# CHARACTERIZATION OF THE REFRACTORY PROPERTIES OF OSIELE-CLAY

By

**Lawal G.I.*, Amuda, M. O. H.*, Ifekoya D.I., Kuforiji, C.U.* and Olokode O.S.****

** Department of Metallurgical and Materials Engineering, University of Lagos, Akoka.*

***College of Engineering, Department of Mechanical Engineering, University of Agriculture, Abeokuta.*

## ABSTRACT

*The result of investigation into the strength characteristic of Osiele clay from Abeokuta, Ogun State, South West, Nigeria with Global Positioning System (GPS) location 7010"5'N and 3026"11'E is presented. The strength characteristics of bulk density, porosity, water absorption rate, cold crushing strength and shrinkage were investigated at firing temperatures of 8000C, 9000C, 10000C, 11000C, 12000C, and 15000C respectively.*

*The analysis revealed that the clay sample is siliceous alumino-silicate with low content of iron III oxide. The water absorption, bulk density and apparent porosity, decreases with the firing temperatures, whereas, the total shrinkage increases as the firing temperature increases. The clay has excellent cold crushing strength at the pre 12000C firing temperature range. The measured physical/mechanical properties of the clay sample compared favourable with those of international standard for fire clay refractory bricks.*

*It is concluded that Osiele clay can be used as a refractory material for the lining of furnaces property particularly for operating temperatures in the pre 11000C range.*

***Keywords**: Strength characteristics, Osiele Clay, refractory properties, firing Temperatures, Furnace Lining.*

## 1.0 INTRODUCTION

Sourcing of appropriate raw-materials from the abundant natural endowments in Nigeria for industrial use has not generated significant success because technical information on the integrity of these endowments is seldom available. Extensive investigation has been carried out on the liquid mineral endowments of the country, however, not much has been done on the solid mineral endowment of which clay is prominent and consequent upon this, adoption of solid mineral on industrial scale is scanty.

The main policy thrust of the economic reform programme of the Nigerian government is engendering national capability in converting the country's endowments into utility products and services for the common good [1]. A great emphasis is placed on exploring and exploiting the abundant solid minerals endowments in the country with a view to diversifying the economic base of the country, improves Gross Domestic Product (GDP) and industrial activity. The central objective of the effort is to increase the contribution of the non-oil resources to the general wealth pool of the country.

A member of these endowments with tremendous potential for economic utilization is clay. Clay deposit is spread across the six geo-political zones of the country [2].

Clay is a major material input in the metallurgical, ceramic, pharmaceutical, paints and allied chemical and petrochemical industries. Metallurgical, Ceramic and Petrochemical industries, however, account for over 80 percent consumption of clay [3]. The metallurgical industries consume clay as refractory brick in the various heat generating and retaining units such as furnaces and regenerators. The petrochemical industries equally consume it as lining material for the critical fluid catalytic cracking unit (FCCU) of the refinery during petroleum refining. A special class of clay material – bentonite, is used in oil exploration as drilling fluid, and as binding element in moulding sand for foundry. The ceramic industry uses it as a major raw material in the production of structural bricks, stoneware's, earthen wares and some other engineering ceramics. The estimated local market share of clay and its product in the GDP of Nigeria is less than 4 percent, while current investment is insignificantly less than 2% of GDP [4]. There is definitely need for increased investment in the clay-solid minerals sub-sector. However, this increased investment is being hampered by the lack of technical information on the integrity of the clay deposits. There is thus, the imperative for complete analysis of the character of the clay deposits.

Several works had been carried out on some clay deposits in the country [5-9] to test their suitability for refractory lining purposes in furnaces. Some of the clay-deposit investigated are Onibode, Ibamajo, Ijoko, Likosi[7],Ife-Kaolin[8], Bauchi clay[9] and Enugu clay[10-11]. These pioneer works gave fairly good results. However, the studies were not extensive. Equally, the thermal shock resistance of some of the investigated clays was poor. Literature survey revealed that published work on refractory characteristics of Osiele clay is scarce [4]. Thus, this work seeks to study the refractory characteristic of Osiele clay. The work intends to contribute to the

growing knowledge about the refractory characteristics of Nigeria clays for the purpose of possible adoption in systems and applications that require clay/clay products.

## 2.0 EXPERIMENTAL METHODS

### 2.1 Sourcing of Materials

The basic raw materials used in this work are clays sourced from Osiele (Abeokuta), South West, Nigeria with GPS location $7^0$10"5' North and $3^0$26"11' East.

### 2.2 Work Procedure

1000g quantity of the clay deposit was weighed and crushed in a laboratory jaw-crusher and sieved with a 420 micro sieve to obtain fine clay sample. The different samples were moulded with the required amount of water at a measure of 10C1 (centiliter)/200g powder material.

The test specimens were then prepared into test laboratory sizes of cylindrical standard mould of 5.0cm length and 5.0cm diameter size. The prepared test specimens were air-dried and later oven dried at 110oC for 24 hrs before firing at 1100oC for another 24 hrs and subsequently placed in a desiccator.

### 2.3 Test Work

#### 2.3.1 Compositional Analysis

The chemical composition of the sample raw materials was carried out using the Atomic Absorption Spectrometry (AAS)[12] and the result is presented in Table 1.

**Table 1: Chemical Composition of Osiele Clay (%**

| Types of Oxide (Raw) | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | $TiO_2$ | MgO | CaO | $Na_2O$ | $K_2O$ | *L.O.I* |
|---|---|---|---|---|---|---|---|---|---|
| *Percentage (%)* | *55.78* | *30.24* | *0.85* | *1.26* | *0.27* | *0.20* | *0.28* | *2.46* | *8.40* |

The dried sample cones are mounted on a ceramic disc and place in a special type of furnace for Pyrometric Cone Equivalent (PCE) measurement. The heating rate is rigidly controlled at 5°C/min as recommended by British Standard. As the sample cones commenced to melt, its apex slowly tilted inward until it eventually touched the base. The standard cone nearest in condition to the sample defines the refractoriness value of the failed cone. The specimens were observed one after the other or the following signs: cracks, change in colour, fissures and distortion.

### 2.3.3 Determination of Bulk Density

The test specimens prepared as above were weighed to the accuracy of 0.001g (dried weight). After which the specimens were individually transferred to a beaker and heated 200°C for 30 minutes to assist in releasing the trapped air. The specimens were cooled and the soaked weight ($W_S$) taken. The specimens were then suspended in water and the suspended weight ($S_S$) taken. The bulk density was calculated from:

$$B_D\ (g/cm^3) = \frac{D\rho_w}{W_S - S_S} \qquad (I)$$

When D is dried weight in (gm), $W_S$ is soaked weight in (gm), and $S_S$ is suspended weight also in (gm) and $\rho_w$ = density of water ($1g/cm^3$).

### 2.3.4 Determination of Apparent Porosity

For each of the specimens, five test cylindrical shape 50 cm x 50 cm size after firing were tested. The dry weight (D) of each specimen was measured, after which the specimen was transferred into and suspended in a vessel of boiling water for 20 minutes. At this time, the boiling was discontinued and the hot water was replaced by cold water. After 30 minutes in cold water, the weight(s) was measured. The soaked specimen was then weighed in air (W)

The apparent porosity is evaluated from the equation:

$$A_P = \frac{W - D}{W - S} x100\% \qquad (II)$$

### 2.3.5 Determination of Cold Crushing Strength

The specimen were placed on a compressive testing machine and load applied axially by turning the hand wheel at a uniform rate till failure occurs using a well calibrated scale. The cold crushing strength is obtained from the formula:

$$CCS = \frac{\text{Maximum load}}{\text{Cross Sectional Area}}\ kN/m^2 \qquad (III)$$

### 2.3.6 Determination of Linear Shrinkage

Seven test samples were made into briquettes of 5.0cm length and 5.0cm diameter in green form. They were marked along a line in order to maintain the same position after thermal treatment. The length of samples was measured using vernier calipers. The samples

were subsequently air dried for 24 hours and oven dried at 110°C for 12 hrs to release combined moisture. They were then fired at different temperatures of between 800°C and 1500°C at intervals of 100°C for 8 hours each. The test samples were cooled to room temperature and measurements taken to give the fired length. The linear shrinkage was calculated using eqn. (IV) and summarized in Table 2 (appendix).

$$\%LS = \frac{D_L - F_L}{D_L} X100\% \quad (8) \qquad \text{(IV)}$$

$D_L$ = Dried Length and

$F_L$ = Fired Length

#### 2.3.7 Determination of Water Absorption Rate

The dry weight of green state samples was obtained $W_1$ (g) and then, immersed in water for two minutes and their wet weight taken $W_2$ (g). The difference in weight was calculated and the percentage absorption was calculated through equation (V).

$$\% \text{ Water absorption} = \frac{W_2 - W_1}{W_1} \text{ x } 100 \qquad \text{(V)}$$

The percentage weight of water absorbed at the other firing temperatures was equally evaluated using equation V.

## 3.0 RESULTS AND DISCUSSION OF RESULTS

### 3.1 Results

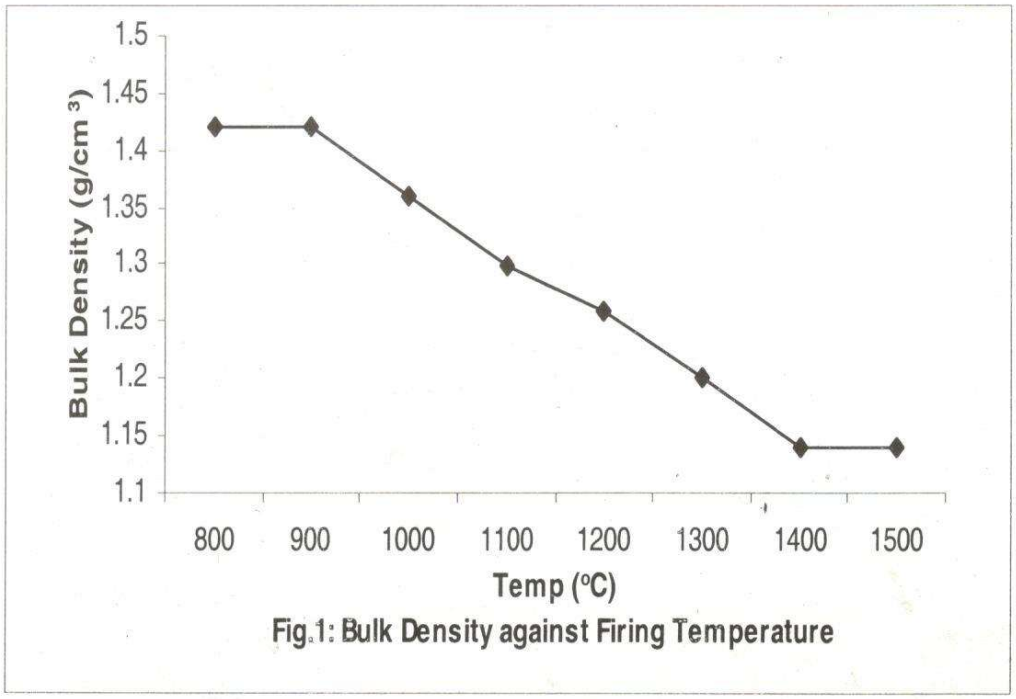


Fig.1: Bulk Density against Firing Temperature

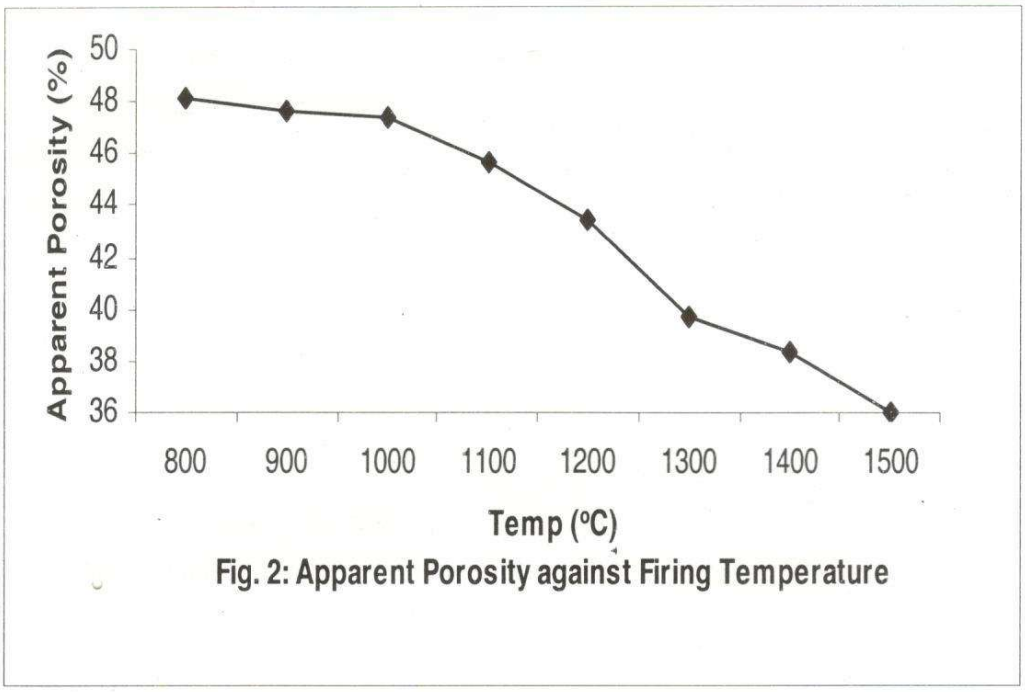


Fig. 2: Apparent Porosity against Firing Temperature

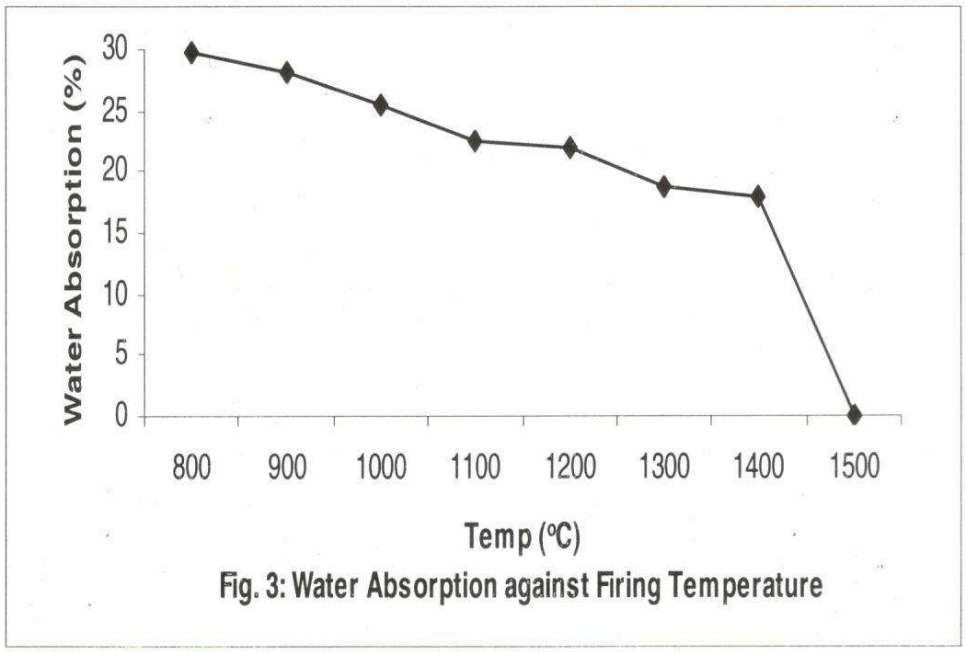


Fig. 3: Water Absorption against Firing Temperature

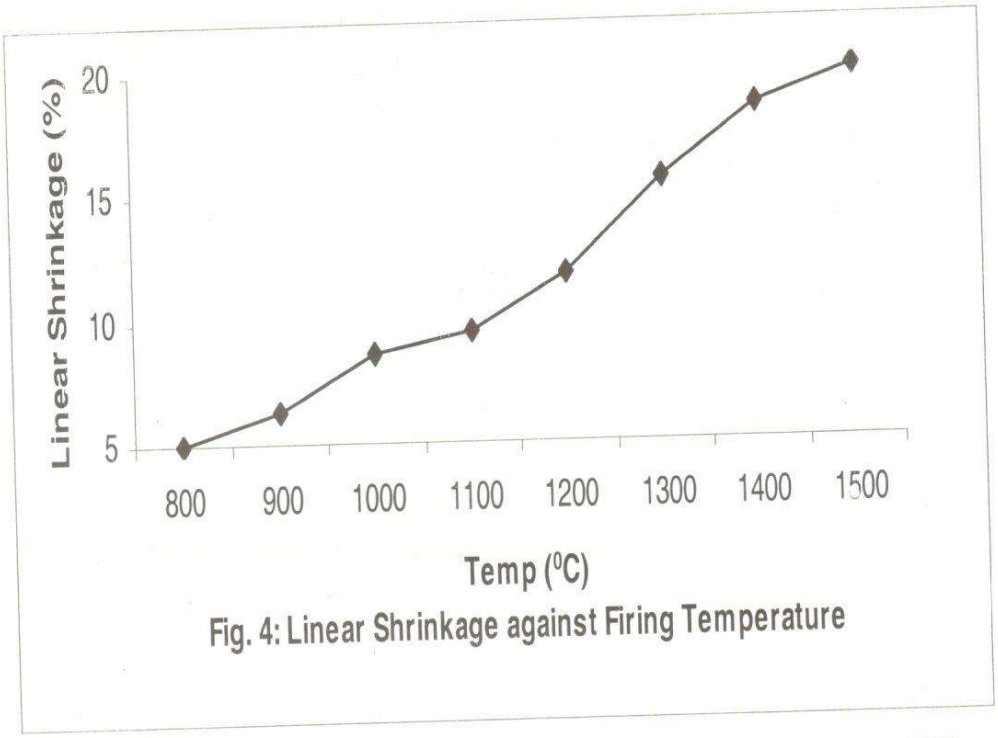


Fig. 4: Linear Shrinkage against Firing Temperature

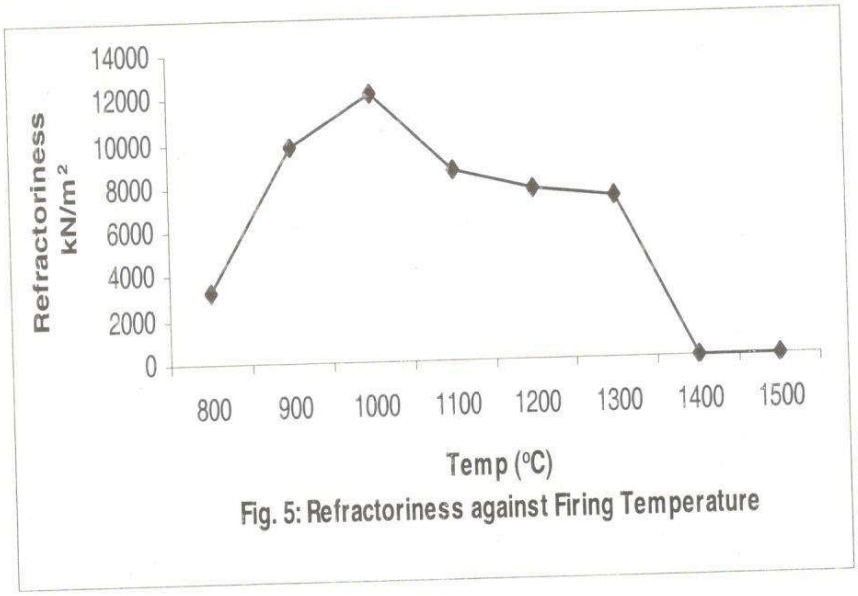


Fig. 5: Refractoriness against Firing Temperature

### 3.2 Discussion of Results

Table 1 gives the mineral constitution of the Osiele clay sample as determined via atomic absorption spectrometry. The sample main mineral constituents, alumina ($Al_2O_3$) and silica ($SiO_2$) are 30.24 wt and 55.78 wt percents respectively. The iron content is 0.85 wt percent. The composition of the sample in reference to the work of Amuda et al [7]

revealed that the sample is semi acidic alumino silicate and that the sample is of the kaolinite group due to the presence of alumina and silica. Alumina has a very high fusion temperature and is responsible for the refractoriness of clay. The higher the alumina in a particular type of clay the better the refractoriness. However, alumina is reputed to have relatively poor thermal shock resistance due to its higher thermal expansion and low thermal conductivity compared to other pure ceramic materials. Silica on the other hand is reported to have low expansion, remarkable thermal shock resistance, low thermal conductivity, excellent electrical insulation up to 1000°C and excellent resistance to corrosion from molten metal and glass. Beyond 1000°C, silica experience polymorphic transformation from quartz to $\alpha$- tridymite and later to $\alpha$-cristobalite at temperatures above 1470°C. At this level, the thermal expansion is between 13-17 percent and the clay sample is likely to start losing its thermal shock resistance integrity as a result of the large volume expansions associated with the inversions of the three minerals.

Analysis of Table 1 revealed that the ratio of $Al_2O_3/SiO_2$ is 30/56. Since the ratio is less than unit, it implies that the Osiele clay sample is expected to have a good thermal shock resistance on account of the low thermal expansion of the silica constituent of the clay in the sub 1000°C. However, beyond 1000°C, the thermal shock resistance of the clay is not guaranteed due to the polymorphic transformation that silica undergoes resulting in volumetric expansion of between 13-17 percent.

The very low iron content of 0.85 wt percent implies that during firing the sample will not change to red because iron III oxide is known to be responsible for red colour appearance of clays at the end of firing [9]. The presence of alkalis containing sodium and potassium is known to lower refractoriness of clay [10]. The two-percent distribution of potassium oxide may have the tendency of reducing the refractoriness of the Osiele clay. This is best prevented by pre-treating the clay sample by soaking in water for two or three days and then drying in the sun. The other trace oxide compounds in their combined proportion are not known to influence the refractory character of clays.

The refractoriness of the clay as determined by the (PCE) method is given in Table 2. The response of the clay to increased firing from 800 to 1500°C is determined by the colour variation and the existence of cracks and distortion in the clay at these firing temperatures. Table 2 revealed that at firing temperatures of 800°C – 1200°C, the colour of the sample was constant at white-brown. There was no crack in the sample at these

temperatures. This however changed at firing temperature of 1300°C. The colour of the PCE sample changed to yellowish brown with slight crack at the bottom. At 1400°C, the colour changed to ash with slight crack at both the top and bottom but no deformation was observed in the sample.

The ash colour was maintained at 1500°C but the clay not only cracked at this temperature, there was also deformation in all the edges of the clay. This shows that the refractoriness of the clay is within the 1300°C bracket. The crack and deformation that were observed at 1300°C, 1400°C and 1500°C respectively, results from the metakaolin [14] that was formed at pre 800°C and the spinel that was formed at pre 1200°C transforming to mullite and crystobalite inducing an expansion of 13-17 percent. This expansion introduced thermal stresses in the clay sample and this resulted in both the crack and deformation that was observed in the sample. The refractoriness evaluated in this study was true refractoriness under no load, if load bearing capacity at these firing temperatures had been investigated, the crack and deformation might occur at lower firing temperature.

Figure 1, is the graph of the variation of bulk density with firing temperature. The figure revealed that the bulk density decreases as the firing temperature increases. The decrease in bulk density with firing temperature was steady and became sharp beyond 1200°C. The bulk density measures the change in weight of clay with respect to the total volume of the material. The total volume is the sum of both closed and open pores. As firing temperature increases, there is release of chemically combined volatile matter leading to weight loss and the closure of internal pores. Besides, there was an expansion of between 13-17 percent when kaolin transforms to mullite and cristobalite. This result in the bulk density being progressively low as the firing temperature is increased. This agrees with the general knowledge that high temperature phases are always less dense than their low temperature counterpart. However, the least bulk density of 1.14g/cm$^3$ at firing temperature of 1500°C still falls within the standard bulk density specified by Chesti[15]. Therefore, the reduction in bulk density as firing temperature increases is not likely to have adverse effect on the structural integrity of the clay sample at these temperatures. This progression was also observed in Figure 2.

In Figure 2, the apparent porosity equally decreases as the firing temperature increases. The apparent porosity is a measure of tendency of gaseous substances to escape within the matrix of the clay material. It is a measure of passage between the matrix and the atmosphere in which the material is placed. As firing temperature proceeds beyond 1000°C, vitrification takes place; the clay structure

gradually breaks down into mullite and cristobalite structure with an amorphous or glassy phase which ultimately fills into the already open pores. The tendency for gaseous escape is thereby reduced. These phenomena of vitrification increases as the firing temperature increases. The apparent porosity is however found to be generally higher than the 20-30 percent standard for fired bricks across the firing temperatures.

The structure of brick has some influence on its load bearing capacity. A brick of high porosity will have less load bearing capacity than one of the same material with lower porosity, as there is less material in the brick to carry the load in the former case. Porous bricks are lighter and therefore do not need to carry heavy load [16].

Figure 3 is the graph of water absorption against firing temperature. The behaviour is similar to the situation observed in Figure 2. The increased closure of internal pores as firing temperature increases ensures that there is reduced space (through vitrification) for water to percolate through and as such reduced the water absorption rate.

Figure 4 presents the result for the linear shrinkage as firing temperature increases. The total shrinkage recorded (Table 2) in the clay sample increased from 5.01 at 800°C to 19.93 percent at 1500°C. The fired shrinkage is determined from the difference between the total and dry shrinkage. The firing shrinkage is very crucial; it gives an indication of the efficiency of firing. The values of total shrinkage recorded in the pre 1200°C firing temperatures falls within the 7-10 percent normal value for kaolin [17]. This implies that firing at temperature beyond 1200°C reduces the firing efficiency; and as such the clay sample is not recommended for use at temperatures beyond 1200°C.

The average cold crushing values generated from Osiele clay sample as firing temperature increases are graphically illustrated in Figure 5. The cold crushing strength first increased with firing temperature until 1100°C. At 1200°C, the cold crushing strength reached a turning point, beyond which a reduction in strength was noticed. The initial increase in strength was due to enhanced bonding brought about by sintering as the temperature increases. However, beyond 1200°C, the mullite/cristobalite transformation begins with its attendant expansion in the material and later fusion took place at 1500°C. This introduced intense volumetric expansion leading to contraction stresses. The crushing strength therefore reduces consequently.

## 4.0 CONCLUSION

Osiele clay has been characterized and strength properties evaluated. The clay is a semi-acidic alumino-silicate group kaolinite clay. The clay refractoriness is in the 1300°C bracket. The

clay has excellent cold crushing strength at pre 1200°C firing temperature. The water absorption rate, porosity and bulk density decreases as the firing temperature increases. The total shrinkage however increases as firing temperature increases.

The cold crushing strength, the porosity and the bulk density falls within standard for fired bricks in the pre 1200°C temperature range.

The property values from the investigation revealed that Osiele clay can be adopted as a refractory material for furnace lining in operating temperatures in the pre 1100°C.

**Acknowledgement**

The authors acknowledge the Technical Staff of the Ceramic Unit of the Federal Institute of Industrial Reasearch, Oshodi, Lagos, Nigeria, for the use of their facilities.

## APPENDIX

**Table 2: Measured Physico-Mechanical Properties of Osiele Clay**

| Property | Temperature °C | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | **800** | **900** | **1000** | **1100** | **1200** | **1300** | **1400** | **1500** |
| Bulk Density ($g/cm^3$) | 1.42 | 1.42 | 1.36 | 1.30 | 1.26 | 1.20 | 1.14 | 1.14 |
| % Apparent Porosity | 48.15 | 47.62 | 47.35 | 45.71 | 43.39 | 39.75 | 38.39 | 36.01 |
| % Dry Shrinkage | 4.21 | 4.86 | 7.09 | 7.66 | 9.53 | 12.25 | 14.57 | 15.68 |
| % Fired Shrinkage | 0.81 | 1.41 | 1.62 | 1.85 | 2.38 | 3.36 | 3.99 | 4.25 |
| % Total Shrinkage | 5.01 | 6.27 | 8.71 | 9.51 | 11.91 | 15.61 | 18.56 | 19.93 |
| Refractoriness $KN/m^2$ | 3250 | 9760 | 12200 | 8630 | 7770 | 7340 | - | - |
| % Water Absorption | 29.78 | 28.18 | 25.39 | 22.45 | 22.06 | 18.72 | 17.90 | - |
| Firing Colour | White Brown | White Brown | White Brown | White Brown | White Brown | Yellowish Brown | Ash | Ash |
| Crack Formation | No Crack | No Crack | No Crack | No Crack | No Crack | Slight crack @ the bottom | Slight crack @ the top and bottom | Crack but shows deformation on all edges |

## REFERENCES


1. NEEDS Document. (2004): Published by National Planning Commission, Abuja, ISBN 0-9741/08-41.
2. Adegoke, O. S. (1980): "Guide to the Non-Metallic Mineral Industrial Potentials of Nigeria". Proceeding of the Raw Materials Research and Development Council, pp. 110-120.
3. Borode, J. O. Onyemaobi, and Omotoyinbo, J. A. (2000): " Suitability of Some Nigerian Clays as Refractory Materials". Nigerian Journal of Engineering Management, vol. 3, pp. 14-18.
4. Raw Materials Research and Development Council (2003): Multi Disciplinary Committee Report of a Techno-Economic Survey on Non-Metallic Mineral Sector (4th Update). RMRDC – Abuja, ISBN: 978-8070-03-5.
5. Hassan, S. B. and Adewara, J. O. T. (1993): "Refractory properties of some Nigerian Clays. NSE Technical Transactions, vol. 28, No. 3, pp. 21-25.
6. Lawal, G. I., Amuda, M. O. H. and Adeosun, S. O. (2005): "The Effect of Cowdung and Graphite on the Properties of Local Refractory Clays". Nigeria Journal of Engineering Research and Development, vol. 4, No. 2, pp. 1-8.
7. Amuda, M. O. H., Lawal, G. I. and Majolagbe, F. O. (2005): Characterization of Some South West Clay Deposit". NSE Technical Transaction, Vol. 40, No. 4, pp. 13-23.
8. Imasogie, B. I. and Aramide, F. O. (2003): "Refractory Properties of Locally Sourced Materials for Lining Salt-Batch Austempering Furnaces", in the proceedings of the Nigerian Materials Congress Society of Nigeria, pp. 76-79.
9. Hassan, S. B. (2003): "The Effects of Silicon Carbide on Some Refractory Properties of Bauchi Clay" in the Proceedings of the Nigerian Materials Congress, Materials Society of Nigeria, pp. 80-84.
10. Odo, J. U. and Neife, S. I. (2003): "The Effect of Additives on Some Refractory Properties of Enugu Clay" in the Proceedings of the Nigeria Materials Congress, Materials Society of Nigeria, pp. 89-92.
11. Iloghalu, F. I. (2000): Improvement of Properties of Nigeria Clays for High Temperature Systems: Unpublished M. Eng. Thesis, Dept. of Metallurgical and Materials Engineering, Enugu State University of Technology.
12. Manual of ASTM Standards of Refractory Materials, 8th Ed. America Society for Testing Materials, Philadelphia.
13. Hussein, A. S. Eti, M. C. and Jombo, P. P. (2005): Investigation Journal of Engineering Research
14. Vaughan, F. C. (1958): Effect of Heat on Kandate Containing Clays, Transaction of Britain Ceramic Society 57, p. 39.
15. Chesti, A. R. (1986): Refractories, Manufacturing, Properties and Applications; Prentice Hall, New Delhi.
16. Norton, F. H. (1968): Refractories, 4th Ed. Pp. 30, 45-191; McGraw Hill, New York.
17. Thring, M. W. (1962): "The Science of Flames and Furnaces". 2nd Ed. Chapman and Hall Ltd. London.