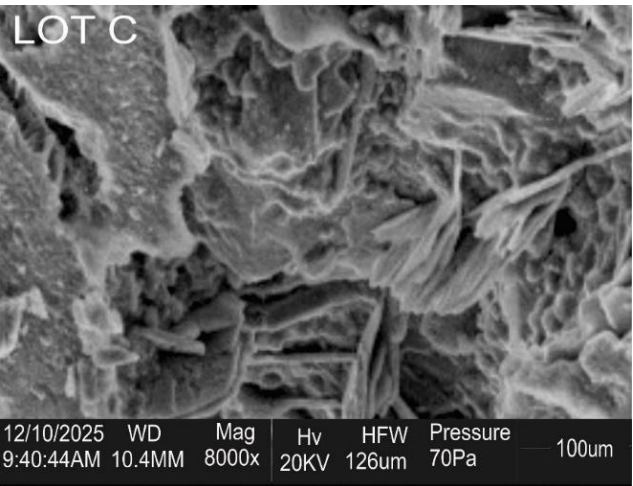


Figure 2c: SEM Images of Gula Clay at 8000x Magnification

diffraction spectrum shows the predominant elements as aluminium, silicon carbon, and oxygen, calcium, iron and pottasium, as the minor elements (Figure 3). The SEM–EDS analysis of Gula clay revealed Si and O as the predominant elements, with unnormalized concentrations of approximately 50.0 and 20.0 wt.%, respectively. Al (12.6 wt.%), Mg (4.0 wt.%), Fe (3.0 wt.%), K (2.0 wt.%), Ca (1.3 wt.%) and C (3.0 wt.%) were also detected. The pronounced Si signal is consistent with the quartz-rich mineralogical composition identified by XRD, while the simultaneous occurrence of Si, Al and K is compatible with the presence of orthoclase. Similarly, the Si–Al–Ca association is consistent with an orthite, whereas the Fe signal is compatible with the presence of goethite. This elemental distribution reflects the specific geochemical provenance of the deposit, indicating the clay’s usefulness

across various industrial processes (Mohammed and Abdullah, 2018).

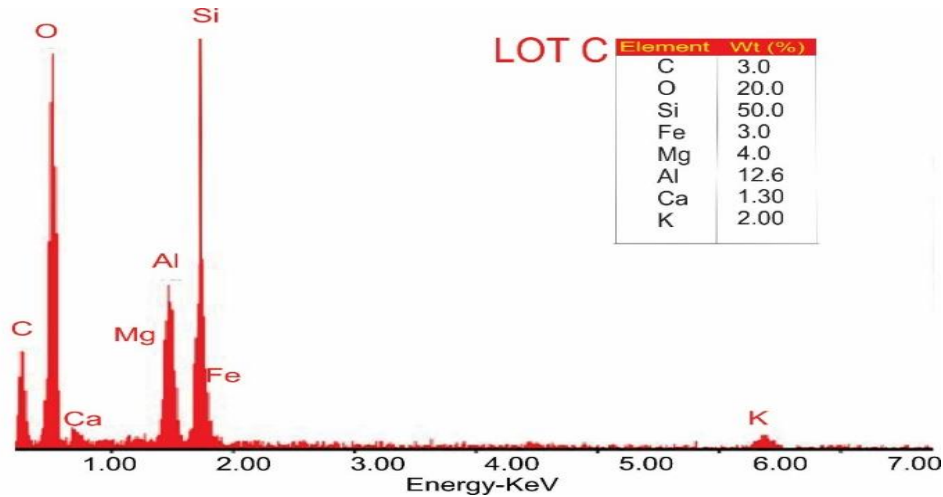


Figure 3: Energy-Dispersive X-ray Spectroscopy (EDS)

Table 3: Physical/Mechanical Properties of Gula clay with Standards.

Properties	Standard	Gula Clay
Apparent Porosity (%)	20-30	23.02
Moisture Content (%)	4-10	4.06
Bulk Density (g/cm ³)	1.7-2.3	1.88
Firing Shrinkage (%)	4-10	7.2
Cold Crushing Strength (MPa)	15-59	26.52
Loss on Ignition	3-15	5
Thermal Shock Resistance (cycles)	20-30	26

Table 3 shows the properties of Gula Clay compared with standard values of refractory clays. It can be observed that Gula clay properties compares favorably with the standards as they fall within the ranges.

The properties and chemical characteristics of Gula clay compare favourably with those reported by researchers that studied various Nigerian clay deposits. These results place Gula clay within the aluminosilicate fireclay category indicating a chemical composition similar to several Nigerian refractory clays. For example, Baruten fireclays from Kwara State studied by Shuaib-Babata *et al.*, (2018) were reported to contain relatively high SiO₂ and Al₂O₃ contents and exhibited apparent porosity of 19.4–25.6%, bulk density of 1.83–1.90 g/cm³, cold crushing strength of 38.7–56.1 MPa, thermal-shock resistance of 23–25 cycles demonstrating their suitability for refractory applications. Also, Isan-Ekiti and Ire-Ekiti clays contained 53.30 and 56.90% SiO₂ and 25.95 and

21.45% Al₂O₃, respectively, with quartz and kaolinite identified as the principal mineral phases as identified by Oke & Nenuwa, (2023). Compared with these clays, Gula clay has a comparable silica concentration. In terms of physical and mechanical performance, Gula clay exhibited 23.02% apparent porosity, 1.88 g/cm³ bulk density, 7.2% firing shrinkage, 26.52 MPa cold crushing strength, and thermal-shock resistance of 26 cycles. These values fall within the ranges reported for Baruten fireclays, especially with respect to porosity, density, shrinkage and thermal-shock resistance.

4 Conclusion

Gula clay was successfully characterized to assess its suitability as a source of refractory materials in our local industries. The Clay was found to be of Alumino Silicate group having 55.14% Silica and 22.72% Alumina. The relatively low Al₂O₃ content may limit the refractoriness of the material, since Al₂O₃ contributes significantly to the high-temperature stability and resistance to softening of clay materials. The presence of 5.2% iron oxide in the clay is relatively high for a refractory clay and could contribute to fluxing during firing, however this can be mitigated by reducing the iron oxide content through beneficiation or by blending with lower iron material to meet refractory specifications. The clay samples showed good physico-mechanical properties such as moisture content, linear shrinkage, bulk density, porosity loss on ignition, cold crushing strength and thermal shock resistance when compared to recommended standards for refractory clays. This makes the clay suitable for refractory applications such as in metallurgical industries as insulating refractories in casting and

melting of iron and steel, smelting furnaces; in the production of refractory bricks for kilns and clinker coolers in cement industries. The clay is found to be good material for refractory applications.

Local exploitation of the deposit could reduce the cost associated with the importation of refractory raw materials while creating economic opportunities in clay extraction, beneficiation, processing and manufacturing. The use of Gula clay as a locally sourced refractory material can provide economic benefits through the development of indigenous alternatives to imported refractory materials. Commercial utilization of this clay could help in value addition to Nigeria's indigenous mineral resources, generate employment within the local supply chain and contribute to the development of a domestic refractory industries. However, the realization of these economic benefits depends on the availability and scale of the deposit, beneficiation and processing requirements, production costs, performance and economic analysis on commercially available refractory materials.

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