Performance Evaluation of Refractory Bricks Produced from Ijero-Ekiti Quartz

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Abstract: This study carries out physico-mechanical and thermal analyses of a produced bricks from Ijero-Ekiti Quartz. This was with the view to investigate the utilization of Ijero-Ekiti Quartz for the development of insulating refractory bricks. The rock samples were collected from the extraction site in Ijero-Ekiti, dried and then grinded into fine powders and then produced into bricks by the addition of selected binder and water to form moldable homogeneous plastic paste. The resulting bricks were dried and then sintered at a temperature of 1200 oC. Analysis were carried out on apparent porosity, Water Absorption (WA), bulk density, shrinkage test, cold crushing strength (CCS), Loss on Ignition (LOI) and Microstructural Examination using Scanning Electron Microscope Energy Dispersive X-ray Spectroscopy (SEM-EDS) to investigate the refractory performance of the produced bricks. The apparent porosity of 43%, water absorption of 0.98%, bulk density of 1644 kg/m3, shrinkage value of 1.71%, cold crushing strength of 2.165 MPa, Loss on Ignition of 0.98% and Refractoriness 1571.68 oC gotten from the tested bricks were found to be within standard values for refractories. The Microstructural image also revealed homogeneity and the spread of silicon round the brick structure which is desired for refractory bricks as it indicates consistent properties and good thermal and mechanical properties.

Keywords: Quartz; Refractory Bricks; Refractoriness; Rock Minerals; Refractory Properties; SEM-EDS.

Received: April 12, 2022. Revised: July 15, 2023. Accepted: August 21, 2023. Published: September 19, 2023.

1. Introduction

Refractories are solid materials that, even when in contact with corrosive gases or liquids, can keep their mechanical performance and resist high temperatures for the needed period. For all high temperature operations, including the manufacturing of metals, ceramics, glass, and cement, refractories are essential (Horckmans *et al.,* 2019).

According to Elngar *et al* (2009), refractories are substances that does not melt at 1580 degrees Celsius. They are slag resistant to cracking and splitting under different temperatures, chemically inert, strong enough to endure the weight and wear of the melted material, and easily moldable into bricks or other types of construction materials. Since the 1960s, refractories have stood as an "essential element" in heat engineering facilities where they have been employed to increase productivity and energy efficiency. Refractories are physically and chemically stable at high temperatures, and robust to thermal shock depending on the operating environment (Atanda *et al* 2012).

The shrinkage after firing a fireclay should be no more than 6-8%, and good fireclay refractories should always be 24-26% plastic. Moreover, a good fireclay refractory shouldn't have more than 25% Fe_2O_3. The fireclay refractory bricks are the most prevalent and widely utilized in all locations where heat is generated due to its ease of supply and relative affordability of the material. To contain hot environments and protect structural parts from high temperatures, fireclay bricks are primarily utilized in furnace building. (Atanda *et al.*, 2012)

Strength is typically not a major factor for a refractory bricks because they are not frequently used to sustain structural loads. The end product's stability and accuracy of dimensions are typically subject to some level of standards. High temperatures generally refer to temperatures over 1000°C or to those at which ordinary metals cannot be used due to melting or oxidation. Refractory materials, which are often nonmetallic, are used to construct furnaces, incinerators, metallurgical furnaces, and insulation. Consequently, a refractory material is a substance used in applications requiring severe heat resistance, such as furnace linings, that will maintain its structure, strength, and chemical identity while exposed to high temperatures.

Esezobor *et al.*, (2015) demonstrated that the use of locally available raw materials yields a high-quality refractory brick factory in Nigeria, capable of producing 200,000 bricks per annum at N1,203.66 per brick as opposed to the 3-years projection of N1 800 of the imported bricks. More importantly, the period of payback for the capital investment was approximately 2 years which will definitely attract investors. Olaiya *et al.*, (2019), conducted a comprehensive Strength, Weakness, Opportunities and Threat (SWOT) analysis on the potential for market opportunities in Nigeria for producing refractory bricks for foundry furnaces by exploiting locally available raw materials which are usually sourced from overseas. Upon the completion of their analysis, they were able to establish that the production of refractory lining materials can flourish in Nigeria as the needed raw materials are widely available and the process technology required is also readily available. In addition, the available market in Nigeria is vast as the foundry industry, which is the largest consumer of this product, is growing progressively.

2. Materials and Methods

2.1 Materials

The materials used for this study is presented in Table 1

Table 1 Materials

S/N	MATERIAL	SOURCE	USE
1	Quartzite	Ijero-Ekiti, Ekiti State, Nigeria	Production of Refractory Bricks
2	Bentonite	Sango, Ogun State, Nigeria	Binding Material
3	Distilled Water		For plasticity and bonding

2.2 Methods

The rock samples were collected from the extraction site in Ijero-Ekiti with the aid of a digger, shovel, and storage container. Figure 1 show an overview image of the site where the sample was collected. Thereafter, the as-collected samples were sun-dried for 3 days to remove moisture content and as well facilitate easy grinding (Moorkah and Abolarin, 2005; Ogundipe *et al*., 2021). The rock sample was mechanically milled (in a laboratory ball mill) into fine powders for further analyses. To ensure complete evaporation of the unbounded moisture content, the powders were heat-treated at 110℃ for 1 hour in an oven. The choice of bentonite as the binder was inspired considering its good thermal stability (Krajewski, *et al*. 2015).

The production of the refractory bricks commenced with conversion of each rock sample's finer powders into a homogeneous plastic paste. This was achieved by adding a 5% binder (bentonite) and 8% water. The binder is added in order to impart green strength and also act as a plastifier (Moorkah and Abolarin, 2005). The mixture was then mechanically stirred vigorously with the aid of a ceramic blunger in a clockwise direction for homogenization. According to Nnuka and Agbo (2000), 8% water is the optimum quantity needed for optimum plasticity. The resulting paste was then poured into the 50 x 50 x 50 mm mould made from steel plate. The as-cast samples were then sintered in a furnace at 100 ℃ for every 10 minutes until the firing temperature reaches 1200℃. The samples were then held at 1200 ℃ for 8 hours and then allow to cool in the furnace for 24 hours (Osarenmwinda and Abel 2014). Different analyses to determine the mechanical properties and the thermal stability of the produced bricks were conducted.

Figure 1: Images of Ijero-Ekiti Mining site

2.2.1 Apparent porosity

The porosity of the produced refractory bricks was determined by immersing the brick in a soaking bath containing water and oil. The immersion time was set to 24 hours and the bath temperature was maintained at 100 ℃. The apparent porosity of each brick samples was measured in compliance with ASTM C20-00 (2015) test standard. Thus, the apparent porosity was estimated as follows;

$$Apparent\ porosity\ (p_a) = \frac{w_{sww} - w_{da}}{w_{sww} - w_{sw}} \times 100 \quad (1)$$

where $w_{sww}, w_{da},$ and w_{sw} are saturated weight of the soaked sample in water, dry weight of the sample in air, and weight of the sample soaked in water.

2.2.2 Bulk Density Measurement of the Bricks

The bulk density of each brick sample was then calculated as follows:

$$Bulk\ Density\ (B_d) = \frac{w_{da}}{w_{sww} - w_{sw}} \quad (2)$$

2.2.3 Shrinkage Test of the Bricks

The shrinkage test is important in the sense that it helps to indicate quantitatively how much volume change can occur with changes in water content during the production of refractory bricks. To determine the shrinkage of the produced bricks, a slanted line of length was drawn across the diagonal of each test sample and designated as l_1. The test samples were later transferred to the furnace and fired to 1000 ℃. To get the final length of each test sample after firing, another line was drawn across the diagonal of the fired test sample and designated as l_2. Thus, the linear shrinkage of the material was evaluated according to Equation 3 as given by the ASTM C596 – 18 test standard (ASTM C596-18, 2018).

$$Lineear\ Shrinkage = \frac{l_1 - l_2}{l_1} X\ 100\% \quad (3)$$

2.2.4 Cold Crushing Strength Test of the Bricks

Cold crushing strength (CCS) represents the ability of a product to resist failure under compressive stress at room temperature, that is, the maximum load per unit area, applied under specified conditions at room temperature, that a refractory material will endure prior to the occurrence of failure. CCS is usually used to determine the mechanical strength of refractory materials. The CCS of each brick was estimated on a universal testing machine in accordance with standards (ASTM C133 - 97, 2021).

2.2.5 Loss on Ignition Test of the Bricks

Loss of Ignition (LOI) is a prevalently methods used for quantifying the organic matter content in soils, however, there is no universal standard protocol to be followed. Several factors may affect its accuracy, such as the type of the furnace, duration and temperature of ignition, the mass of the sample, and nature of the samples (Tomeczek and Suwak, 2002). The LOI is often expressed as a percentage dependent on either the unignited sample weight or the ignited sample weight. It is the difference between the sample's initial weight and its weight after ignition. The mechanical water that was left over after drying is first driven off before heating a sample. The weight decreases on the total weight of the prepared brick samples, expressed as a percentage, is the LOI in this study. Hence, the weight loss by each brick was calculated as their weight difference before and after firing, and consequently, the LOI at 1200°C was determined according to equation 4 as described in ASTM D7348 – 13(2013) test standards.

$$LOI = \frac{W_1 - W_2}{W_1} \times 100 \tag{4}$$

where W_1 is the initial weight of the brick sample before firing, and W_2 is the final weight of the brick sample after firing. The LOI of the brick samples was estimated in accordance with (ASTM D7348-13, 2013).

2.2.6 Determination of the Refractoriness of the Bricks

The refractoriness of a material is a measure of its capability to survive exposure to high temperatures without undergoing any notable deformation. In other words, it is the measurement a refractory material's fusibility which shows where the material loses its rigidity. In this research, the sample bricks refractoriness was determined by Shuen's formula (Odewale and Alamaka 2019). To determine the refractoriness of a refractory brick using Shuen's formula, the content of alumina and the content of other oxides besides SiO_2 in the refractory brick were determined. This was done through chemical analysis of the brick sample using the X-ray X-ray fluorescence Spectroscopy (XRF). The refractoriness of bricks was then calculated using the following formula:

$$K = \frac{360 + Al_2O_3 - RO}{0.228} \tag{5}$$

Where, K is the Refractoriness (°C), Al_2O_3 is the Alumina Content in the refractory, and RO is the sum of all the oxides beside SiO_2 in the sample

2.2.7 Microstructural Examination of the Bricks

The structural morphology of the brick sample was examined with the aid of a Scanning Electron Microscope energy dispersive X-ray spectroscopy (SEM-EDS) Phenom Prox model with an accelerating voltage of 15 kV. The test sample was mounted on aluminium stubs and sputter-coated prior to the SEM examination. The emissions of X-ray were analyzed using an energy dispersive detector, which discriminates among X-ray energies. The molar concentration in % was calculated after the intensity of each element present was determined by the EDS scan, thereafter the image was saved. The Scanning Electron Microscope Energy Dispersive X-ray spectroscopy (SEM-EDS) is a useful method for analyzing the composition of the surface of a specimen

3. Result and Discussion

3.1 Bulk Density

The bulk density offers a broad indication of the quality of the refractory bricks, the higher bulk density (low porosity) refractories are thought to be of superior quality. This is due to the fact that a greater bulk density improves the stability of the volume, heat capacity, and resistance to abrasion and slag penetration (Kipsanai *et al.,* 2017). The bulk density value of the sample was calculated to be 1644 kg/m^3 as presented in Table 2. Unfortunately, this value is lower than the typical bulk density value for fireclay refractories which should be 1910 kg/m^3 according to Krishnamurthy (2022). Nonetheless, the value is close to the range of 1700 to 2300 kg/m^3 for refractories clay as indicated by (Abuh *et al*., 2014; Oyelaran, 2014; Ojonimi *et al*., 2016). A similar value of 1640 kg/m^3 has been reported for Egbahieme clay with similar composition by Ojonimi *et al*., (2016) and Borisade *et al*., (2021).

3.2 Apparent Porosity

A decreased porosity can have a negative impact on thermal shock resistance while improving strength, load carrying capacity, and corrosion resistance (by less slag penetration). The pores that are found in highly porous brick prevent cracks from forming. Because of this, the degree of porosity is a trade-off considered is depending on the particular service situation(s). The Apparent Porosity Value (APV) gotten for the brick sample is 43 %.

A similar finding was reported by Obidiegwu *et al*., (2014) when they investigated the stability of steel slag as a refractory material using bentonite and boric acid as binders (Abubakar *et al*., 2014). Although, in comparison with standard siliceous fireclay, the observed APVs for the RBS is higher than the acceptable APVs (20-30 %), the use of binders with more closely packed particles in the RBSs and an appropriate temperature-time treatment is recommended to take care of the issue (Nwajagu, 2005). A study similar to this involved the utilization of sawdust as an additive which resulted in apparent porosity values of 30.23%, 39.5%, 45.45% and 46.15%, respectively. Comparison with the control sample revealed an improvement in apparent porosity value of up to 200% between 5% to 20% sawdust additive composition. Moreover, an increase in additive composition was observed to correspond to an increase in porosity value. These findings demonstrate that the fired bricks' apparent porosity values vary based on the quantity of additives utilized, with a percentage improvement in porosity ranging from 104% to 199% for rice husk additive compositions of 5% to 20%. (Safeer *et al*., 2017).

3.3 Linear Firing Shrinkage

The Linear Shrinkage Value (LSV) of 1.71 % was obtained but was lower than the recommended standard LSVs in the range of 4 – 10 %. However, some researchers have reported that lower LSVs are more desirable and values below 4 % would be acceptable. This is an indication that the produced RBSs would be less susceptible to volumetric shrinkage. Moreover, the observed low LSVs for the RBSs can be attributed to the ability of the bentonite binder to confer dimensional stability on the RBSs as it does not shrink as much as the rock samples under temperature-time treatment. A similar finding can be found in the work of Obidiegwu *et al* (2014). N'Jock et al. (2013) also attributed the low value of LSV (1.01 %) obtained for Ozanagogo clay to the better thermal stability and anti-shrinkage property of grog, which is 90 % of their brick composition.

3.4 Loss on Ignition Result

The Loss on Ignition (LOI) is the organic volatile matter loss on heating and this affects the values of shrinkage and porosity. The LOI value 0.98 % was observed for the RBSs in this study and is similar to the findings of Shuaib-Babata *et al.,* (2018) where they studied fireclays from Baruteen Local Government Area of Kwara State, Nigeria investigated for refractory applications. This presents the amount of moisture the RBS could hold or the percentage of weight reduction of the sample (Osarenmwinda and Abel, 2014). The low LOI value obtained in this investigation suggests that the RBS contains a minimal amount of organic and/or hydrated components.

3.5 Cold Crushing Strength

As presented in Table 2, the CCS for the RBS is 2.165 MPa which is the maximum amount of stress that the brick can withstand before it fails. This value falls within the compressive strength standard values as recommended by Arthur and Gikunoo (2020) which is between the range of 0.981–6.867 MPa. Low strength of the bricks can be attributed to the high porosity of the brick sample.

3.6 Water Absorption (WA)

Water absorption (WA) is a measure of the amount of water the brick sample can take up. From table 2, the WA for the RBS is 0.98% which is relatively low compared to the study by Ajala and Badarulzaman (2016). Although, the specific effects of WA on refractory materials depend on the type of refractory and the conditions in which it is used. WA is an important property of refractories as it can affect their strength, durability, and resistance to thermal shock. The higher the water absorption of a refractory, the more porous it is and the more susceptible it is to damage from thermal cycling and other stresses. Conversely, lower water absorption means a denser and stronger refractory that can withstand higher temperatures and more severe conditions. Researches have however suggested that WA of less than 1% are considered low absorption, while those with more than 6% are considered high absorption (Cheng and Zhang, 2010).

3.7 Refractoriness

The chemical compositions of the refractory material as analyzed by the XRF is presented in table 3. The ability of refractory bricks to withstand high temperatures without cracking, melting, or degrading is critical to the performance and longevity of these industrial applications. Refractoriness is the ability of the refractory bricks, to withstand high temperatures without undergoing significant physical or chemical changes. As presented in the table 2, the refractoriness of the RBS is gotten to be 1571.68 ºC which is similar to the 1550 ºC gotten from Egyptian magnesite by Ewais *et al.* (2018). In general, the accepted range of refractoriness is typically above 1,500°C (Acar, 2020; Olaiya *et al*. 2015; ASTM C71-18, 2018).

Table 2: Refractory Properties of Ijero-Ekiti Quartz Brick

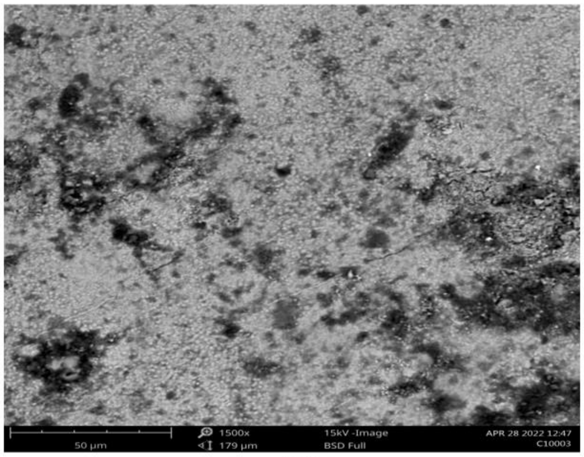
Properties	**Ijero-Ekiti Quartz Brick**	**International Standard**
Bulk Density	1644 kg/m^3	300 to 3200 kg/m^3
Apparent Porosity	43 %	20-30 %
Linear Firing Shrinkage	1.71 %	0.3-2.5 %
Loss on Ignition	0.98 %	0.5-3 %
Cold Crushing Strength	2.165 MPa	0.981–6.867 Mpa
Water Absorption	0.98 %	1 to 10%
Refractoriness	1571.68 ^oC	> 1300 ^oC

Table 3: Chemical Composition of Ijero-Ekiti Quartz gotten from XRF

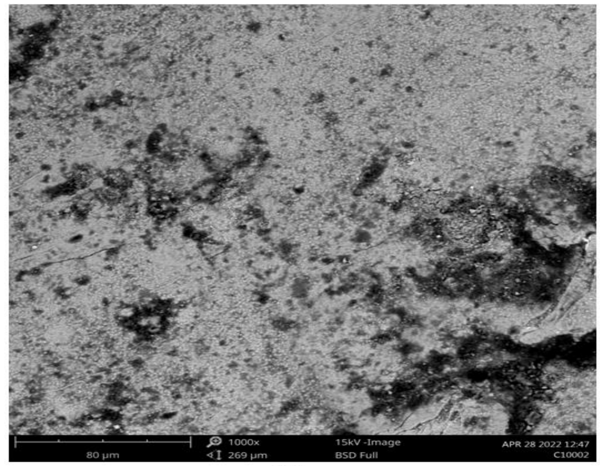
Component Type	SiO_2	Al_2O_3	Fe_2O_3	SO_3	CaO	K_2O	TiO_2	Others
Conc (wt.%)	92.15	3.09	2.53	0.53	0.31	0.22	0.15	1.02

3.7 SEM/EDS

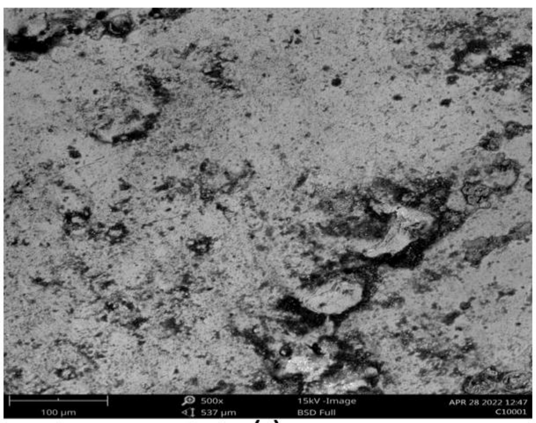
From Figure 2 presenting the SEM and EDS of the sample, of the RBS he microtextural arrangement consists of several ring layers (rims) indicative of reactions occurring in different steps. The inner part of the brick is a relic of the original refractory material and is surrounded by a first and a second rim characterized by needle-like silicon crystallizations into different and complex matrixes. From these in relation to its EDS result presented alongside, silicon is discovered to have the highest atomic concentration and weight concentration, followed by Aluminum, iron, carbon, oxygen, titanium etc. as the case may be and in proportion to the percentage of the applied bonding agent. The concentration and the distribution of silicon throughout the bricks contributes to the overall strength of the refractory bricks (Gado *et al*., 2020). Figure 2: SEM Images of the quartz brick at magnification (a) x1500 (b) x1000 (c) x500 and (d) its EDS analysis



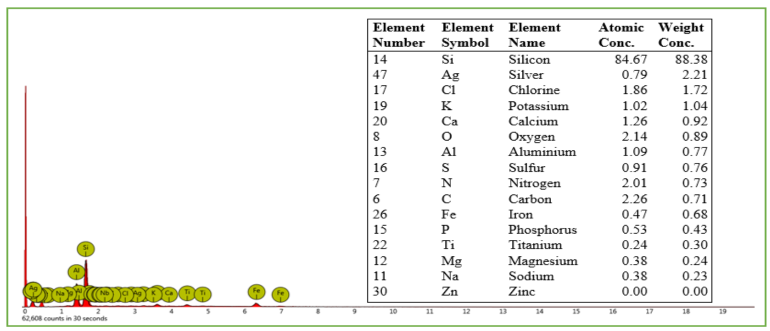
(a)



(b)



(c)



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	84.67	88.38
47	Ag	Silver	0.79	2.21
17	Cl	Chlorine	1.86	1.72
19	K	Potassium	1.02	1.04
20	Ca	Calcium	1.26	0.92
8	O	Oxygen	2.14	0.89
13	Al	Aluminium	1.09	0.77
16	S	Sulfur	0.91	0.76
7	N	Nitrogen	2.01	0.73
6	C	Carbon	2.26	0.71
26	Fe	Iron	0.47	0.68
15	P	Phosphorus	0.53	0.43
22	Ti	Titanium	0.24	0.30
12	Mg	Magnesium	0.38	0.24
11	Na	Sodium	0.38	0.23
30	Zn	Zinc	0.00	0.00

(d)

4. Conclusion

The bulk density and porosity measurement conducted in this study provides important information about the compactness and packing of the quartz sample as refractory materials. Both results showed that the refractory bricks were in the acceptable range for refractory materials. This suggests that the use of bentonite as a binder helps to improve the mechanical stability of the produced bricks.

The linear firing shrinkage and Loss on Ignition measurement provided important information about the dimensional stability and thermal expansion behavior of quartz brick. The results showed that the linear firing shrinkage of these refractory materials was in the range of acceptable percent. The results demonstrated the potential of this combination of materials as a promising option for high-temperature applications in the industry.

The study showed that the cold crushing strength carried out on the refractory material suggest that the materials could be a promising candidate for high-temperature applications in the industrial refractory furnace, as high cold crushing strength is essential for ensuring the structural integrity of refractory materials under stress. Also, the water absorption results of the study showed that RBS is of low water absorption. The homogeneity of the brick crystals and the presence of high silicon as presented in the SEM/EDS result indicate that the possess high tendency to resist thermal shock and the brick's refractoriness backed this up.

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