

Performance Characteristics and Evaluation of Silica Refractory Bricks from Quartzite Rock

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Abstract

This study focused on the development of insulating refractory bricks using quartzite, with a comprehensive analysis of their mechanical and thermal properties. The bricks were made from quartzite with 10% bentonite binder and water, added for mixing. Various tests were done (X-ray Fluorescence, Thermogravimetric and Differential Thermal Analyses, apparent porosity, shrinkage, Loss on Ignition (LOI), compressive yield stress, and water absorption (WA)), following American Society for Testing and Materials' procedures. Microstructural analysis of the sample was observed using Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy (SEM-EDS). These results provided valuable insights into the stability of the developed refractory bricks at high temperatures. Analysis of chemical composition from quartz bricks revealed significant SiO₂ content (94.303%), emphasizing the material's superior refractory properties. LOI (0.5%) and shrinkage (0.2%) results indicated enhanced performance with good thermal stability and strength. Compressive stress yield indicated that the material exhibits good strength relative to the amount of binder used. WA results confirmed that the brick sample is suitable for high-temperature applications. SEM/EDS analysis unveiled intricate microstructural features, affirming the bonding agent's influence on the material's composition.

Keywords: Differential Thermal Analysis; refractory material; Scanning Electron Microscopy-Energy/Dispersive X-ray Spectroscopy; silica quartzite bricks; Thermogravimetric Analysis.

Introduction*

Quartz (SiO₂) is extremely common and chemically durable [1]. It is very resistant to weathering and it often persists as sands and gravels when another mineral erode

*The abbreviation list is in page 608.

away. Quartz is found almost everywhere on Earth, and it forms in all kinds of environments from the intense heat of igneous rocks to the pressure-cooked zones of metamorphic rocks, and even within more ordinary sedimentary formations [1]. Quartz is tough; it stands up well to both chemical breakdown and physical wear. Thanks to its strength and abundance, quartz can be mined in many parts of the world. In Nigeria, it is especially plentiful and plays an important role in several industries [2]. At its core, quartz is a hard, crystal-clear mineral made of silicon and oxygen. It is used in everything from glass and ceramics to electronics. What makes it so valuable in manufacturing is its unique ability to handle high heat and resist chemical corrosion, qualities that make it indispensable across a range of industrial processes [1-2].

Quartz can be found in several states across Nigeria, including Ogun, Osun, Kogi, Kwara and Kaduna. The quality and size of these deposits vary, but some areas contain especially high-grade quartz that is ideal for industrial use [2-3].

In recent years, the Nigerian government has renewed its interest in developing the mining sector, with a particular focus on quartz and other solid minerals. There has been a strong push to attract foreign investment, and new policies have been put in place to make the industry more appealing and better regulated. These efforts reflect a broader strategy to position mining as a key contributor to Nigeria's economic growth and diversification [4].

Quartz is the most common form of crystalline silica and ranks as the second most abundant mineral found on the Earth's surface [5-6]. Its presence spans diverse rock types, which include igneous, metamorphic and sedimentary formations. Despite the widespread abundance of quartz deposits and its inclusion in almost all mining operations, the discovery of high-purity and commercially viable deposits is relatively infrequent. Silica sand deposits, a common derivative of quartz, are predominantly extracted through open pit operations, although alternative methods such as dredging and underground mining are also utilized in their extraction [5, 7].

When it comes to key performance factors like resistance to heat and thermal expansion under load (also known as creep), using quartzite with high chemical purity is essential. The stability, volume and transformation behavior of raw materials are critical in achieving bricks with the durability and performance needed for demanding applications [7]. Chemical composition of quartzite is a key factor in determining whether it is suitable for use as a raw material in silica refractory production. Impurities like alumina and alkalis tend to lower the melting point, which limits the brick's performance and the range of applications it can be used for [5, 7].

It is also important to assess how quartzite behaves while heated, as this affects its overall reliability in high-temperature environments. Silicon Dioxide (SiO_2) is the main ingredient in silica refractories. To help bind the material together, calcium-based compounds, often sourced from calcium hydroxide, are added during production. These bonding agents play a vital role in shaping the brick's structure and

performance once fired. It is noteworthy that these refractories may contain contaminants present in raw materials used during their production [7]. These refractories are designed to withstand high temperatures and various mechanical and chemical stresses. [8]. Refractories find extensive application in fundamental metal industries, particularly in the steel-making process through basic oxygen furnace. In this process, molten iron extracted from the blast furnace undergoes purification to eliminate impurities such as C, S, P, Mn, etc. This purification is achieved by introducing O into the molten iron [9].

Various refractories can be synthesized based on the production process and nature of raw materials. The fields of application of refractories are diverse, contingent on the properties of each type. The performance of a refractory, characterized by resistance to thermal shock, is directly associated with the texture and mineral richness, including mullite, corundum, periclase, dolomite, spinel and alumina [10].

Although quartz is one of the most abundant and chemically stable minerals in the Earth's crust, its potential as a high-performance refractory material, particularly in its rock form, remains underexplored. Previous studies have largely focused on more conventional refractory raw materials such as bauxite, alumina and magnesia, despite quartz's exceptional thermal resistance, chemical inertness and structural integrity under extreme conditions [11-14]. Recent investigations have highlighted the importance of high-purity silica, especially quartzite, in silica refractory applications, due to its low thermal expansion and excellent thermal shock resistance [12,13]. However, the literature often overlooks the untapped potential of naturally occurring quartz rock deposits, especially in regions like Nigeria, where the material is not only abundant but also variably rich in purity and particle size, two critical factors for refractory suitability [2, 14].

Given the renewed governmental focus on mining and industrial diversification in Nigeria, along with the global demand for sustainable and cost-effective refractory materials [3, 4], this study is both timely and essential. By evaluating the feasibility of Nigerian quartz rock for refractory applications, this research addresses a critical gap in existing literature and opens the door to localized, economically viable alternatives to imported materials. It also aligns with broader industrial goals of reducing dependency on traditional refractory inputs and leveraging locally sourced, high-performance raw materials for high-temperature manufacturing environments.

Materials and methods

Materials

The materials used for the development of refractory bricks were sourced and extracted from mining sites in Nigeria. The locations of the materials and functions are presented in Table 1.

Table 1: Materials used for refractory brick production.

S/N	Material	Use	Location
1	Quartzite	Production of refractory bricks	Ijero-Ekiti, Ekiti State
2	Bentonite	Binding material	Sango, Ogun State
3	Distilled Water	Plasticity and bonding	

Methods

Ground quartzite powder was mixed with 10% bentonite and 8% water to form a plastic paste. This mix was established in accordance to the optimal bentonite content and best water content for plasticity reported by [15, 16], respectively. The mixture was stirred until homogeneous using a ceramic blender, then molded into steel forms (50 × 50 × 50 mm), compacted by ramming and dried (at 100 °C, for 8 h) followed by firing (at 1200 °C, for 8 h). The mixture underwent vigorous clockwise manual stirring using a ceramic blunger to ensure homogeneity.

The refractory bricks were then produced through casting into oil-lubricated cubicle molds made from steel plates (50 x 50 x 50 mm). After the pastes were fully set in the molds, the product was compacted through ramming, as depicted in Fig. 1. Once the ramming process was concluded, molded or as-cast samples were removed from the molds. Then, moisture content was removed from these samples by drying in an oven set at 100 °C, for 8 h, and then they underwent heat treatment in a furnace for another 8 h, at 1200 °C [17].



Figure 1: Rammed refractory bricks.

Chemical composition

Chemical composition of the powdered raw samples was analyzed by X-ray Fluorescence (XRF) Phillips PW-1800 model. This technique relies on principles of quantum chemistry and atomic physics. During the process, the samples were exposed to the full range of photons, including principal radiation released from an X-ray tube. As a result, the elements in the samples emit secondary fluorescence with their characteristic X-ray line spectra. To detect and analyze this emission, a two-unit

system was used, consisting of the main channel simultaneous wavelength dispersive spectrometer, and a computer terminal for processing data and for control.

Thermogravimetric (TGA) and Differential Thermal (DTA) Analyses

To carry out TGA and DTA of the rock sample, 10 mg of the rock sample were prepared and placed in an aluminum pan. The sample in the pan was then heated gradually at a rate of 10 °C/min from room temperature up to 600 °C. As the sample was being heated, weight loss was measured under a nitrogen atmosphere flowing at 50 cm³/min. The heating of the sample occurred at the temperature range from 30 to 220 °C, at the rate of 10 °C/min, and then the reverse cooling from 220 back to 30 °C, at the same rate, and held for 3 min.

Loss on ignition (LOI)

LOI is commonly presented as a percentage of the difference in the weight of the sample before and after ignition. It represents the disparity between the sample's initial and final weight. Before firing a sample, the residual mechanical water is first eliminated by heating in an oven. The difference in weight between initial and final weight of the bricks after firing, expressed as a percentage, accounts for LOI.

Shrinkage test

Shrinkage test offers quantitative insights into the volume changes that may occur due to variations in water content during refractory brick manufacturing. To assess brick shrinkage, rectangular bricks, measuring 50 x 50 x 50 mm, were produced from identical rock samples through compaction via ramming. Each test sample had a slanting line (L₁) drawn across its diagonal. These samples were then subjected to a furnace, heated to 1000 °C. Post-firing, another line (L₂) was drawn across the diagonal to determine final length. The material's linear shrinkage was calculated using Eq. (1), following American Society for Testing and Materials (ASTM) guidelines [18].

$$\text{Shrinkage Test} = \frac{L_2 - L_1}{L_1} \times 100 \quad (1)$$

Apparent porosity

Conducting porosity assessment of the refractory bricks, each of them was immersed in a bath comprising water and oil. Soaking duration was set at 24 h, and the bath maintained a temperature of 100 °C. Following ASTM test standards, the apparent porosity for each sample brick was subsequently determined [19]. Estimation of apparent porosity (P_a) was observed using Eq. (2).

$$P_a = \frac{w_{sww} - w_{da}}{w_{sww} - w_{sw}} \times 100 \quad (2)$$

where w_{da}, w_{sw} and w_{sww} are the weight of the samples: dry in air, soaked in water and saturated by soaking in water, respectively.

Compressive stress at yield

Compressive yield stress represents maximum stress level at which the sample can be subjected to compression without undergoing plastic deformation. This stress limit indicates the point at which the material experiences squeezing forces, but does not exhibit permanent changes in its shape during the testing process. Compressive test was performed using a Universal Testing Machine (Instron), in accordance with [20], for cold crushing strength. Samples were oven-dried at 110 °C, and tested under displacement control at loading rates from 0.5 to 1 mm/min, with loading recorded continuously. Yield was identified as proportional limit and the point where the stress-strain curve first deviated from linearity, while also peak compressive strength, when failure occurred without a clear plastic region.

Water absorption (WA) test

Eq. (3) focuses on WA tests, crucial for evaluating the porosity and permeability characteristics of refractory materials. This test involves quantifying the water absorbed by a refractory material sample within a specified timeframe. Fired test samples underwent boiling for 24 h, at 100 °C, followed by a 4 h water immersion. WA was determined by calculating the weight difference before and after immersion, utilizing Eq. (3), as outlined in ASTM standards [19].

$$WA = \frac{W_s - W_d}{W_d} \times 100 \quad (3)$$

where W_s and W_d are sample's weight after boiling at 100°C and dry weight, respectively.

Microstructural analysis of the sample

The analysis of a material's microstructure involves the study of its small-scale internal arrangement, encompassing dimensions, morphology and spatial arrangement of its constituent particles or grains [21]. This investigative process, commonly carried out through optical or electron microscopy, is crucial for understanding the characteristics and performance of the material. Utilization of microstructural examination encompasses identifying defects or impurities within the material, aligning with methodologies advocated by [22, 23]. This approach facilitates the comprehensive assessment of material properties and behavior.

Morphological analysis was conducted using a Scanning Electron Microscope with energy dispersive X-ray spectroscopy (SEM-EDS) of model Phenom Prox, manufactured by Phenom World in Eindhoven, Netherlands. The prepared sample was examined under the SEM at 500× magnification. After scanning, elemental composition was calculated based on intensity of signals detected during EDS analysis, with each element's concentration expressed as percentage. Data were then used to produce a detailed image, which was saved for further study. Overall, SEM-

EDS proved to be an effective method for analyzing the surface composition of the sample, offering clear insights into the material's elemental structure.

Results and discussion

Chemical composition of quartz

Chemical composition of the quartz sample is presented in Table 2. Refractory materials characterized by a SiO₂ content exceeding 94.30 wt.% exhibit enhanced resistance to high temperatures, rendering them well-suited for applications requiring elevated temperature endurance. These materials possess superior refractory properties, making them ideal for use in environments where withstanding extreme temperatures is crucial [24, 25].

Table 2: Chemical composition of quartzite.

Component type	Conc. [wt.%]
SiO ₂	94.303
Fe ₂ O ₃	1.335
V ₂ O ₅	0.014
MnO	0.023
Cr ₂ O ₃	0.087
Co ₃ O ₄	0.012
NiO	0.002
CuO	0.040
Nb ₂ O ₃	0.005
MoO ₃	0.006
WO ₃	0.007
K ₂ O	0.148
CaO	0.307
SO ₃	0.553
Al ₂ O ₃	2.379
Ta ₂ O ₅	0.045
ZnO	0.002
Ag ₂ O	0.010
Cl	0.617
ZrO ₂	0.002
Rb ₂ O	0.003
BaO	0.097
SrO	0.002

While silica content (94.303%) was promising, the presence of a fluxing agent such as iron oxide (Fe₂O₃), at a concentration of 1.335 wt.%, was a concern. Fluxing agent tends to lowers melting point of silica. In silica refractories, this compromises refractoriness under load, a key property that indicates the material's ability to maintain shape and strength at high temperatures. A higher Fe₂O₃ content accelerates softening and deformation of the bricks when exposed to operational heat. Alkali oxides (K₂O = 0,148 wt.%, SO₃ = 0.553 wt.%) are recognized as among the most harmful impurities in silica refractories, due to the low melting eutectics with SiO₂,

which cause reduction in softening temperature and high temperature strength [26]. K_2O/Na_2O impaired slag resistance and dimensional stability at working temperature. Measured Fe_2O_3 and minor CaO , BaO and other fluxing oxides can further lower refractoriness through the formation of iron and alkaline earth silicate. Cl (0.617 wt.%) and SO_3 (0.553 wt.%) also present potential volatility and corrosion issues on firing. While quartzite's high SiO_2 is promising for silica refractory usage, alkali and flux impurity profile indicate that it is beneficial.

However, it is crucial to note that Al_2O_3 concentration in refractory materials exhibits a straightforward correlation with the material's resistance to high temperatures. Specifically, an increase in Al content leads to a proportional elevation in the material's resistance to extreme heat [24].

TGA and DTA

TGA and DTA results are presented in Fig. 2.

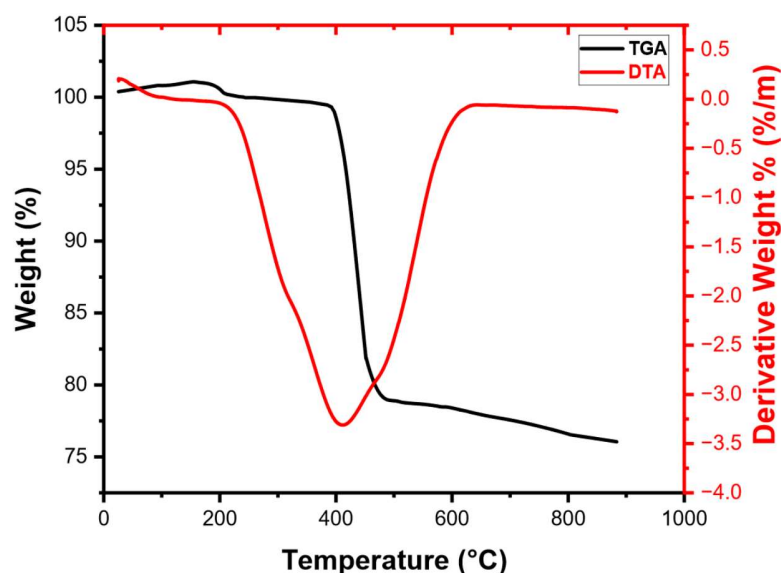


Figure 2: TGA and DTA analysis of brick sample.

Results revealed that the sample possesses desirable thermal qualities. TGA thermogram shows a single broad loss of 21%, which could be traced to the water molecule removal [26]. The first loss of absorbed and interlayer water happened from 200 to 300 °C, with possible combustion of organics, and then dihydroxylation of the clay that is endothermic occurred from 350 to 450 °C. Meanwhile, endothermic peak of DTA also indicates removal of last traces of OH^- in the form of H_2O , from 350 to 450 °C, which can exist up to 600 °C, according to [27, 28, 29]. This also aligns to the report of [30] that Aloji Fireclay mineral was dehydroxylated at 639.67 °C. The

outcome of the TGA and DTA analysis informed good thermal stability of the sample at high temperatures above 1000 °C.

Loss on ignition

As presented in Table 3, LOI, which is organic volatile matter loss during firing, has a direct effect on shrinkage value.

Table 3: Measured properties of the refractory brick (sample) compared to reference ranges.

S/N	Characteristics	Sample result	Literature ranges [33-35]
1	LOI	1.7%	0.5-3 %
2	Compressive stress at yield	2.70 MPa	1 – 20 MPa
3	WA	1.69847%	1 - 10%
4	Apparent porosity	26%	20-30%
5	Shrinkage	0.2%	0.3-3.5%

An average value of 1.70% was observed for LOI of refractory bricks sample (RBS). The outcome falls within the range of standard LOI values from 0.98 to 7.19%, as reported by [31]. Hence, measured LOI (0.5 %) is at the lower end of typical values, suggesting minimal organic or volatile content after firing. Literature reports optimal binder effects around 10%, which is widely accepted optimal proportion in literature, for achieving strong bonding and stable performance in refractory bricks.

Shrinkage

From the result in Table 3, it was observed that 10% bentonite binder content (BBC) in Refractory Bricks Sample (RBS) exhibited 0.2%. Therefore, although the effect of BBC was insignificant for RBS, it was observed to be significant for quartz's RBS. Hence, some researchers have suggested that lower linkage shrinkage value is preferable, with values below 4% deemed acceptable [31-32].

Apparent porosity

From the obtained result, apparent porosity increases using 10% bentonite binder for refractory bricks, with apparent porosity value (APV) of 26% observed [33]. Thus, it can be concluded that bentonite binder is of very good porosity, which has helped in maintaining overall APV of RBS under examination, presenting them to be favorable for lightweight insulation.

Compressive stress at yield

From the obtained result, compressive stress at yield (2.70 MPa) was the result of incorporating a 10% binder into the quartz sample. Consequently, data analysis

suggests a noteworthy trend. Compressive stress at yield of samples rose proportionally with an increase in binding content. This indicates the samples have enhanced capacity to endure stress before experiencing failure or deformation [34].

Water absorption (WA)

WA is a crucial parameter, calculated as the sample's absorbed water weight divided by its dry weight. Refractory brick samples revealed WA value of 1.69847%. This metric is vital in evaluating the material's porosity and its potential susceptibility to environmental factors. The observed percentage indicates the amount of water the sample can absorb, influencing its overall durability and performance in various applications. Understanding and controlling WA is essential for optimizing the material's resistance to environmental conditions, ensuring long-term stability in practical applications [33].

Scanning Electron Microscopy-Energy Dispersive X-ray Spectroscopy

SEM micrograph in Fig. 3 reveals a granular, unevenly fused microstructure typical of a refractory brick predominantly made from quartz [33].

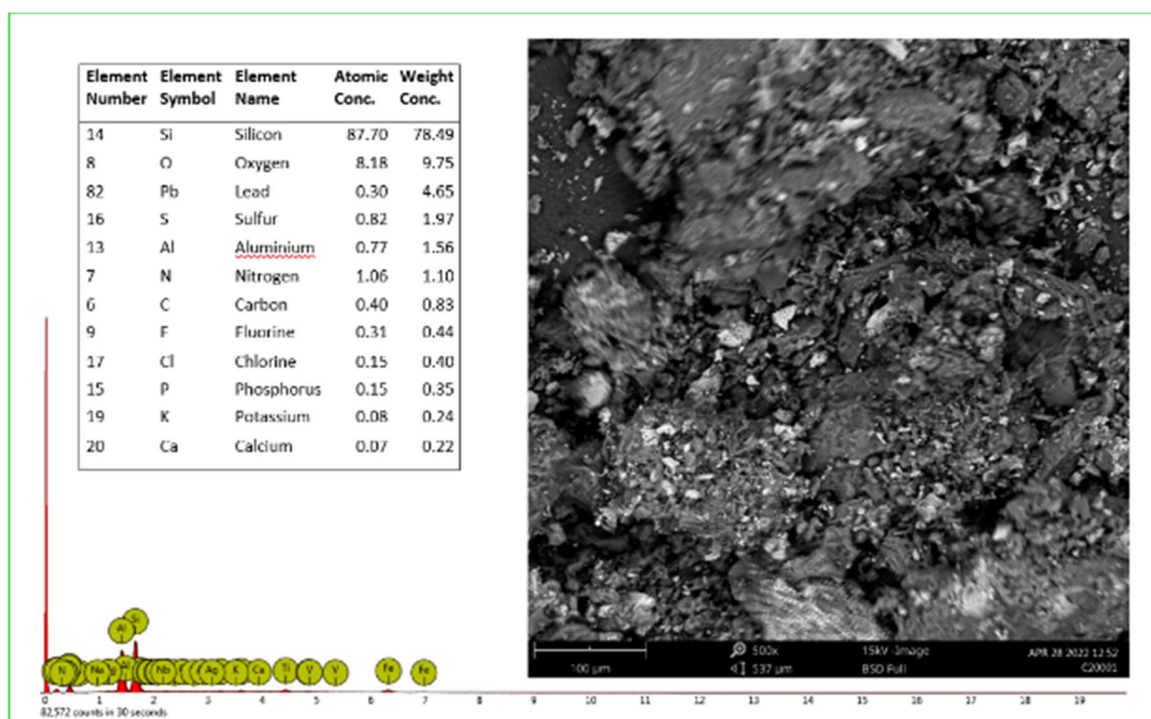


Figure 3: SEM and EDS analysis of the brick sample.

The presence of these sharp, well-defined grains suggests that the material may have undergone moderate sintering, without complete vitrification or densification. Bright

spots visible in the backscattered electron image suggest the presence of elements with higher atomic numbers, potentially corresponding to minor or trace elements like lead or aluminum. The distribution of these bright regions appears scattered, indicating localized concentrations rather than uniform dispersion. Accompanying EDS data confirms that silicon is by far the dominant element, with an atomic concentration of 87.7% and a weight concentration of 78.49%. This is entirely consistent with the brick being primarily composed of SiO₂. Oxygen, as expected, is also present in substantial amounts (9.75% by weight), supporting the conclusion that silica is the base material [34-35].

What stands out, however, is the presence of several trace elements that hint at contamination. Lead (Pb), although only present at 0.3% atomic concentration, contributes a surprisingly high 4.65% by weight, raising potential concerns. Lead is not a typical ingredient in refractory compositions, and its presence could affect the material's performance, especially at elevated temperatures, not to mention its environmental and health implications. Other detected elements such as S (1.97% by weight), Al (1.56%), N, and small traces of C may result from impurities in raw materials or from the firing environment. Aluminum may contribute to forming aluminosilicate phases, which could improve thermal stability, but the concentration here is relatively low [36-39].

Conclusion

In summary, quartzite-based brick showed very high silica content and low LOI, indicating good thermal stability. Its high apparent porosity and moderate compressive strength are consistent with an insulating refractory. WA was low (1.7%), supporting suitability for high-temperature use. A noteworthy finding is the presence of trace lead in SEM/EDS analysis, which may require further investigation. Overall, these results suggest local quartzite is promising for silica refractories, but further work (e.g., optimizing binder content, improving purity) is needed. Compressive yield strength (1.7 MPa) is comparable to lightweight silica bricks, but relatively low, which limits structural load capacity. Nonetheless, it indicates the brick can survive minor compressive stress before failure. These findings are especially relevant for Nigeria, where developing high quality silica refractories from abundant local quartzite could reduce dependence on costly imports. Addressing issues like trace lead and optimizing binder and purity levels would enhance the economic value and industrial potential of Nigerian quartz deposits.

LOI findings indicate that bricks have good thermal resistance. Compressive stress at yield (MPa) of the quartz sample was 1.3525, signifying its ability to withstand stress before failure or deformation. From the result, it was observed that 10% BBS in RBS exhibited 0.2% for shrinkage test. Also, when using 10% bentonite binder for refractory bricks, APV of 46% was observed. WA analysis shows that, for refractory

brick samples, quartz was 1.69847%. SEM and EDS analyses confirmed that the brick is primarily quartz-based, with a structure that supports high-temperature resistance. Some fluxing elements were found to be elements that provide good thermal and chemical stability, which is in line with the outcome of TGA and DTA analyses. However, partial sintering, combined with the presence of potentially harmful elements like lead, points to areas where formulation or processing could be optimized.

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Authors' contributions

O. B. Ogundipe: contributed to conceptualization, experimental investigation, data analysis; prepared the original version, revising and editing. **B. V. Omidiji and H. A. Owolabi:** contributed to conceptualization and revising. **P. P. Ikubanni, O. S. Adesina and R. A. Adebayo:** contributed to experimental studies and data analysis. **J. B. Ajewole, T. S. Olabamiji and B. O. Abolarinwa:** contributed to methodology, material sourcing and editing. **A. A. Salawudeen:** contributed to manuscript revision, reference editing and improvement of the presentation. All authors reviewed and approved the final manuscript.

Abbreviations

APV: Apparent Porosity Value
ASTM: American Society for Testing and Materials
DTA: Differential Thermal Analysis
EDS: Energy Dispersive X-ray Spectroscopy
LOI: Loss on Ignition
RBS: Refractory Bricks Sample
SEM: Scanning Electron Microscopy
SiO₂: Silicon Dioxide
TGA: Thermogravimetric Analysis
WA: Water absorption

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