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Performance Evaluation of Refractory Bricks Produced from Nigerian Fireclays Blended with Zircon

By Ochieze, P. U. & Esezobor, D. E.
University of Lagos

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PERFORMANCEEVALUATIONOFREFRACTORYBRICKSPRODUCEDFROMNIGERIANFIRECLAYSBLENDEDWITHZIRCON

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Performance Evaluation of Refractory Bricks Produced from Nigerian Fireclays Blended with Zircon

Ochieze, P. U. [α] & Esezobor, D. E. [σ]

Abstract- In Nigeria, importation of several additives usually used in blending fireclays is on the increase. Similarly, other materials available in the country are currently underutilized. These materials could be used as additives to alter/improve the refractory properties of fireclay. The performance behavior of refractory bricks produced from a single and multi-component blending of Osiele and Ikorodu fireclays with Azara-Lafia zircon was studied. The chemical compositions of the raw materials were determined using the Atomic Absorption Spectrophotometer (AAS-PG990AFG). The Osiele and Ikorodu fireclays were blended with Azara-Lafia zircon at varying mass proportions of 10 – 35%. The blended samples were mixed with water (3 – 4%), molded into bricks using the Hydraulic Press (Paul-Weber D-7084) to 300KN. The bricks were air dried for 24 hours and later fired in the Carbolite RHF-16/15 furnace at 800°C to 1200°C. Cold crushing strength test were conducted using Testometric M-500-30KN D7940. The results showed that Osiele clay and Ikorodu clay consist of 30 – 31% Al_2O_3 and 41 – 51% SiO_2 which belongs to the alumino-silicate groups while the zirconia content in Azara-lafia is 65.3%. The bricks produced from Osiele clay and Ikorodu clay with no zircon additive has refractoriness values of 1500°C and 1400°C, thermal shock resistance of 20 and 24 cycles, compression strength of 132KN/m^2 and 108.4KN/m^2 respectively. The refractoriness of the bricks produced from Osiele clay and Ikorodu clay with zircon additives of 10 – 35% has a constant value of 1300°C. The fired linear shrinkage, loss on ignition, compressive strength and apparent porosity decreased linearly with increasing percentage of zircon. These bricks exhibited poor thermal shock resistance as they failed in the first cycle. The result shows that the Azara-Lafia zircon additives are found unsuitable for blending fireclays in the production of high-quality refractory bricks. However, these bricks can find application in lining of ovens, dryers, and furnaces used for various purposes in the industries operating below 1200°C.

Keywords: zircon, osiele clay, ikorodu clay, refractory bricks, refractory properties.

I. Introduction

Refractory materials are said to be high resistant products to the generation of high temperatures of the process at the furnaces and reactors (Kingery et al., 1976). The reliability of refractory for specific applications is determined for its chemical attack resistance to molten slags and molten products, as soon as its mechanical and thermal propertiesarein use (Guzmán et al., 2006). It is also known that all refractory materials deteriorate during application and a common factor in the destruction is the temperature. For this reason, development of new refractory materials with high-temperature resistance and very high resistance in contact with aggressive environments is ongoing. Refractory materials are made in varying combinations and shapes depending on their applications.

Then need to achieve the best performance is a requirement in selecting refractory materials for different applications, such that sudden failure of lining materials is prevented, thus avoiding loss of capital, equipment, energy, and products (Esezobor et al., 2014; Apeh et al., 2011).

These applications include the use in furnace linings for high-temperature operations such as melting of metals for casting, heat treatment of materials to change or enhance the refractory properties, or in the heating of materials to change their shape during metal working operations such as forging or rolling.

Some refractory materials such as zirconia find operations in the lining furnaces for glass production (Guzmán et al., 2006). Zirconia (ZrO_2) which is an oxide form of Zirconium comes as a white powder which possesses both acid and base propertiesis a component in some abrasives, such as grinding wheels and Send Paper (Kerbs, 1998).Because of its mechanical strength and flexibility, sintering into Ceramic Knives and other blades is possible.

In combined form with silica is called Zirconas it exists with silica (SiO_2) in the form of Zircon sand ($ZrSiO_4$). Zircon is a mineral occurring in tetragonal crystal, usually of a brown or grey color. It is sometimes used as a false gemstone.

Zircon Send is used in laboratory crucibles, in metallurgical furnaces, and as a refractory material in developing AZS refractory materials (Lide, 2007–2008).

Zircon has properties such as high melting point of about 2700°C, high thermal insulation, and high strength up to a temperature of 1500°C, reduced porosity, cracking and warping arresting functions and the ability to bind with clay when formed (Guzmán et al.,

Author α: Masters Research student, Dept. of Metallurgical and Materials Engineering, University of Lagos, Nigeria.
e-mail: ochiezeup@gmail.com
Author σ: Professor, Dept. of Metallurgical and Materials Engineering, University of Lagos, Nigeria.

2006). Hence, most zircon is used directly in high-temperature applications. Use of zircon in aggressive environment such as in molds is possible due to its chemical resistant ability.

However, this material is also refractory, hard, and resistant to chemical attack. Hence, the consideration of producing bricks by blending this readily available zircon sand with some selected fireclays to enhance their properties. This will solve the problem of shortage of refractory bricks in Nigeria thereby improving the economy of the nation and also provide a reasonable overview of the current status of this type of refractory brick.It will also provide a summary of recent information concerning the characteristics of this refractory and subsequent blending of Nigerian fireclays with zircon sand for the production of quality refractory bricks.

The research studies the performance behavior at a laboratory scale of refractory bricks produced from selected Nigerian fireclays blended with varied volume fractions zircon sand for high-temperature furnace linings by characterizing their physical, chemical, mechanical, and thermal properties in relation to industrial standards. In effect, it will help to investigate the effects of the volume fractions of zircon sand on the physio-thermal and mechanical properties of the refractory bricks produced from selected Nigerian fireclays.

II. Methodology

a) *Materials*

- Fired clay samples - from Ikorodu in Lagos State and osiele in Ogun State, South West of Nigeria.
- Zircon - from Azara-Lafia in Nassarawa State, North Central of Nigeria.

b) *Equipment*

The equipment used in the project includes: Furnace (Carbolite RHF-16/15, Insulating brick lining 501/20ED Model No. 8477 230 -400 volts, England), Oven, Electronic Weighing Apparatus, Hydraulic Press (Paul-Weber D-7084Remshaden-Grunbaoh, Max. pressure: 350KN), Compressive Strength Machine (Testometric M-500-30KN, D7940 Seidner+CO GMBH, Germany), Atomic Absorption Spectrophotometer (AAS-PG990AFG),Hammer crusher (Shanghai Shibang Model 1183), Ball mill, Sieves of mesh sizes of 1183 microns to 150 microns, Tongs, Beakers, Conical flask.

c) *Experimental procedure*

The chemical analysis of the clay was carried out using an Atomic Absorption Spectrophotometer. The samples were crushed using jaw crusher and hammer crusher and ground with a ball mill in Nigerian Building and Railway Research Institute (NBRRI) Otta, Ogun state. The ground samples were further soaked, dried, and sieved through mesh sizes 600 to 150 μm. The clay samples were blended with zircon at various proportions of 10%, 20%, 30% and 35% by mass (Table 3.2). The blended mix samples were done in conformity with ASTM standards 1989 for tests by mixing with water (3 – 4%), molding and pressing with a hydraulic press to 300KN. The molded bricks were air dried for 24 hours and later fired in the Carbolite RHF-16/15 furnace up to 1200°C. The refractories bricks were tested for refractoriness, thermal shock resistance, cold crushing str

i. *Apparent Porosity Test*

Representative pieces of test bricks were prepared and air-dried for 24 hours. Apparent porosity test procedure was carried out and the apparent porosity was calculated using equation 1;

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

ii. *Bulk Density Test*

Three test pieces of each clay bricks measuring 60mm x 60mm x 15mm were molded and the bulk density was calculated using equation 2 from the bulk density experimental procedure.

$$\text{Bulk Density} = \frac{D \times \rho_w}{W-S}\ (\text{g/cm}^3) \quad (2)$$

iii. *Fired Linear Shrinkage Test*

Fired linear shrinkage was calculated from equation 3 and test pieces were made into standard slabs for this experiment.

$$\text{Fired Linear Shrinkage} = \frac{D_1 - F_1}{D_1} \times 100 \quad (3)$$

Table 1: Chemical composition of materials

Materials	Chemical Composition Analysis (%)									
	Al_2O_3	SiO_2	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	ZrO_2	LOI
Osiele Clay	30.31	51.73	1.46	0.28	0.16	0.62	2.31	0.07	-	13.08
Ikorodu Clay	37.10	41.46	0.10	1.32	0.92	0.20	0.12	-	-	18.78
Zircon Sand	0.47	29.56	0.39	-	-	-	-	0.14	65.30	4.14
Standard *(Devon)	26 – 70	35 - 57	0.5 - 1.6	<0.7	<0.2	<0.10	<2.0	<1.1		12 – 15

iv. *Moisture Content Test*

Dried sample was weighed and then placed in a furnace and heated to a constant temperature of 110°C for 24hours. To calculate the moisture contents the following expression in equation 4 played a role;

$$\text{Moisture Content} = \frac{W - W_1}{W} \times 100 \quad (4)$$

v. *Cold crushing strength (CSS) Test*

Fired test pieces of clay samples prepared to a standard size of 76.2mm^3 on a flat surface used to conduct a compression strength analyses was made and the Compression Strength (C.S) was calculated from equation 5;

$$\text{Comp. Strength} = \frac{\text{Maximum load (KN)}}{\text{Cross Sectional Area } (m^2)} \quad (5)$$

vi. *Refractoriness Test*

Refractoriness was determined via pyrometric cone equivalent on test pieces carried out in the Carbolite RHF-16/15 furnace in reference to ASTM standard C-24 (ASTM, 2008).

vii. *Thermal Shock Resistance Test*

Samples measuring 50mm by 75mm fired in a furnace maintained for 10 minutes at 900°C and cooled repeatedly. The numbers of heating and cooling cycles for each specimen was recorded.

viii. *Loss on Ignition Test (LOI)*

Dried mass of the test sample at 110°C was heated in a muffle furnace to a temperature of 900°C for 3hours in a clean and dried porcelain crucible. The loss on ignition (LOI) was calculated using equation 6

$$\text{LOI} = \frac{m_2 - m_3}{m_2 - m_1} \times 100\% \quad (6)$$

Where, m_1 = weight of the crucible (g); m_2= weight of clay and crucible (g); m_3 = weight of the dried clay and crucible (g).

III. Results

a) Chemical Analysis

The chemical analyses of the refractory raw materials are shown in Table 1.

The major constituents of clay samples are silica and alumina while that of zircon is zirconia. The silica content in Osiele clay is greater than 50%, while the alumina content is about 30% and iron oxide is 1.5% with other substances such as oxides of sodium, titanium, and calcium are in small proportions. Ikorodu clay has silica content less than 50%, alumina content greater than 30% with iron oxide of about 0.1% and other oxides still exist in traces. The result shows that the clay samples belong to the family of alumino-silicate and semi-acid refractory since the alumina value falls within the classification of the standard value range of 25-45% (Gupta, 2008). The presence of impurities in alumino-silicate refractory, such as, Fe_2O_3, K_2O, MgO, lowers the refractoriness and service limit of the bricks. The values of the clay sample's loss of ignition are also within the recommended range of 6 - 15% for kaolinitic clay. The Zircon sand contains 65.30% of zirconia, 29.56% of silica and traces of iron oxide and titanium oxide. Zircon would increase the corrosion resistance of the refractory materials but alternatively reduces the binding properties of the zircon with the clay samples. The presence of high silica content (29.56%) in the zircon when bound with the clay materials can lead to the susceptibility of the bricks to spalling at temperatures below 600°C, hence decrease in strength of the refractory bricks. The presence of traces of Iron in the form of haematite (Fe_2O_3) compounds in clay acts as a flux and causes fusion which subsequently affects the refractoriness of the product.

b) Physical Properties

The essential brick properties like moisture content, apparent porosity, and bulk density are in the result. Observation during the processing and firing of the materials showed that zircon absorbed less water from clay which enhanced its mouldiness. The refractory products were soft, and the water absorption rate differs in the two clay samples. The moisture content of the bricks produced from the samples is high because the addition of zircon needed more water before it could bind with the fireclay samples.

The apparent porosity of the refractory bricks produced from clay samples with no zircon additive which are 15.27% and 16.11% respectively falls below the standard value of 20 – 30 according to Chesti (1986) while their bulk densities on the other hand of 2.20 and 2.36 g/cm^3 respectively falls within the standard range of 2.2 – 2.8 g/cm^3 showing the presence of few pores in the refractory bricks.

The apparent porosity of the refractory bricks decreased linearly with the introduction of zircon in increasing percentages for the two clay samples while their bulk density had a linear increase as shown in Figures 1 and 2 respectively. The low apparent porosity and high bulk density hinders the entrapping of gases in the material during operation, and will enhance the life-span of the refractory brick (Gupta, 2008). The values increased with the addition of fine grains of zircon which decreased the number of pores in the refractory bricks thereby increasing the bulk density of the material. The addition of zircon makes the refractory bricks material dense.

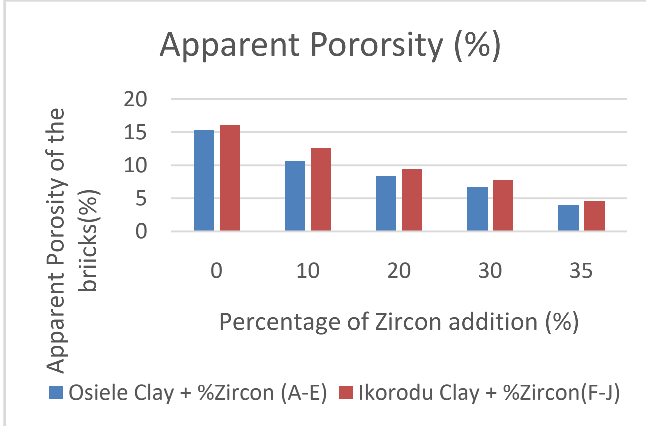

Figure 1: The variation of apparent porosity of the refractory bricks with percentage zircon addition

c) Mechanical Properties

The mechanical property of the tested refractory material was the compression strength (cold crushing strength). The behavior without and with the addition of zircon are shown in Figure 3.

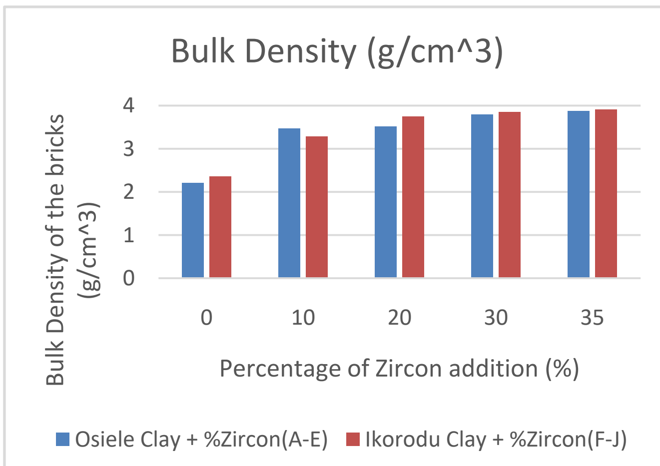

Figure 2: The relationship between bulk densities of the refractory bricks with percentage zircon addition

The refractory bricks produced from the Osiele clay and Ikorodu clay with no zircon additive has high compressive strengths of 131.95 KN/m^2 and 108.39 KN/m^2 respectively. These values fall within the recommended range of 15.0 KN/m^2 minimum values. The presence of high silica content in the clay samples may have caused this high strength. Silica content above 46.5% indicates free silica which enhances the strength of refractory materials (Gunter, 2005; Esezobor et al., 2014). The strength of the refractory bricks produced from the samples, however, decreased appreciably as a result of the addition of zircon as shown in Figure 3. The addition of 10% zircon to Osiele clay slightly reduced the compressive strength of the refractory brick but further additions of zircon (20%, 30%, and 35%) drastically lowered the compressive strength of the refractory bricks produced. From 10% addition of zircon to Ikorodu clay lowered the compressive strength of the refractory material so much that it failed below 15 KN/m^2 (recommended for refractory materials). Hence, the cold crushing strength of the refractory bricks produced with zircon above 30% for Osiele clay and the addition of zircon to Ikorodu clay falls below the standard value of 15 KN/m^2 minimum recommended. These additives also enhanced clay cracking and rupture during firing as it occurred in the case of increasing percentages of the zircon.

However, the addition of zircon significantly reduced the cold crushing strength of the refractory material as the samples have values that are below the recommended standard value. Thus, the refractory bricks produced have less resistance to load in compression, and the presence of zircon worsens this load-bearing capacity.

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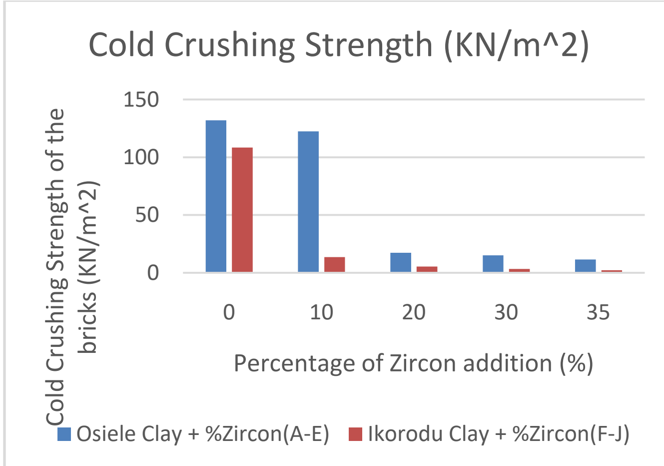

Figure 3: The relationship between crushing strength of the refractory bricks with percentage zircon addition

d) Thermal Properties

Table 2 shows the results of the thermal properties such as refractoriness and thermal shock resistance.

As seen in the result in Table 2, the average fired linear shrinkage for refractory bricks produced from the Osiele clay sample with no zircon additive is 11.4%, which falls above the range for conventional fireclay materials when compared to a standard value, while that of Ikorodu with no zircon additive of 9.4% is within the recommended values of 2 – 10% (Chesti, 1986). The materials, however, will have a superior interlock of grains, which will consequently enrich the strength o refractory when in operation.

Table 2: Thermal properties of the refractory bricks produced from the samples

Sample (%)	Refractoriness (ºC)	TSR (cycles)
100Os	1500	20 + (Good)
90Os+10Zr	1300	Cracks at initial firing (Poor)
80Os+20Zr	1300	Cracks at initial firing (Poor)
100Ik	1400	24 (Good)
90Ik+10Zr	1300	Cracks at initial firing (Poor)
80Ik+20Zr	1300	Cracks at initial firing (Poor)
Standard*	1500 – 1750	20

However, Figure 4shows that there was a linear decrease with the increase in zircon addition for refractory bricks produced from Osiele clay samples whereas the behavior of the refractory brick produced from Ikorodu clay was not uniform with the addition of 10% zircon. However, there was a linear decrease in the fired linear shrinkage on the refractory bricks produced from Ikorodu clay sample with further zircon additive (20%, 30%, and 35%). The zircon does not burn off during firing but has a shrinkage arrest function which is adequate for refractory production.

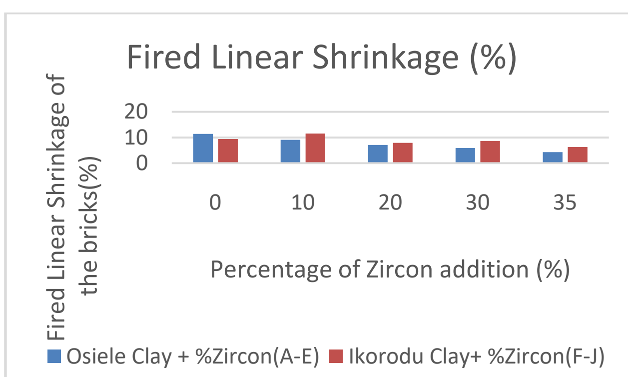

Figure 4: The relationship between fired linear shrinkage of the refractory bricks with percentage zircon addition

The loss on ignition on refractory bricks produced from the samples that contain no zircon addition with values of 7.65% and 9.67% respectively, fall within the recommended range of 2-13% for fireclays (Gupta, 2008). It means that the amount of organic matter content in the clay samples used to produce the bricks is low. But with the introduction of percentages of zircon to the clay samples, the loss on ignition of the refractory bricks increased linearly with increasing percentage zircon addition. Figure 5 shows this variations.

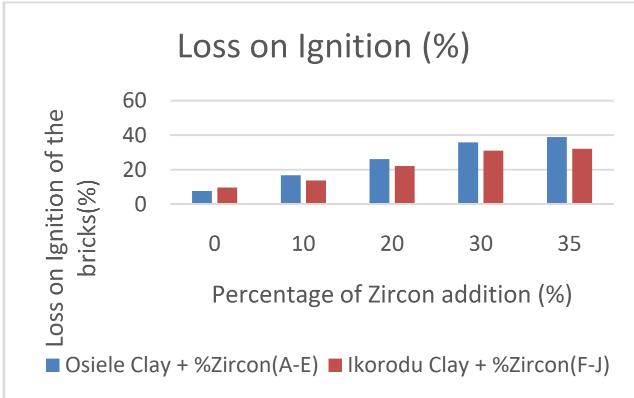

Figure 5: The variation of loss on ignition of the refractory bricks with percentage zircon addition

The thermal shock resistance of the refractory bricks produced from the two clay samples without the addition of zircon are 20 cycles and 24 cycles respectively. These values are within the acceptable values of 20-30 cycles. The refractory bricks produced by the addition of zircon to the clay samples failed at the initial firing stage of the test. It shows that the addition of zircon to the clay samples imbibes poor thermal shock resistance behavior in the fireclay refractory materials.

The refractoriness value obtained for the refractory bricks produced from the clay samples falls below the range of 1,500 – 1,750°C for the fireclay according to Table 2. This low refractoriness can be as a result of the amount of alumina present in the samples. The addition of zircon in percentages of 10, 20, 30 and 35 reduced the refractoriness to a constant value 1300°C for the refractory bricks produced from these samples as seen in Table 2. This behavior can be said to be that the binding property of the clay during the rick formation at 1300°C slowed down due to introduction of zircon. Hence, the drop in refractoriness of the refractory bricks from samples with the addition of any percentage of zircon used for the analysis to a temperature of 1300°C.

IV. Conclusions

The investigation carried out on the addition of zircon to Osiele and Ikorodu fireclays revealed that the service properties of Osiele clay as compared with Ikorodu clays have favourable results. Addition of Azara–Lafia zircon to these fireclays will reduce these properties. The chemical analysis of the clay materials revealed the percentage composition of the raw materials. The Azara-Lafia zircon contains 65.30% zirconia. The silica content for Osiele and Ikorodu clays are 51.73% and 41.46% respectively. The alumina content is 30.31% for Osiele clay and 37.10% for Ikorodu clay. The composition of the clay materials indicated that they are richer in silica than alumina and belong to the alumino-silicate group. The high impurities, such as, Fe_2O_3, CaO, TiO_2 and Na_2O which are very much present in the clays have drastically affected the service properties of the clays by reducing its refractoriness (observed from the increasing loss on ignition with increasing percentages of zircon additive), reducing the binding property and also the strength of the refractory material produced.

With the addition of 10% zircon to Ikorodu clay, the refractory materials becomes dense and with good compressive strength but low refractoriness and thermal shock resistance. The refractories produced from further addition of zircon to Ikorodu clay and the addition of any percentage of zircon to Osiele clay have poor thermal shock resistance, low compressive strength with refractoriness of 1300°C. These refractory bricks cannot find application in lining furnaces used for high-temperature applications above 1200°C. However, they can work furnaces with temperature below 1200°C.

From the results of the research carried out, zircon is an industrial material in the mineral processing industry which can serve as reinforcement for refractory bricks production. Based on the outcomes of the research, we can conclude that:

1. The decrease in cold crushing strength with increasing weight percentage of zircon show that low strength refractory brick can be made from this blend.
2. The refractory brick made with 0-10wt% zircon has the lowest value of firing shrinkage, porosity and with acceptable range of thermal shock resistance. This implies that there could be a possibility to produce good refractory brick from the blend.
3. These refractory bricks have properties which can be compared favourably with Indian fireclay refractory hence can reduce refractory importation.
4. Use of these refractory bricks for furnace lining and casting ladles is possible.

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5. The Azara-Lafia zircon additives are found suitable as blending materials for production of dense refractory bricks in clay for better performance to be used only in ovens operating at temperatures below 1200°C, ladles and transfer pots.

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