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Research Article

Production and Characterisation of Refractory Bricks for Cement Kiln Burning Zone Application

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Abstract

Cement clinker is produced in a rotary kiln at a sintering temperature of 1450 ℃. Because of this temperature, the kiln body is lined with refractory bricks. The failure of these refractory bricks is a challenge to cement producers because of its high impact on production and cost. Today, Nigerian cement factories depend on expensive imported refractories to survive. This work addresses this challenge by producing quality and less expensive refractory from locally sourced materials. The refractory materials were selected based on the thermal load for kiln burning zone, the material composition, particle size distribution and purity and refractory properties following ASTM C133 standard. The bricks were produced using compression moulding techniques at 125 MPa. The results showed that the thermal load for the kiln burning zone is 14.43 GJ/m^2hr. Dolomite and zirconia material characterized using XRF and SEM-EDX reveals that its composition, particle size distribution and purity meet the requirement for cement kiln burning zone refractory material. The absence of K_2O, SO_3 and Na_2O in the composition makes it suitable for cement kiln refractory application. The properties of the refractory analysed based on ASTM C133 standard showed that the optimum value for apparent porosity, bulk density, cold crushing strength, modulus of rupture, thermal conductivity, refractoriness and refractoriness under load were 16%, 3.42g/cm^3, 95.89 MPa, 15.14 MPa, 1.56 W/mK, 1800 ℃ and 1648 ℃ respectively. Thermal shock resistance of 30 cycles at 1500 ℃ and 1600 ℃. These properties were better off compared to the commercial refractories with the following properties apparent porosity, bulk density, cold crushing strength, modulus of rupture, thermal conductivity, refractoriness and refractoriness under load were 17%, 3.2g/cm^3, 90.0 MPa, 14 MPa, 2.1 W/mK, 1800 ℃, 1350 ℃ and 30 cycles at 1500 ℃. This means they have better performance and longer service life. The incorporation of zirconia in the bricks increases its wear resistance, stability at high temperature and better coating index. This concludes that the locally produced refractory addresses the concern of cement producers and presents a good opportunity for investors in the area of refractory bricks production in Nigeria.

Keywords

Cement Clinker, Cement Kiln, Burning Zone, Refractory Properties, Dolomite, Zirconia, Thermal Load

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1. Introduction

Cement clinker is produced by heating a finely ground and carefully blended mixture of the raw material, mainly limestone or shale, clay, sand and iron to a temperature up to 1450 ℃ in a rotary kiln [1]. The rotary kiln is a steel tube, slopes slightly and slowly rotates on its axis using variable speed drive between 0.5 and 4.2 rpm. The kiln body is lined with refractory bricks to protect it from high temperatures, hot and corrosive clinker melt and chemical attacks. Depending on the nature of the chemical reaction, material and temperature inside the cement kiln, the kiln is divided into zones as presented in Figure 1. The highest temperature zone is the sintering (burning) zone [2]. At this zone, the temperature reaches 1450 ℃. in some kilns, the sintering temperature reaches 1600 ℃.

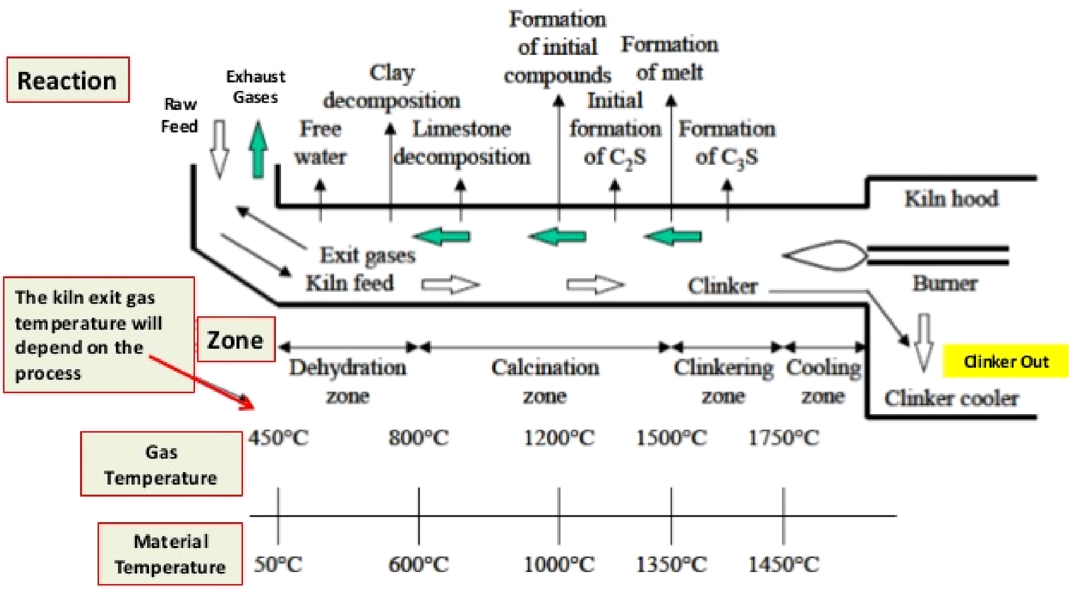

Figure 1. *Diagram of dry process kiln showing the main zones (Rongfeng et al.; 2021).*

The failure of the refractory bricks especially in the burning zone is a problem for many cement industries because each failure results in poor performance due production loss and increase in production cost (incurred cost to get the kiln back to its normal operation). For instance, the cost of relining one metre of 4.0 m diameter kiln is above 8.83 million Naira (USD 17,600). This amount represents only the cost of the refractory. The cost of installation, the hidden cost associated with loss in production, and the extra fuel required for pre-heating and maintaining the kiln temperature are not included.

To achieve good refractory life in cement kilns, especially at the burning zone, the refractory must have good hot strength, resistance to abrasion, compatible chemical composition, and sound thermal characteristics. In addition, it must have the ability to:

Protect the steel shell against heat. Material and gas temperature inside the rotary kiln surpass the maximum working temperature recommended for carbon steel. Without refractories the kiln shell would be destroyed by heat. As a result, as soon as the refractory lining fails, the kiln must be shut down for lining repair. The overheated areas on the kiln shell are commonly known as “hot spots” or “red spots” as shown in Figure 2.

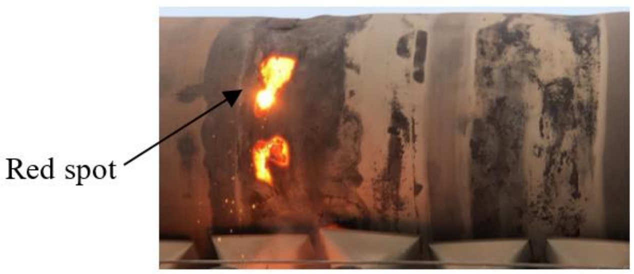

Figure 2. *Refractory failure that created a red spot on the kiln shell.*

1. protect the kiln shell against abrasion.
2. minimize heat loss through the kiln shell. Part of the heat supplied to the kiln system is lost as radiation through the steel shell.
3. control the flow of material through the kiln. The kiln load travels under the combined action of kiln rotation and slope. Cam linings, dams, tumblers, and trefoils in the kiln oppose material flow allowing some control of material residence time.

4. promote heat, transfer to the kiln load. Tumblers, trefoils, and profiled linings induce material tumbling and mixing, which in turn promote heat transfer from gas to solids, from refractory to load, and within the load itself through agitation and surface renewal as presented in figure 3.

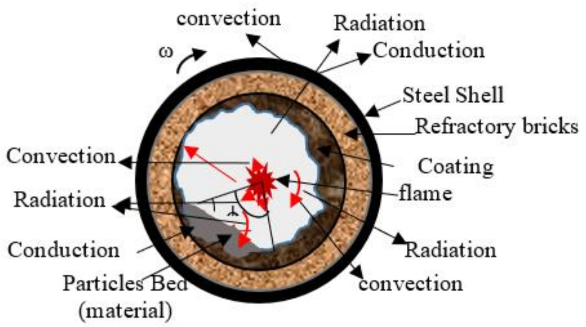

Figure 3. *Heat transfer phases within the rotary cement kiln.*

2. Cement Kiln Burning Zone Refractory

2.1. Sintering Zone of a Cement Kiln

The sintering zone of a cement kiln is a high-temperature region where the raw materials undergo significant chemical and physical changes to form clinker. The refractories used in this zone must possess certain critical properties to withstand the harsh conditions and ensure optimal kiln performance. The cement kiln sintering zone refractories should withstand temperatures up to 1650 ℃ without losing structural integrity or deteriorating, resist corrosion from alkaline compounds, such as K_2O and Na_2O, and acidic compounds like SO_3, endure sudden changes in temperature without cracking or shattering, maintain strength and durability under high temperatures and mechanical stress, minimize heat transfer to reduce energy losses and protect the kiln shell, resist wear from moving clinker and kiln debris, maintain their shape and size under high temperatures and chemical attacks and withstand the high temperatures without deforming or softening.

Common refractories used in the sintering zone include Magnesia-chrome bricks, Alumina-chrome bricks, Spinel bricks and Monolithic refractories (example castable and gunning mixes). These refractories are carefully selected and designed to meet the demanding conditions in the sintering zone, ensuring optimal kiln performance, energy efficiency, and clinker quality.

Studies done involving the improvement of cement kiln burning zone refractory bricks to improve their service life prove there is room for improvement of refractory properties to prolong it life span. Burning zone refractory was changed from fire clay and alumina bricks to basic bricks in the late 80’s to improve its performance and life span as reported by [3, 4]. First trials were carried out with magnesium and magnesia chromite bricks in the burning zone. Magnesia chromite bricks became the standard basic brick in the hot section of the kiln, while magnesia bricks exhibited bad performance due to their poor structural flexibility [4]. Magnesia chromite bricks were later rejected because of their environmental impact which resulted in an increasing demand for chromite-free basic bricks despite their excellent performance [4]. Dolomite bricks were later introduced in the burning zone but exhibited similar positive results as magnesia chromite bricks in moderate climatic environmental conditions but the bricks are sensitive to CO_2, sulphur, and humidity, which affects their performance in hot climatic zones or when secondary fuels are used [3, 5, 6]. Magnesia spinel brick grades, sintered spinel, in situ spinel, fused spinel, magnesia hercynite bricks, and magnesia pleonastic bricks have been developed for rotary kilns burning zone and high loaded areas like the tire area. The performance of these bricks does not perfectly meet the cement kiln burning zone requirement [3]. For this reason, more studies and improvement are required.

To achieve a successful refractory lining in the sintering zone, a rigorous study must be performed taking into consideration all constraints throughout the production with regard to the parameters governing the process of choosing the correct refractory for the zone. For the cement kiln burning zone, thermal load determination is one crucial parameter that decides the lining types, property and quality requirement.

2.1.1. Cement Kiln Sintering Zone Thermal Load

With increasing production per net kiln volume (ton/dm^2), thermal load increases, and lining material must be designed accordingly to be able to take up that thermal load. From experience, the normal thermal load of cement kilns remains in the range 14 -22 GJ/m^{2}h, whereas the recommended maximum thermal load in the burning zone is 24.3 GJ/m^{2}h. Equation (1) is used to determine the thermal load for the kiln burning zone.

$$T_l = \frac{5.24\times10^{-6}F\times N}{D^2} \tag{1}$$

Where: F = fuel burning rate per hour (kg/hr),

N = net calorific value of the fuel (kcal/kg), and

D = effective internal diameter of the kiln after Refractory lining.

For large diameter kilns where the thermal loading is high and alternate fuels are used, the basic refractories are preferred over high alumina products, the raw material chemistry, fuel used, and operational discipline play a major role in deciding the refractory lining life in the burning zone. The selection of the refractory for the burning zone depends upon the kind of stresses the refractory lining is exposed to. The operating conditions that prevail in different cement plants are not the same.

2.1.2. Zirconia-Containing Dolomite Bricks

Zirconia-containing dolomite bricks are made by adding a small amount of ZrO_2 when manufacturing dolomite bricks. ZrO_2 forms a zirconate with the lime phase in the dolomite, which not only improves the refractoriness (2340℃) of the dolomite brick, but also forms microcracks during calcining. The formation of micro-cracks can limit the propagation of stress cracks in the brick. The thermal spalling of the refractory brick is reduced, and the thermal shock stability of the refractory brick is improved. And the improvement of anti-stripping performance can better protect the stability of forming kiln coating. Therefore, the micro cracks produced by this reaction can improve the thermal shock resistance and hydration resistance of dolomite bricks. At the same time, the bulk density of dolomite bricks is about 5% lower than that of magnesia chrome bricks, and the brick thickness can be appropriately thinned by about 10%, which makes dolomite bricks have a better cost performance [7, 8].

2.2. Properties of Refractory Lining for Cement Kiln Burning Zone

Refractory properties are often used to predict, select and prescribe refractories for specific applications. Below are refractory properties that ensure the burning zone lining withstand the harsh conditions in the kiln and maintain its integrity and performance over time.

2.2.1. Physical Properties of the Refractory Bricks

Bulk Density

Bulk density is the mass per unit volume of a refractory material. It is a measure of the weight of a given volume of the refractory. Pountouenchi *et al.*, [9] stated that the bulk density provides a general indication of the product quality. It is considered that the refractory with higher bulk density (low porosity) will be better in quality. An increase in bulk density increases the volume stability, the heat capacity, as well as the resistance to abrasion and slag penetration. The standard bulk density value of refractory material for cement kiln burning zone refractory lining ranges from 1.71-2.28 g/cm^3. According to Assa *et al.;* [10], bulk density increases at higher temperatures. He reported that the bulk density of zirconia containing refractory bricks is 2.9 g/cm^3 and 2.97 g/cm^3 after curing at 1500℃ and 1600℃ respectively. Pountouenchi *et al.*, [9], also reported similar phenomena. Equation 2 is used to determine the density of refractory material:

$$Bulk\ density = \frac{D}{(W-S)} \rho_W \tag{2}$$

Where; D = dried weight,
W = soaked weight;
S = suspended weight;
ρ_W = density of water.

Apparent porosity

Apparent porosity is the measure of the effective open pores in clay into which molten metal, slag, fluxes, and vapours can penetrate into a refractory material. The apparent porosity of refractory material can be determined using the immersion principle. Samples with low apparent porosity have greater resistance to penetration by slags and fluxes, and usually lower gas permeability than those with high porosity [9]. Likewise, high porosity in refractory materials translates to increased air pockets and improved thermal insulation particularly in a pyrolysis plant where linings are not exposed to fumes and vapour [3, 10]. The standard values range for porosity of clay materials is 15-17% [3]. Tatič *et al.;* [3], shows that ferrous alumina spinel brick's apparent porosity is only 13-15%, which is about 2-4% lower than that of magnesia-chrome brick and within the standard value for cement kiln burning zone refractory brick. The apparent porosity can be determined based on the ASTM Standard C133 using the following equation:

$$Apparent\ porosity = \frac{W-D}{W-S} \times 100 \tag{3}$$

Where, W = Soaked weight
D = Dried weight
S = Suspended weight

Refractoriness

Refractoriness of refractory is the temperature at which the material softens under its own load. It is the measure of fusibility of the material. The refractoriness indicated by Pyrometric cone equivalent (PCE) should be higher than the application temperatures. This is a measure of the ability of a material to resist failure at high operational temperatures especially in metallurgical and related industries [11]. Materials with refractory value less than 1,500^{0}C are not suitable for use in metallurgical industries due to their inability to withstand the conditional operating temperatures of such industries [11]. In some cases, refractoriness in materials could be considered as that property in which the material will deform under its own load. The refractoriness of a clay sample is one of the properties that are directly related to its softening temperature which is expressed as its Pyrometric Cone Equivalent (PCE) a number that represents the softening temperature of a refractory specimen of standard dimension [9, 12]. The recommended range for dolomite-zirconia refractories is 1600-1750℃ [10]. Refractoriness of a material is determined using pyrometric cone equivalent method. For instance, in the work of [10], an investigation on the refractory of dolomite-zirconia was conducted using the PCE method. The results showed that the samples have a uniform refractoriness value of 1700℃ which is within the recommended value for use in cement industries [13]. The addition of an impurity in the refractory material lowers its fusion point/refractoriness depending upon the amount of impurity present, the melting point of the lowest fusing constituent and the capacity of the lowest fusing constituent to dissolve the material of higher fusion point at comparatively low temperature as reported by [14].

Refractoriness under load (RUL)

The refractoriness under load (RUL) test gives an indication of the temperature at which the bricks will collapse, in service conditions with similar load. Refractoriness under load of a refractory material is a measure of its failure resistance to the combined action of heat and load. RUL is the softening temperature of a refractory brick (indicated by breaking of the test specimen) when a load of 2 kgf/cm^2 is applied as per the standard test procedure [9]. Refractory bricks generally having more than two constituent compounds fail under load conditions at much lower temperature than the fusion point of the highest melting and predominant constituent [9]. This signifies that the temperature corresponding to the refractoriness under load of a refractory brick is much lower than its refractoriness [9]. For cement kiln burning zones, refractoriness should be more than 1800 ℃.

2.2.2. Mechanical Properties of the Refractory Bricks

Mechanical strength is the ability of a refractory material to withstand mechanical stress, load, and impact without failing or degrading. This property is measured by compressive strength, Flexural strength, Tensile strength and Modulus of rupture. The mechanical properties of materials at high temperatures generally differ from those measured at room temperature. For this reason, refractory materials must be tested at the temperatures of their intended use. According to [15, 16] the mechanical strength properties measured for the purpose of high temperatures performance such as cement kilns are the cold crushing strength and Hot modulus of rupture.

Cold Crushing Strength (CCS)

Cold Crushing Strength is the ability of refractory materials to withstand abrasion and loading without damaging or crumbling into powdered form. The CCS are determined on a hydraulic compression testing machine. The minimum value for cold crushing strength recommended for cement kiln burning zones is 60 MPa [17]. Researchers have made efforts in testing the CCS of some selected materials. For instance, characterization of Obe town clay deposits in Enugu state of Nigeria for refractory production was conducted by [18]. Results obtained showed that Obe clay tested a minimum strength of 21000.15 kg/cm^2 much higher than the recommended value. This high value implies satisfactory crushing strength rendering the clay suitable for slag and flux transportation. However, in the work of [17], the result obtained for fire bricks clay samples of Kona (white & black clay) subjected to CCS test were 252.25 kg/cm^2 and 267.48 kg/cm^2. These values imply a low cold crushing strength because they fell below the recommended value. They recommended an improvement of the crushing strengths through blending with quartz and other additives.

The cold crushing strength of refractory clay can be determined using equation number (4):

$$CCS = \frac{maximum\ load(KN)}{cross\text{-}sectional\ area\ (m2)} = \frac{P}{A} \quad (4)$$

P = Applied Load

A = Area of applied load

Modulus of Rupture (MOR)

The HMOR measures the bending strength of a refractory material at high temperatures. It's an indicator of a refractory ability to withstand thermal stress and mechanical loads in high-temperature environments. It is the indication of flexural strength of a refractory material at elevated temperatures. [19, 20] reported that MOR of refractory material is determined based on the environmental condition. [21] reported that Nru clay MOR has a directly proportional relationship between the temperature and shrinkage. For every increase in temperature, the clay body compresses further. This phenomenon account for the increase in the MOR values of Nru clay with temperature increase. It can also be considered from the perspective of bond formation in the glassy phase of the fired material as reported by [22]. The MOR of cement kiln burning zone is dependent of the type of refractory material in application and the working temperature. However, [23] recommended 4 - 6 MPa at 1450 ℃ for dolomite-based refractories, 6 - 8 MPa at 1450 ℃ for magnesia-based and dolomite-zirconia refractories.

The MOR of refractory material can be determined using equation number (5):

$$Modulus\ of\ rupture\ (MPa) = \frac{3PL}{2BH^2} \quad (5)$$

Where: P = Load applied when the specimen failed

L = Distance between the centre line of the lower bearing edges of the equipment.

B = width of the broken specimen

H = Height of specimen (mm).

2.2.3. Thermal Properties of Refractory Bricks

Thermal shock resistance

Thermal shock resistance is the ability of a refractory material to resist thermal stress caused by rapid temperature changes, without suffering damage or failure. Thermal shock resistance is the fracture of a body resulting from thermal stresses induced by rapid temperature changes. Because ceramic materials are brittle, they are especially susceptible to this type of failure. The thermal shock resistance of many materials is proportional to the fracture strength and thermal conductivity, and inversely proportional to both the modulus of elasticity and the coefficient of thermal expansion. The burning zone should have high thermal shock resistance because it is the high temperature area of the kiln. A little thermal change or increase in thermal load which may come from frequent stoppage of the kiln, sudden collapse of the protective coating and poor heating and cooling of the kiln during light up and cooling of the kiln affect the refractory performance according to [24, 25]. When the thermal stress caused by temperature change exceeds refractories strength, the catastrophic fracture would happen macroscopically [25]. The behaviour of TSR of refractory is often evaluated by the residual strength of the specimen after several cycles of air or water quenching to the hot sample. The repeated heating and cooling numbers are also used to characterize the thermal shock [25, 26].

Thermal conductivity:

Thermal conductivity is defined as the quantity of heat that will flow through a unit area in the direction normal to the surface area in a defined time with a known temperature gradient under steady state conditions. Thermal conductivity depends upon the chemical and mineralogical compositions as well as the glassy phase contained in the refractory and the application temperature [27, 28]. Low thermal conductivity is desirable in cement kiln refractories for conservation of heat by providing adequate insulation. The thermal conductivity of refractory materials for cement kiln burning zones typically ranges from 1.5 to 4.0 W/mK, depending on the type of refractory material and the working temperature [29]. The thermal conductivity was calculated Fourier's law presented in equation 6.

$$\lambda = \frac{Q \times \Delta x}{(T_{hot} - T_{cold}) \times A} \tag{6}$$

Where: Q = Heat flux at the center of the test sample (field of measurement) [W]

λ = Thermal conductivity of the material test sample [W/m.K]

Δx = Thickness of the test sample [m]

A = Section area of the field of measurement at the center of the test sample [m^2]

T_{hot} = Temperature of the hot surface of the test sample [K]

T_{cold} = Temperature of the cold surface of the test sample [K]

Table 1. *Properties of Refractory Material for cement kiln.*

Properties	**Properties of cement kiln burning zone refractory**
Apparent porosity (%)	14 - 18
Bulk density (g/cm^3)	2.85 - 3.0
Cold crushing strength (Mpa)	50 - 90
Hot modulus of rupture (Mpa)	10 - 15
Thermal shock resistance, (cycle)	20 - 30
Refractoriness (^{o}C)	1700-1800
Refractoriness under load (^{o}C)	1500 - 1600
Thermal Conductivity (W/mK)	2.4 - 2.9

Source: Tatič *et al*, [3]

2.2.4. Scanning Electron Microscopy

This is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the sample's surface topography and composition [30].

Shahid *et al.*, [31] stated that SEM is a versatile advanced instrument which is largely employed to observe the surface phenomena of the clay. The sample is shot in a SEM using high energy electrons, and the out coming electrons/X-rays is analysed. These outgoing electrons/X-rays give information about topography, morphology, composition, orientation of grains, crystallographic information, etc. of a material. Morphology indicates the shape and size, while topography indicates the surface features of an object or "how it looks", its texture, smoothness or roughness. Likewise, composition means elements and compounds that constitute the material, while crystallography means the arrangement of atoms in the materials. SEM is the leading apparatus that is capable of achieving a detailed visual image of a particle with high-quality and spatial resolution of 1 µm. Magnifications of this kind of apparatus can extend up to 300,000 times. Although SEM is used just to visualize surface images of a material and does not give any internal information, it is still considered as a powerful instrument that can be used in characterizing crystallographic, magnetic and electrical features of the sample and in determining if any morphological changes of the particle has occurred after modifying the sample surface with other molecules [32].

According to [33], morphologies, as observed by scanning electron microscopy (SEM), can also be useful in identification of clay minerals. Kaolinite shows a variety of morphologies, including platy, pseudo-hexagonal particles, booklets and vermicular stacks. Halloysite also shows generally tubular morphologies although spherical particles are also common. Illite appears as sheets or large flack crystals or as fibres and chlorite clusters of bladed or platy crystals.

3. Materials and Methods

The material and experimental procedure (refractory formulation method, production of refractory and determination of refractory property) are fully discussed in this section.

Materials

The materials for this work were collected from Ashaka cement plant, Gombe state, and Fika, Yobe State, Nigeria. These materials are: Dolomite stone, Zirconia sand, Solid fuel (coal) commercial refractory brick and Kiln feed.

Equipment

The equipment to be used for this study are presented below:

Cement Kiln Polysius cement rotary Kiln, 4 m diameter, 72 m length, Inclined at 3^o and average speed of 2.22 rpm.

Thermography camera Fluke TI300 + 60 Hz Thermal Imager, 320 x 240. Model: Ti300+ 60HZ.

Laboratory crusher AG1290 Laboratory Jaw Crushers, 380 V 50 Hz.

Laboratory mill Parth Industrial Laboratory Ball Mill, Motor Power: 10 HP, Voltage: 380-415 V, Frequency: 50 Hz.

Set of sieves, Technic Woven, Type; Weave Wire Mesh, Frame Diameter: 92 mm, Wire Mesh Diameter: 0.02 to 8 mm.

XRF Machine PANalytical B. V., Axion - Cement x-ray spectrometer: XRF.

SEM TESCAN model, type VEGA 3 LMH and model no VG9731276ZA. 50/60 Hz, 230 V and 1300 VA.

Refractory Bricks cutting machine. Refractory Brick Cutting Machine. Model: Dvlbcm 001. Brand: dvl Engineers.

Muffle Furnace 1800°C Compact Muffle Furnace (4.7x4.7 x 4.7", 1.7L) w/ Kanthal Super-1900 Heating Element - KSL-1800X-KS, AC 208V-240V single phase, 50/60 Hz. heating rate 0 - 10 ℃ /min.

Electrical transversal strength machine model 235, digital weighing machine, spiral balance, sieves, mortar, pestle, moulds, pair of tongs, strong thread, heat conduction testing machine model H9406/02877 PA Hilton, pyrometric cone, meter rule and Vernier caliper.

Method

Determination of burning zone thermal load for the selected Kiln

The thermal load for the kiln was determined using equation 1 presented in the literature. Polysius cement rotary Kiln, 4 m diameter, was used in determining the effective diameter, D.

Characterisation of dolomite and Zirconia samples

Chemical Analysis of the two selected materials (dolomite and zirconia) were analysed to determine their chemical constituents and mineralogical composition. The chemical analysis was carried out at Ashaka cem quality laboratory using X-ray Fluorescence (XRF) Machine while the SEM-EDX was carried out in National Steel Raw Material Exploration Agency, Kaduna.

Production of refractory bricks

Material for the refractory bricks' formulation was selected based on the information obtained from kiln feed, damage bricks chemical composition and thermal load. Dolomite and zirconia were chosen as the raw material for the refractory production.

Material Preparation for Refractory Production

The dolomite rock and zirconia sand collected were first crushed and then grind in a laboratory ball mill, weighted and graded using sieved as presented in Table 2. After grading each material was thoroughly mixed in a mixing machine.

Table 2. *Granulometry of the material for refractory production.*

Material	Particle size distribution	Sizes	Percentage
Dolomite	Coarse particles	100-500µm	25%
	Medium particles	50-100µm	35%
	Fine particles	10-50µm	15%
	Dust	1-10µm	25%
Zirconia	Coarse particles	50-200µm	10%
	Medium particles	20-50µm	15%
	Fine particles	5-20µm	25%
	Dust	1-5µm	30%

Manufacturing Processes of Refractory Bricks

The dolomite, zirconia and the binder where mix based on the formulation as 67.83% dolomites, 27.17% zirconia and 5% binder (composition of alumina and magnesia base binder) according to ASTM C133. Each of the material particle distributions is based on the formulation in Table 2. 10 kg of water was added to the mixture and mixed thoroughly for 5 minutes. The mixture is poured into the sample as shown in Figure 4. The mixture in the mould were pressed at 125 MPa pressure into raw bricks in a hydraulic press for 2 hours.

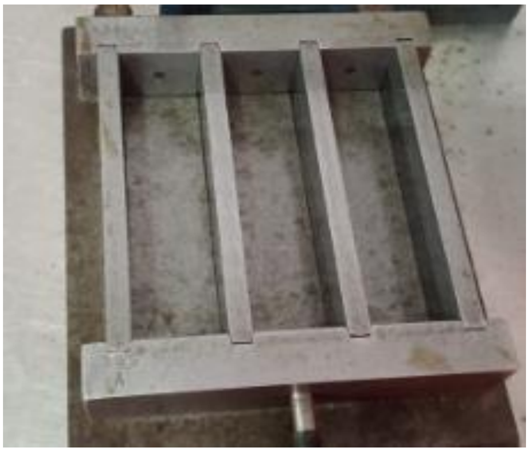

Figure 4. *Moulds for refractory production.*

Drying of the bricks was conducted on large drying floors (heated by waste heat from kilns) where the bricks are laid out in an open array. Drying is done to eliminate the hygroscopic water and increase the green strength of the bricks. The bricks are gradually heated to 1500 ℃. This temperature is maintained and controlled for 2 hours to allow complete heat diffusion and phase transformations. The bricks were allowed to cool with the furnace to avoid a rapid cooling rate.

Determination of refractory bricks properties

Apparent Porosity and bulk density

The apparent porosity and bulk density were determined according to ASTM C133 standard. Each test specimen measuring 30 mm x 30 mm x 20 mm was cut from the refractory brick and dried in an oven at 110℃ to a constant weight (D). The dried specimen was suspended freely in distilled water and boiled for 2 hours, cooled to room temperature and its weight (S) noted. The specimen was removed and the soaked or saturated weight in air (W) was recorded. The bulk density and apparent porosity were calculated from equation 2 and 3.

This procedure was repeated when the refractory was fired at 1200℃, 1300℃, 1400℃ 1500℃ and 1600℃ and the apparent porosity and bulk density were calculated and noted.

Determination of refractoriness

The refractoriness or softening point was determined using the method of pyrometric cone equivalent (PCE) in accordance with ASTM C133. The test pieces were mounted on the refractory plaque along with some standard cones whose softening points are slightly above or below that expected of the test cones. The plaque was then inserted into the electric furnace. The temperature was raised at the rate of 5℃/min during which softening of Orton cone occurred along with the specimen test cone. The temperature was further raised until the tips of the test cones had bent over the level with the base. Then the plaque bearing the specimen was removed from the furnace and the test cones examined when cold. The test cones were then compared with the standard cones and the test materials were assigned the pyrometric cone equivalent (PCE) of the standard cone that it resembled most in bending behaviour. The refractoriness of each test cone is the number of the standard pyrometric cone that had bent over to a similar extent as the test cone. The temperature corresponding to the cone number was read off from the ASTM Orton series.

Determination of Refractoriness Under Load (RUL)

The RUL test was carried out as described in ISO 1893. A cylindrical test piece of 50 mm in diameter and height with coaxial bore of 12.5 mm was prepared. The test piece was subjected to a compressive load of 0.2 MPa and heated at a rate of 5℃/min until the maximum test temperature or until a particular percentage of deformation was reached. The deformation of the test piece is recorded as the temperature increases, and the temperatures corresponding to specified proportional degrees of deformation are determined.

Cold Crushing Strength (CCS)

In determining the cold crushing strength (CCS) of the refractory samples, cubicle specimens 50 mm x 50 mm x 50 mm were made from the refractory samples. The test pieces were fired to 1200℃ and allowed to cool to room temperature before the tests were carried out. Hydraulic load was applied on the test piece until the test piece failed to support the load. The maximum recorded load was taken as the crushing load and the CCS was calculated using equation (4).

This procedure was repeated when the refractory was fired at 1300℃, 1400℃, 1500℃ and 1600℃ and the CCS were calculated and noted. Figure 5 showed the refractory lining in the muffle furnace.

Modulus of Rupture

Modulus of rupture (MOR) using 3-point bend tester in line with ASTM C133 standard for testing modulus of rupture. The rectangular test pieces 150 mm x 50 mm x 25 mm were dried at 105°C until a constant weight was obtained. They were fired to their firing temperature of 1200℃ in the kiln. The electrical transversal strength machine was used to determine the breaking load, P (kg). A vernier caliper was used to determine the distance between supports L (cm) of the transversal machine. The modulus of rupture was then calculated from equation 5.

Figure 5. *Sample in a muffle furnace for CCS testing.*

This procedure was repeated when the refractory was fired at 1300℃, 1400℃ and 1500℃ and the MOR was calculated and noted.

Thermal shock resistance

Air quenching methods are proven to give the most reliable results when compared with the behaviour of refractories in Cement kiln burning zone refractory, hence it was used to determine the thermal shock resistance. Sample of size 114 mm × 64 mm × 64 mm was prepared as shown in figure 6. The sample was placed in a muffle furnace at 950℃. After the sample was put into the furnace, the furnace temperature was monitored and maintained at 950℃ for 30 minutes. The sample was removed and blown with compressed air free from water for 5 minutes. After cooling, the sample was bent with a maximum stress of 0.3 MPa. If the sample is damaged under the action of 0.3 MPa bending stress, it is considered to have failed the thermal shock. If the sample withstands the effect of 0.3 MPa stress, it is considered to have passed the thermal shock and the test is repeated until the sample is damaged or the thermal shock reaches a predetermined number of times. This procedure was repeated for 1200℃, 1300℃, 1400℃, 1500℃ and 1600℃.

Thermal conductivity

The thermal conductivity test was conducted according to ASTM C133. The thermal conductivity of the dolomite - zirconia brick was determined using a hot guided plate method. Test sample was prepared in a circular shape of size 4 mm thick with a surface diameter of 25 mm. It was then transferred to the thermal conductivity apparatus for thermal conductivity measurement at room temperature. In the hot guarded plate apparatus, the test sample was placed in between two iron rods. Thermocouple sensors were inserted below and above the surfaces of the sample to observe the temperature at the lower and the upper surfaces respectively as the heat flows through the refractory sample for 30 minutes. The thermocouple was connected to the data logger; it records the temperature with respect to time. The hot surface was heated to 1200 ℃, 1300 ℃, 1400 ℃, 1500 ℃ and 1600 ℃. 5 Watts input power was used to investigate the thermal conductivity of the material.

4. Results and Discussion

The results obtained from the material characterization for refractory production, refractory produced, refractory property test and cost benefit analysis are fully discussed in this chapter.

Thermal load determination for refractory selection

The thermal load for the kiln under study was determined using equation 9 presented in the literature review. The following parameters are measured and imputed in the equation.

Burning zone brick thickness = 220 mm = 0.22 m

Diameter of the kiln burning zone before putting the bricks = 4 m

Fuel feed rate, F = 8.5 t/hr = 8500 kg/hr

Calorific value of the fuel used, N= 4200 kcal/kg of coal

Burning zone effective diameter after refractory installation = 4 - (2 x 0.22) =3.6 m

The thermal load is

$$T_l = \frac{5.24\times10^{-6}F\times N}{D^2} = \frac{5.24\times10^{-6}\times8500\times4200}{3.6^2} = 14.43\ GJ/m^2hr$$

The thermal load (T_L) for the kiln is 14.43 GJ/m^2hr which is within recommended values for the kiln of this size according to [34]. It is found by experience that thermal load beyond 13 GJ/m^2h., basic refractories such as magnesia spinel, Doloma, dolomite-zirconia, etc work much better [34]. Dolomite and zirconia material were selected because they can withstand this thermal load or even beyond.

Chemical Composition Analysis of Dolomite

The chemical composition of the locally found dolomite in Fika, Yobe state is shown in Table 3. Calcium oxide (CaO) and Magnesium oxide (MgO) are the two major components of the dolomite refractory. According to ASTM C133 requirement, for a material to be suitable for burning zone refractory production, it composition must fall within CaO 30 - 35%, MgO 20 - 25%, SiO_2 1 - 5%, Al_2O_3 0.5 - 2%, Fe_2O_3 0.5 - 2% and other impurities less than 1%. The compositional analysis of Fika dolomite in table 4 showed that it meets this requirement. The purity level to be achieved are as follows: CaO + MgO should be greater than 95%, SiO_2 + Al_2O_3 + Fe_2O_3 should be less than 5% and Other impurities should be less than 1%. Fika dolomites have 94.61% of CaO + MgO, 4.51% of SiO_2 + Al_2O_3 + Fe_2O_3 and 0.88% other impurities. According to Liu *et al.*, [35], high preponderance of SiO_2 has some deleterious effects on the refractoriness of refractories. SiO_2 in small quantities is also known to be a good source of tricalcium silicate which is regarded as the most stabilized form of dolomite refractory material. The low content of SiO_2 and Fe_2O_3 in the dolomite deposit makes it suitable for refractory production. The presence of these fluxing oxides contributes to high crushing strength, high slag resistance and a much higher melting point which makes the dolomite desirable for refractory use. Liu *et al.*, [35] reported similar results. The absence of SO_3, K_2O Cl_2 and Na_2O in the dolomite further prove its suitability for use as cement kiln burning zone refractory material. These oxides react with the basic oxides on the refractory bricks and the kiln feed at high temperature to form $CaSO_4$ and $MgSO_4$. the formation of $CaSO_4$ and $MgSO_4$ weaken the bonding texture of the bricks which will lower its strength and cracking will occur on the refractory. SEM images also reveal that the micro pores are well developed in dolomite crystals.

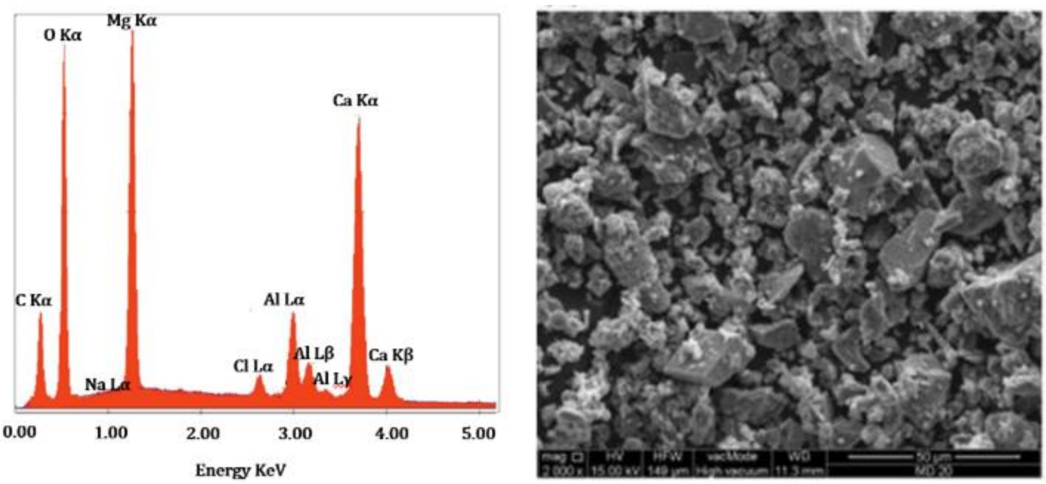

Figure 6. *SEM-EDS image of Dolomite sample.*

Table 3. *Chemical composition of dolomite.*

Major Chemical composition of dolomite sand (wt.%)							
Dolomite	**CaO**	**MgO**	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**MnO_2**	**LOI**
Chemical comp. (%)	34.83	23.96	2.21	0.24	0.35	0.13	32.54

Characterisation of zirconia sand

The major chemical constituent of the locally found zirconia in Gulani, Yobe state is shown in Table 4. Zirconia oxide (ZrO_2), content in the composition is 47.82% which is adequate for use as refractory material for high temperature application. Other major constituents SiO_2, Fe_2O_3 and SnO_2 found in the zirconia sample put it at a very good advantage because their oxide gives it better thermal resistance, thermal conductive and excellent resistance to chemical attack [36]. Potassium oxide K_2O, Sodium oxide (Na_2O), Sulphur (S) etc which are impurities that affect the material properties of cement kiln refractory are not found in the zirconia composition. making it more suitable for the cement kiln refractory material [36, 37].

Table 4. *Chemical composition of zirconia.*

Major Chemical composition of Zirconia sand (wt.%)					
ZrO_2	**SiO_2**	**TiO_2**	**Fe_2O_3**	**Al_2O_3**	**SnO_2**
47.82	4.55	0.18	1.03	1.45	0.23

The composition of ZrO_2 particles was viewed in SEM-EDX characterization. Figure 7 shows the EDX spectra and SEM image of the zirconia respectively. The EDX spectra clearly show the strong intense peaks relating to Zr and O. The weight and atomic percentage of Zr and O, respectively, are 80.65, 17.45 and 43.28, 53.41 respectively. The SEM image indicated wider distribution of the pores. It was observed that these larger pores tend to impede the movement of propagated cracks. This improved the thermal shock resistance of the material. Beside this, the large pores promoted good insulating property by providing air in the void. [34, 38, 39] observed the similar trend in their study.

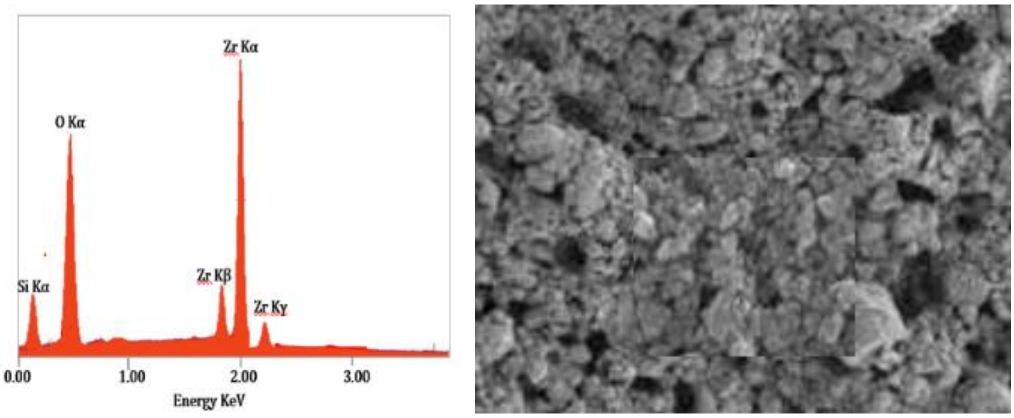

Figure 7. *SEM-EDS image of Zirconia sample.*

Chemical characterisation of the refractory produced

The chemical composition analysis of the produced bricks was displayed in Table 5. The composition of the major oxides is CaO, MgO, ZrO_2, Al_2O_3 and SiO_2 and compound in the bricks meets the compositional requirement for dolomite-zirconia bricks for cement kiln burning zone. According to ASTM C133 standard, dolomite containing zirconia brick must have a high purity, with a minimum of 95% of the total composition being dolomite, zirconia, and other specified oxides. The reproduced refractory brick has 99.73% of dolomite, zirconia, and other specified oxides as presented Table 5. The refractory must have a low level of impurities, with a maximum of 1% of other impurities such as sodium oxide, potassium oxide, and titanium dioxide. The produced bricks have 0.27% impurities. The refractory meets the requirement of the standard to function effectively as a cement kiln burning zone refractory. ZrO_2 content enhances chemical resistance, mechanical strength, thermal shock resistance and CaO contributes to refractoriness and binding property of the refractory bricks produced as reported by [38]. The composition and purity of the refractory showed that the refractory will have longer life and stable performance compared to the commercial bricks whose compositional analysis reveal the presence of sodium oxide, potassium oxide and other impurities above 1%. These impurities level, aids wear and shortens the life of the refractory.

Table 5. *Composition of the Dolomites - Zirconia refractory.*

Dolomites - Zirconia refractory bricks						
Oxide	**CaO**	**MgO**	**ZrO_2**	**SiO_2**	**Al_2O_3**	**Others**
Comp.%	39.32	28.14	21.75	5.23	3.15	2.14

Microstructure

The microstructure of the brick shown Figure 8 showed that is in accordance with the formulations studied (EDX) indicating that it had ZrO_2 (monoclinic zirconia) and $CaMg(CO_3)_2$ (dolomite) compounds. The EDX analysis confirmed good distribution of material particles in the bricks.

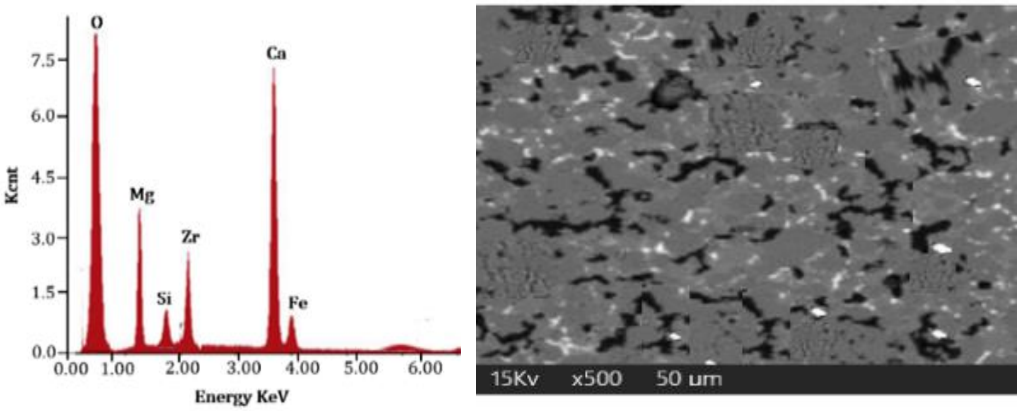

Figure 8. *EDX - SEM image of the dolomite - zirconia refractory brick.*

Effect of Sintering Temperature on Apparent Porosity of DZ Refractory Brick

Figure 9 shows the variations of the porosity of DZ refractory at different sintering temperatures in the range of 950 °C -1600 ℃. A high porosity of 19.4% was observed for samples sintered at 1200 ℃. However, an increase in sintering temperature was accompanied with a decrease in the pore size, so the porosity was reduced to 16.2% at 1600 ℃. This is because as temperature increases the number of fusing of particles, hence the porosity between the particles decreases. Similar phenomenon was reported by [40-42]. The apparent porosity for the reference commercial refractory was found to be 17% while that of the produced refractory was found to be stable between 1400°C to 1600 ℃ and its value is 16.1 to 16.4%. This implies that the fusing of the material particles is completed (no more pores size reduction). Dolomite-zirconia bricks produced have better porosity and lie between the accepted value of porosity for cement kiln burning zone refractory bricks which is 15 - 18%. These results correlate with the result of bulk density and thermal conductivity of the refractory bricks.

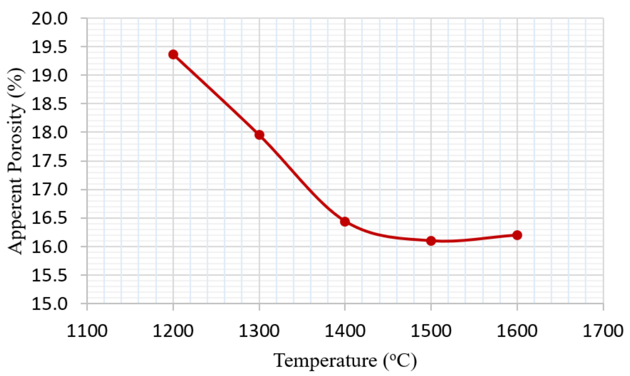

Figure 9. *Effect of sintering temperature on apparent porosity of DZ refractory brick.*

Effect of Sintering Temperature on Bulk Density of DZ Re-

fractory Brick

Figure 10 shows the effect of the sintering temperature towards density. The increment of sintering temperature from 950℃ to 1600℃ increases the density of refractory 2.10 g/cm^3 to 3.42 g/cm^3. The increase in temperature increases the number of fusing particles in the refractory which results in decreasing the number of pores and the density of the refractory. Bulk density of Dolomite-zirconia brick was affected by the presence of CaO and MgO. They contribute in the formation of chemical components of low fusion temperature which flow into the pores and result in an increase in the density of the brick can enhance good properties such as abrasion resistance, slag resistance, MOR and thermal conductivity as reported by [43, 44]. Between 1500°C to 1600°C notice negligible change in the bulk density. This implies that the fusion of the oxide in the refractory is completed and the bulk density of the material is 3.40 g/cm^3 which is within the standard value for bulk density of cement kiln refractory material for burning zone which is around 2.5 to 3.5 g/cm^3.

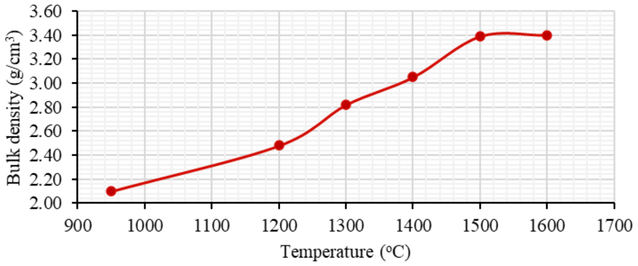

Figure 10. *Effect of Sintering Temperature on Bulk Density of DZ Refractory Brick.*

Refractoriness of Dolomite - zirconia refractory bricks

The refractoriness of the brick samples was determined using polymetric cone equivalent. The brick sample has Pylymetric Cone Equivalent (PCE) of seger cone 29, as it can withstand the deformation temperature of about 1750℃ before fussing or bending under its own weight. However, the commercial refractory bricks have the highest overall PCE (as supposed) of seger cone 32, as it can withstand the deformation temperature of about 1700℃ -1800℃. The refractoriness of bricks produced is less than 1800℃ for cement kiln burning zones according to ASTM C24. However, its value exceeds the working temperature of the burning zone which is usually between 1250℃ to 1450℃. This makes it suitable to be used in the cement kiln burning zone. Similar result was reported by [9].

Refractoriness under load (RUL)

The plot in figure 11 shows a RUL measurement on a test piece of a Dolomite-zirconia brick with increasing temperature. At 1200℃, the test piece reaches its maximum expansion. Deformations of 0.5%, 1.0% and 2.0% occurred at 1450℃, 1600℃ and 1648℃ respectively. These values are lower than the recommended value for RUL for cement kiln burning zone which is between 1700℃ -1800℃. However, the refractory can still work perfectly in the cement kiln burning zone since the working temperature is 1450℃ and they are always protected with coating that lowers the heat transfer to the refractory as reported by [9, 45, 46].

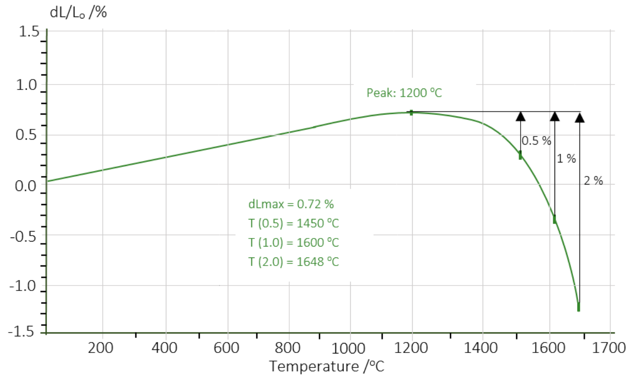

Figure 11. *Refractoriness under load for Dolomite - zirconia brick.*

Effect of Sintering Temperature on Cold Crushing Strength of DZ Refractory Brick

The correlations between CCS with sintering temperatures were shown in figure 12. The figure shows that the CCS increases as the sintering temperature increases. The CCS increases from 90.41 MPa to 95.89 MPa. When the temperature increased by 100℃ from 1200℃ to 1600℃. The increment of sintering temperature caused the enhancement of the number particles which were combined with the other particle making the single solid part, hence the value of CCS is increasing. In this process, the temperature was increased lower than material the melting point temperature. At this temperature, the atoms in the materials disperse across the borders of the particles, blending the particles jointly and making a compact hard piece. Same phenomenon was reported by [47, 48]. The CCS for the refractory meets the minimum requirement for it to function at the cement kiln burning zone whose value is between 40 MPa.

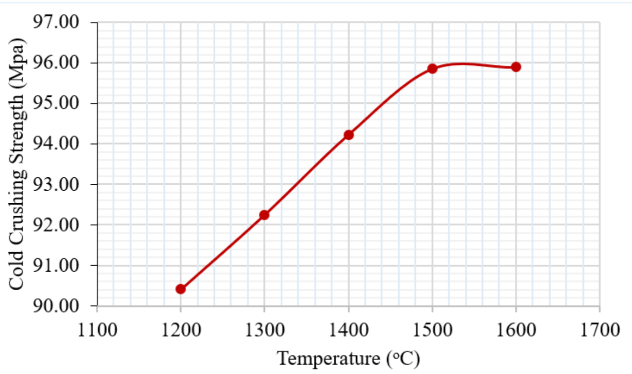

Figure 12. *Effect of sintering temperature on CCS.*

Effect of Sintering Temperature on Modulus of Rupture of DZ Refractory Brick

Modulus of rupture is an indication of the transverse strength of the refractory. DZ refractory indicated values for modulus of rupture from Figure 13 are 14.05 MPa, 14.21 MPa, 14.57 MPa and 15.15 MPa for 1200 ℃, 1300 ℃, 1400 ℃, 1500 ℃ and 1600 ℃ sintering temperatures respectively. It was observed that the transverse strength of the refractory bricks grows with increase in firing temperature. This could be traceable to the development of some phases like the mullite in the structure of refractory brick which is responsible for strength improvement. At 1500 ℃ and 1600 ℃, the MOR stabilized which implied that the highest value that can be achieved is 15.15 MPa. From the result it is clear that the MOR for the refractory produced meets the requirement to be use as cement kiln burning zone refractory brick since its value falls within recommended value ($> 10\ MPa$) according to ASTM C133 standard. [49, 50] reported similar results.

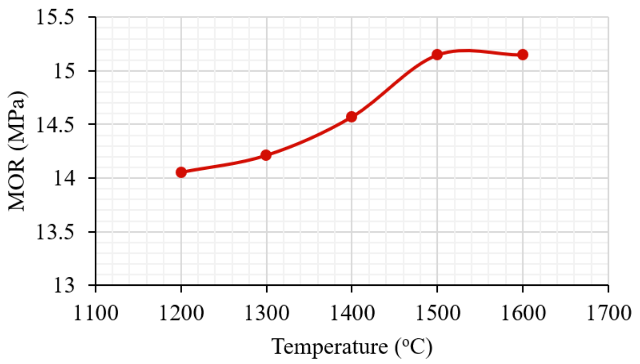

Figure 13. *Effect of Sintering Temperature on Modulus of Rupture of DZ Refractory Brick.*

Thermal Shock Resistance of Dolomite-Zirconia refractory

From the analysis on the DZ refractory bricks, the thermal shock resistances (TSR) fall within the accepted value of 30 -36 cycles. However, with the increase in temperatures that is, 950 ℃, 1200 ℃, 1300 ℃, 1400 ℃, 1500 ℃, and 1600 ℃ the thermal shock resistance of the material decreases (Table 3). The decrease in the TSR of the materials under different temperatures was as a result of the material leaving a very high temperature to a very low temperature zone; as expansion and contraction of the materials at elevated and low temperatures were very high due to the porosity of the materials. Consequently, the shock on the material will be very high leading to the decrease in the cycles. This means that the material will record more cycles under low temperatures than in high temperatures which is evidence in the results obtained. Figure 14 shows the trend of TSR with the increase of temperatures on the material. Similar result was achieved by [9].

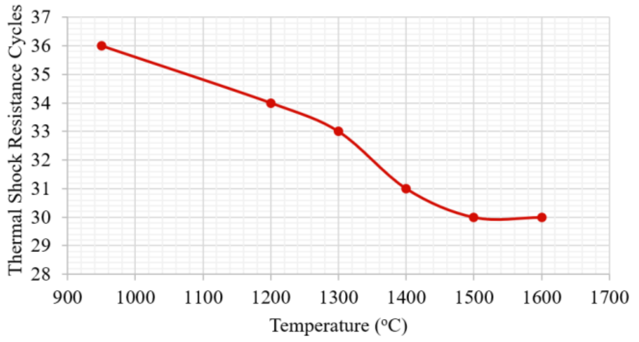

Figure 14. *Thermal Shock Resistance of Dolomite-Zirconia refractory.*

Thermal Conductivity

The thermal conductivity values for bricks developed as indicated in Figure 15 were 2.15 W/mK, 1.93 W/mK, 1.72 W/mK, 1.56 W/mK and 1.65 W/mK respectively. the decrease in the thermal conductivity of the brick is because as the temperature increases, the atoms in the materials dispersed across the borders of the particles, blending the particles jointly and making a compact hard piece into the pores sizes reduces resulting in low thermal conductivity. These results correlate with the apparent porosity results. They show that materials which are more porous have less thermal conduction capacity and as such possess better insulating property. The thermal conduction of a brick material is a function of pore sizes and their distribution in the brick structure. The voids in the pores contain air which is a poor conductor of heat and therefore, resist thermal flow as reported by [51, 52].

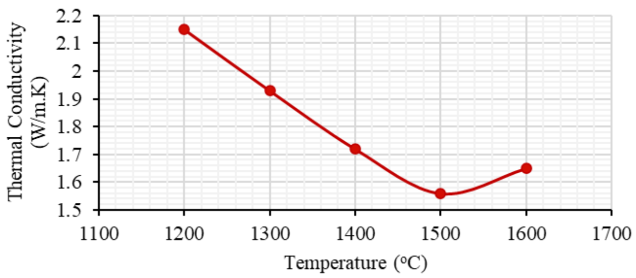

Figure 15. *Effect of sintering temperature on thermal conductivity.*

Summary of refractory properties for cement kiln burning zone

Table 6. showed the summary of the refractory lining properties for cement kiln burning zone refractory. the properties give the minimum required properties irrespective of the type of refractory material used.

***Table 6.** Summary of the refractory properties for cement kiln burning zone.*

S/No	Refractory Properties	Produced refractory bricks	Burning zone refractory according to [3]	Commercial bricks in use	Burning zone refractory Based on ASTM C133
1	Apparent Porosity (%)	16	15-20	17	15 - 18
2	Bulk density (g/cm^3)	2.1 - 3.4	2.8 - 3.2	3.24	2.5 - 3.5
3	Cold Crushing Strength (MPa)	90 - 96	65 – 120	90	50 - 120
4	Modulus of Rupture (MPa)	14 - 15	10 – 15	14	> 10
5	Thermal shock resistance (cycle at 1000 - 1600 ℃)	30 - 36	20 – 30	30	25 -35
6	Refractoriness (℃)	1800°C	1700-1800	1800	1700
7	Refractoriness under load (℃)	1450 - 1648	1500 - 1600	1350	1500 - 1800
8	Thermal conductivity (W/mK)	1.6 - 2.2	1.5 - 2.5	2.1	2 - 4

5. Conclusions

The conclusions drawn from the result obtained are outlined below and gives a detailed summary of the major milestones from the study.

The thermal load for the kiln is within range where basic refractory can perform efficiently in the burning zone.

Dolomite materials characterized meet the requirement for cement kiln burning zone refractory production in terms of composition, particle distribution and purity having 94.61% of CaO + MgO, 4.51% of SiO_2 + Al_2O_3 + Fe_2O_3 and 0.88% other impurities. 47.82% of Zirconia oxide (ZrO_2), and the presence of SiO_2, Fe_2O_3 and SnO_2 found in the zirconia sample put it at a very good advantage because their oxide gives it better thermal resistance and excellent resistance to chemical attack. Potassium oxide K_2O, Sodium oxide (Na_2O), Sulphur (S) etc which are impurities that affect the material properties of cement kiln refractory are not found in the zirconia composition making it more suitable for use a cement kiln refractory material.

The bricks produced exhibits better properties compared with the commercial one use in the kiln as presented in Table 6. This means it has high resistance to chemical attack and thermal shock making it more stable at high temperature with good potential for longer service life that address the concern of cement producers on refractory failure. This directly translates to lower production costs and increased profitability for cement manufacturers.

Abbreviations

DZ	Dolomite -Zirconia
TSR	Thermal Shock Resistance
MOR	Modulus of Rupture
PCE	Polymetric Cone Equivalent
CCS	Cold Crushing Strength

Author Contributions

Samuel Audu Seth: Conceptualization, Data curation, Formal Analysis, Funding acquisition, Investigation, Resources, Writing – original draft

Mohammed Ahmed Bawa: Project administration, Supervision, Validation, Visualization, Writing – review & editing

Aje Tokan: Project administration, Supervision, Validation, Visualization, Writing – review & editing

Jacob Shekwonudu Jatau: Project administration, Software, Supervision, Validation, Visualization, Writing – review & editing

Conflicts of Interest

The authors declare no conflicts of interest

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