

## The Influence of Agro-Forestry Wastes Additive on the Thermal Insulating Properties of Osiele Clay

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### Abstract

The influence of Agro-forestry wastes additives on the insulating properties of Osiele clay for the production of high quality insulating refractory bricks was investigated. The clay sample from Osiele, Abeokuta in Ogun State, Nigeria was crushed, pulverized, sieved and its chemical composition, apparent porosity and bulk density were determined. The clay sample blended in different proportions with binder and rice husk, coconut shell, saw dust and maize cob were moulded into refractory bricks of 100 mm x 25 mm x 24 mm. The bricks were subjected to thermal shock resistance, linear shrinkage, thermal conductivity, and cold crushing strength tests. Mineralogical analysis and micro-structural examination of the bricks were conducted. The chemical composition result indicates that the clay belong to alumino-silicate group while the basic mineralogical contents are quartz, corundum and mullite. It was observed that the bricks produced with mixture of clay and coconut shell possessed the highest cold crushing strength and bulk density. However, the bricks exhibit lowest linear shrinkage and moderate porosity. The SEM micrograph revealed the formation of mullite phase in the bricks sintered at 1150°C. The results therefore indicate that the clay blended with coconut shell, are most suitable for the production of insulating refractory bricks.

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**Keywords:** refractory bricks, wastes, conductivity, alumino-silicate, porosity, strength

### INTRODUCTION

Refractories are generally ceramic materials that are capable of withstanding high temperatures as well as other strains exerted on them such as abrasion, impact, thermal shock, chemical attack and high level loads at elevated temperatures (325 - 3500°C) [Gupta O.P. 2004]. Other characteristics include resistance to thermal shock caused by alternate heating and cooling, porosity (depending upon the service conditions) and ability to conserve heat (insulating property). Hence the refractory products in form of bricks are principally used in furnace construction, to confine hot atmospheres, and to thermally insulate structural members from excessive temperatures (Callister, 2007). They are also used in the lining of many types of metallurgical furnace such as blast furnaces, open-hearth furnaces, soaking pits, heat-treatment furnaces, kilns etc for the conservation of energy in order to reduce the cost of production (Obidiegwu et al, 2014).

Clays in certain regions of various countries are preferred for use in refractory products based on their reputation as successful raw materials for refractories. The choice of clay for the production of furnace refractory lining depends on its constituent. Clays with high iron content exhibit low refractoriness (Titiladunayo and Fapetu, 2011). In addition, 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. The thermal conductivity of refractory clays depends on the chemical properties, mineralogical composition, silica content of the refractory and on the application temperature (Jonker, 2006). The foregoing actually suggests that low thermal conductivity is desirable for conservation of heat, as the refractory acts as an insulator. Porous refractory bricks of low thermal conductivity find wider applications in low temperature heat treatment furnaces, where the low heat capacity of the refractory structure minimizes the heat stored during the intermittent heating and cooling cycles. The high porosity of the brick is created during manufacturing by adding combustible materials to the mix, such as diatomite, Naphthalene, foaming agents etc. During firing, the additives burn out, creating internal pores (Bhatia, 2011). However, these materials are not easily available in the country, therefore alternative additives are needed.

In Nigeria, Due to industrial growth, huge volumes of agro – forestry wastes are generated annually in the country. The disposal of these wastes pose a series of socio-economic and health problems. Thus, creative methods need to be developed to harness the huge endowment of refractory raw materials and agro – forestry wastes into the production of insulating refractory bricks.

This present paper investigates the influence of combustible agro-forestry waste on the thermal insulating property of refractory bricks produced from Osiele clay.

## MATERIALS AND METHODS

### Materials Preparation

The raw materials used for this work were fireclays obtained from Osiele in Ogun State of Nigeria, bentonite (binder) purchased from Nigerian local market as well as agro-forestry wastes (saw dust, rice husk, coconut shell and maize cob) collected from farm lands and refuse bins. The as-received clay and agro-forestry wastes were dried, crushed with a jaw crusher and ground in a ball mill. The pulverized materials were later classified with various sieves (212-600  $\mu\text{m}$ ). Pre-test were done on the various sizes to determine the appropriate sizes for the experiment.

## EXPERIMENT

### Chemical Analysis

The Chemical analysis of the raw materials was carried out using Atomic Absorption Spectrometer (AAS) (model PG 990AFG). 50 g of the clay weighed using METTLER PJ 300 digital weighing machine was mixed with flux (sodium carbonate) on a manual roller, in the proportion of 1:5. This was done to reduce the melting point of the clay. The mixed sample was evaporated in silica evaporating dish to dryness on a steam bath. The dish was heated to 400-500°C in a muffle furnace until the sample transforms to a white ash. For sodium determination, it was heated to 600°C. The chemical composition of the raw materials was further determined in accordance with ASTM 323-56 (2011).

### Determination of Linear Shrinkage

The shrinkage properties of the pressed samples were determined by measuring the dimension of both the green and indurated bricks, using a digital vernier caliper. The three sides of the bricks were measured while the average linear shrinkage was evaluated using equation 1

$$\text{Total Linear Shrinkage} = \frac{(L_0 - L_1)}{L_0} \times 100\% \quad (1)$$

Where

$L_0$  = The original length of the green sample (mm),

$L_1$  = The Final length of the sample after firing (mm).

### Thermal Shock Resistance

This is the test that determines the ability of the refractory bricks to withstand rapid changes in temperature without cracking. The test samples were placed in a muffle resistance furnace, at temperature of 900°C for 10 minute and cooled for 10 minutes in

the air. The cooled samples were returned to the furnace with retention time of 10 minutes. The process was repeated until the samples cracked. The numbers of heating and cooling cycles were recorded against each sample.

**Thermal Conductivity:** The thermal conductivity of the test samples were evaluated using the expression in equation 2.

$$K = \frac{2.303MC\theta[\log(\theta_1/\theta_2)]}{A \times \tau} \quad (2)$$

Where,

$K$  = thermal conductivity of the specimen, (W/m°C)

$T_s$  = temperature of steam (°C),

$T_1$  = Initial temperature of water in conical flask (°C),

$T_2$  = Final temperature of water in conical flask (°C),

$\tau$  = Time (s),  $A$  = Specimen area, (m<sup>2</sup>),

$M$  = mass of water in conical flask (kg),

$C$  = specific heat capacity of water in conical flask (J/kg°C),

$\delta$  = thickness of specimen (m),

$\theta_1 = T_s - T_1$  and  $\theta_2 = T_s - T_2$ .

**Bulk Density:** This is the ratio of mass of the material to its bulk volume and it is expressed in g/cm<sup>3</sup>. Bulk density and apparent porosity were determined in accordance with ASTM C373-88, 2006 Standard Test Methods for Bulk Density, Apparent Porosity and Water Absorption of Refractory Bricks and Shapes. The dried samples with weight ( $D$ ) were soaked in hot (boiled) water for 3 hours. Thereafter, the soaked weight ( $W$ ) and suspended weight in water ( $S$ ) were taken. Equation 3 was used to evaluate the bulk density of the bricks

$$\text{Bulk density} = \frac{D \times \rho_w}{W - S} \quad (3)$$

Where,

$D$  = The dried weight, (g),  $W$  = The soaked weight, (g),  $S$  = The suspended weight, (g),

$\rho_w$  = Density of water (g/cm<sup>3</sup>)

**Apparent porosity:** Apparent porosity is the ratio of open pores to the bulk volume of the material. The apparent porosity expressed in percentage was calculated using equation (4).

$$\text{Porosity} = \frac{(W - D)}{(W - S)} \times 100 \quad (4)$$

Where,

$W$  = The soak weight (g),  $D$  = The dried weight (g),  $S$  = The suspended weight (g)

**Cold Crushing Strength (C.C.S):** The C.C.S was determined in accordance with ASTM C133-97(2008) E1, using universal testing machine (Testometric M-500-25kN). Rectangular samples of 100 mm x 25 mm x 24 mm were placed each on the machine and the load was applied axially by turning the hand wheel until failure occurs. The Cold

crushing strengths (CCS) of the samples were calculated using equation (5).

$$CCS = \frac{\text{Maximum Load (KN)}}{\text{Cross Sectional Area (m}^2\text{)}} \quad (5)$$

**Mineralogical Analysis:** Mineralogical analysis was conducted on the samples using X-ray Diffraction (XRD) machine; Philips X-pert model PW 1830 generator diffractometer with Cuka radiation source from the Mechanical Engineering Laboratory in the University of Ottawa, Canada.

**Micro Structural Analysis:** The micro structural examination of the samples was done on Scanning Electron Microscope (SEM). (Zeiss, model EVO10) at the Department of Mechanical Engineering Laboratory in the University of Ottawa, Canada.

The results of the various experiments conducted in the study are presented in Table 1, Figures 1- 13 and Plates 1- 4.

**RESULTS AND DISCUSSION**

The chemical composition of Osiele clay, bentonite and the agro-forestry wastes are presented in Table 1

Table 1 Chemical Composition of the Raw Materials and Agro-forestry wastes used

Materials	Level of Parameter (wt %)									
	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	ZnO	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	L.O.I
Osiele clay*	30.31	51.73	1.46	0.28	0.16	-	0.62	2.31	-	13.06
Bentonite	23.10	55.40	4.40	2.50	0.20	-	3.00	3.00	-	8.40
Rice husk	3.93	80.0	0.41	0.25	3.84	-	0.67	1.45	-	9.45
Sawdust	4.10	67.20	2.30	5.80	8.61	-	0.10	1.49	0.01	10.39
Maize cob	1.09	37.26	2.78	3.15	2.10	-	0.04	37.09	-	16.18
Coconut shell	15.60	45.05	12.40	16.20	0.57	0.30	0.45	0.52	0.22	8.69

\*Osiele clay contains 0.07 wt percent of TiO<sub>2</sub>

The results in Figures 1 and 2 indicate the increase of linear shrinkage as a function of the enlargement of the grain size of combustible agro wastes as well as the concentration of added wastes. This is as a result of large pores created by the combustibles which try to shrink during firing thereby increasing linear shrinkage

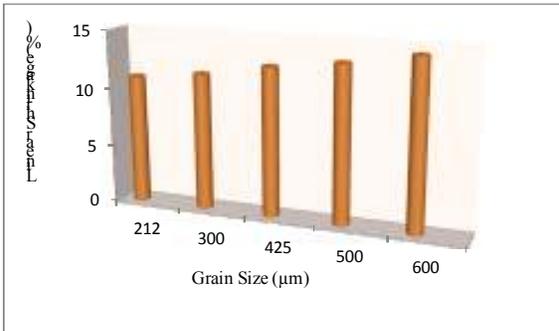


Figure 1 Effect of grain size of combustibles on Linear Shrinkage of bricks

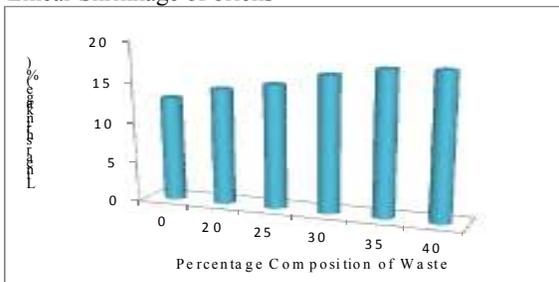


Figure 2 Effect of Percentage Composition of Wastes on Linear Shrinkage of Bricks

The sample prepared with maize cob has the highest percentage (15%) of linear shrinkage as shown in Figure 3. This can be attributed to its relatively high potassium oxide (K<sub>2</sub>O) content and subsequently low melting point. The former is lowered in the samples prepared from sawdust, rice husk and coconut shell (Table1). The presence of K<sub>2</sub>O enhances shrinkage of the bricks (Chukwudi, 2008)

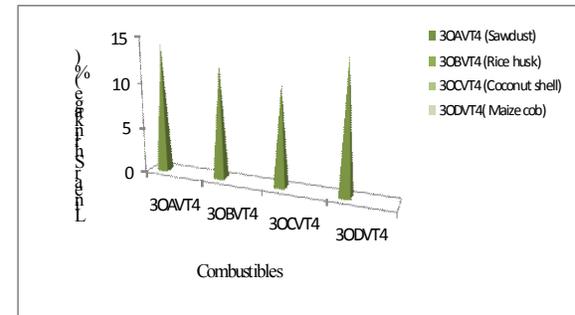


Figure 3 Linear shrinkage of refractory bricks produced with various agro-forestry wastes

The increase in grain size decreases the thermal shock resistance (figure 4). This can be attributed to the increase in pore sizes which act as stress concentrators that were evidenced in Plate 4.

As the grain size increases in Figures 5, the thermal conductivity decreases. This could be as a result of large pores that have been created (Plate 3). The pores hinder heat transfer from one particle to another. Since heat transfer in solids is mainly by conduction. Heat transfer by **Conduction** occurs via the transfer of energy from particle to particle in a material. When there is increase in porosity,

entrapped air comes between the particles inhibits the rate of heat transfer.

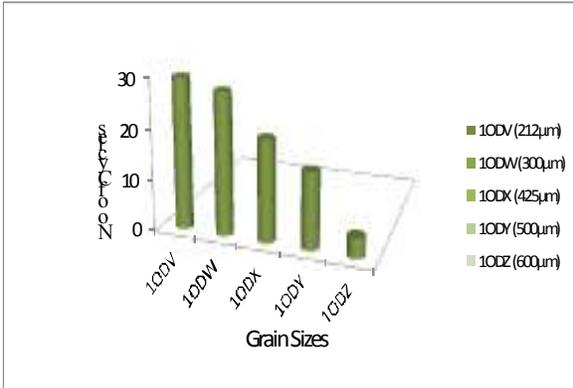


Figure 4 Variation of thermal shock resistance with grain sizes

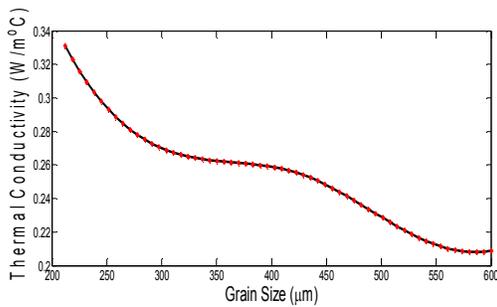


Figure 5 Variation of Thermal Conductivity with Grain Size

The bulk density decreases with increase in percentage composition and grain sizes of agro-forestry wastes. During sintering, the value of porosity increases as a result of the decomposition of minerals and combustion of organic additives which result to the decline in the bulk density as can be seen in Figures 6 – 7.

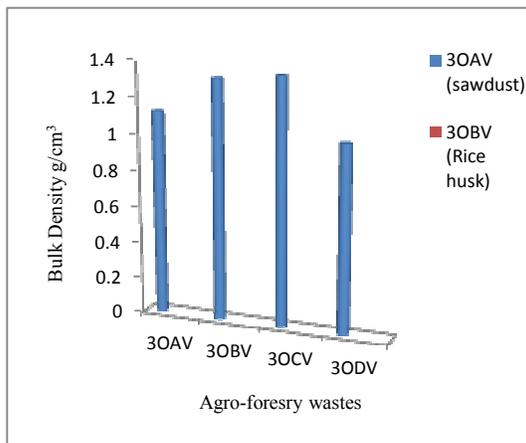


Figure 6. Variation of bulk density of bricks with the percentage composition of agro-forestry wastes

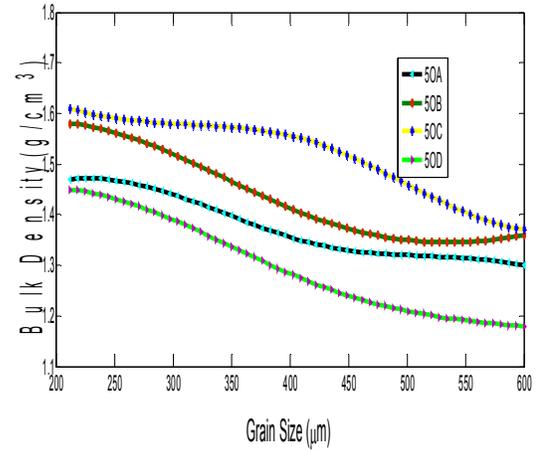


Figure 7 Variation of Bulk Density with Grain Size

Most of the samples (bricks) have high percentage of apparent porosity because of the combustion of the additives (agro-forestry waste) in the material. The same trend was observed in samples with high grain coarseness of the agro wastes (Figure 9).

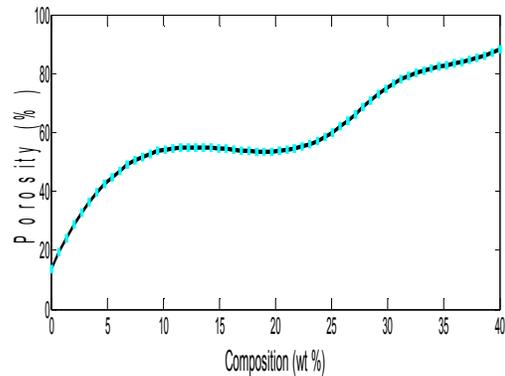


Figure 8 Variation of Porosity with Composition

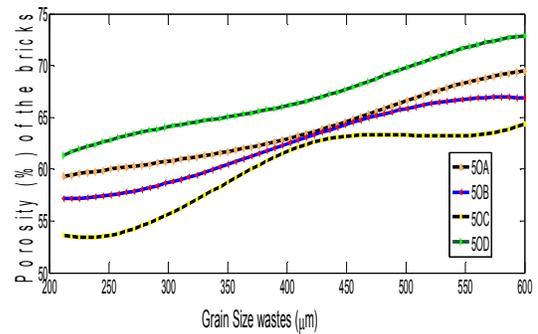


Fig. 9 Variation of Porosity with Grain Size

Figure 10 shows the results of variation of cold crushing strength of bricks with firing temperatures.

The CCS increases with increase in firing temperature. This is as a result of bonding that has taken place at high temperature, thereby increasing the strength of the material through agglomeration of the particles.

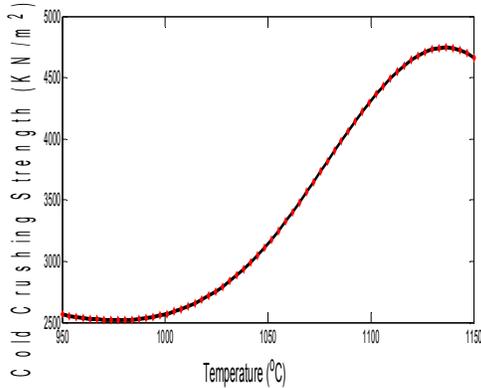


Figure 10 Variation of CCS With Firing Temperature

Figure 10, Variation of cold crushing strength with firing temperature.

The results of the Mineralogical analyses carried out using XRD and interpreted using International Centre for Diffraction Data (ICCD) software are shown in Figures 11 to 13. The identified basic mineralogical constituent of the sample for this study include Quartz  $\text{SiO}_2$  (ICCD 00-005-0490), Corundum  $\text{Al}_2\text{O}_3$  (ICCD00-042-1468) and Mullite  $2\text{Al}_6\text{SiO}_{13}$  (ICCD01-083-1881). The XRD revealed a change of clay phase when sintered from  $1000^\circ\text{C}$  to  $1200^\circ\text{C}$ ,

(Figures 11 to 12). The peak of Quartz ( $\alpha$ ) and Corundum ( $\beta$ ) reduces when sintered from  $1000^\circ\text{C}$  to  $1200^\circ\text{C}$ . This is due to formation of mullite Phase ( $\gamma$ ), (Johari et al, 2010). A new phase of mullite developed between the temperatures of  $1100^\circ\text{C}$  to  $1200^\circ\text{C}$ . The peak also appeared sharp, showing a highly crystalline compound.

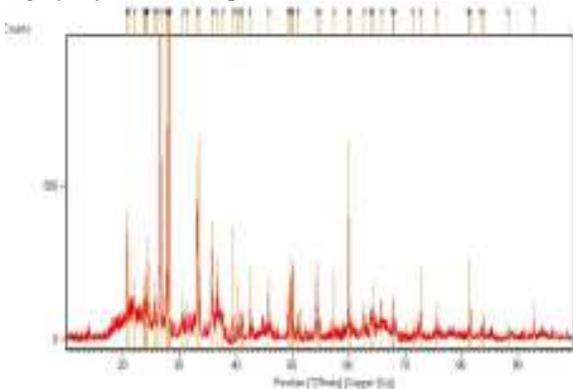


Figure 11 Sample produced with clay only and fired at  $1000^\circ\text{C}$  (OT1)

Colour	Mineral	Formula	Symbol
	Mullite	$2\text{Al}_6\text{SiO}_{13}$	$\gamma$
	Quartz	$\text{SiO}_2$	$\alpha$
	Corundum	$\text{Al}_2\text{O}_3$	$\beta$

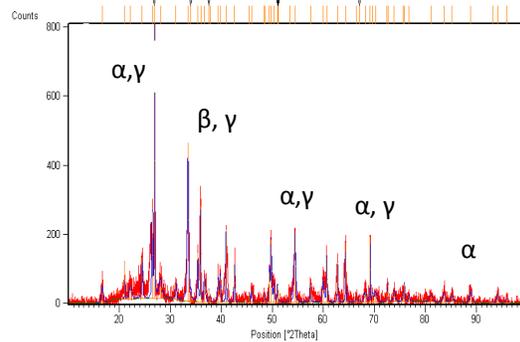


Figure 12 brick with only clay and fired at  $1200^\circ\text{C}$  (OT5).

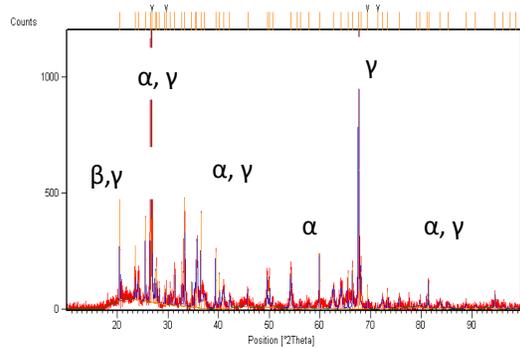


Figure 13, brick with coconut shell (SOC) fired at  $1150^\circ\text{C}$

In the control unfired bricks without any form of additives, pores were invisible in the microstructure as can be seen in Plate 1. However, pores were observed in Plate 2 where sintering occurred. The volatile matters in the samples escaped during firing. The porosity increases with the addition of agro-forestry waste as shown in Plate 3. This is as a result of combustion of the wastes during sintering. It can also be observed in Plate 2, that sample OT1 sintered at  $1000^\circ\text{C}$  contains quartz and Corundum having trapezoidal and oval shape respectively. As the temperature increases to  $1150^\circ\text{C}$  a needle like shape was observed showing the formation of a highly crystalline new compound Mullite, while the quartz phase gradually disappeared (Plates 3- 4).

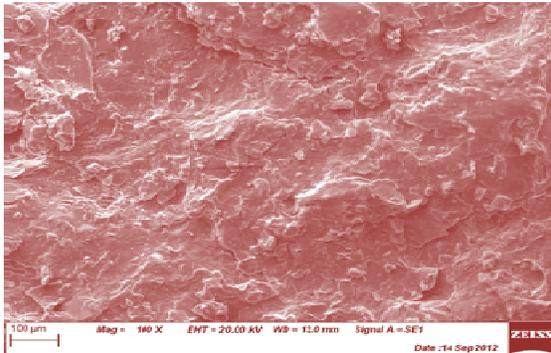


Plate1 SEM of Unfired brick Mag. X100

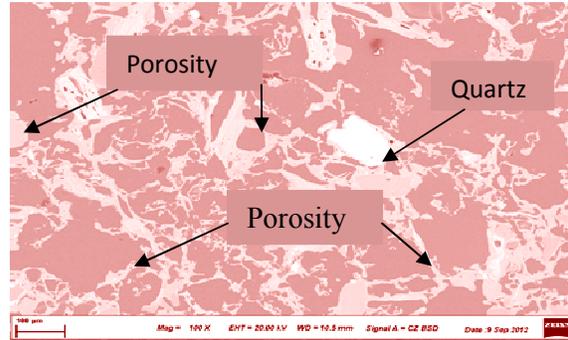


Plate 4 SEM of brick with coconut shell additive sintered at 1150°C

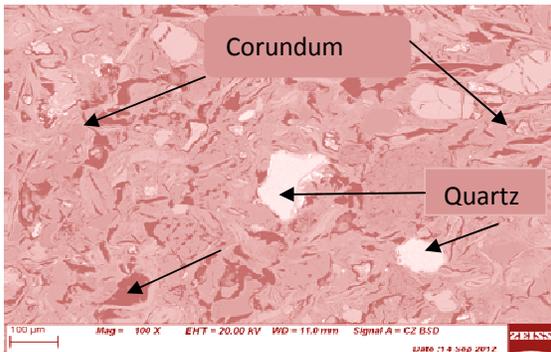


Plate 2 SEM of brick with only clay fired at 1000°C Mag. X100

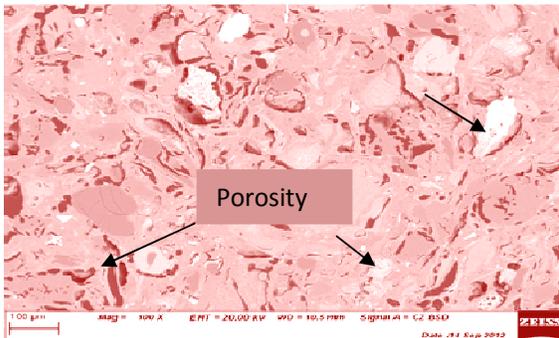


Plate 3 SEM of brick with only clay sintered at 1150°C Mag. X100

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## CONCLUSION AND RECOMMENDATION

It can be concluded that:

- High quality insulating refractory bricks can be produced from Osiele fireclays with agro-forestry wastes in the range of 25-30 wt percent
- The physical, mechanical and thermal characteristics of the produced insulating bricks are dependent on the grain sizes of the agro-forestry waste used. In this research, high quality insulating refractory bricks were produced with agro-forestry wastes of grain sizes between 212-300 µm, and firing temperature in the range of 1100-1200°C.

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