International Journal of Materials, Methods and Technologies
Vol. 1, No. 10, November 2013, PP: 189-195, ISSN: 2327-0322 (Online)
Available online at http://ijmmt.com

**Research article**

# DEVELOPMENT OF REFRACTORY BRICKS FROM NIGERIAN NAFUTA CLAY DEPOSIT

**A. A. Jock[1*], F. A. Ayeni[1], L. S. Jongs [1], N. S. kangpe [2]**

[1]National Metallurgical Development Centre, Jos, Nigeria

[2] University of Jos, Jos-Nigeria

*Corresponding Author: alsanja@yahoo.com (+234 8036055325)

## Abstract

Refractories are important engineering materials and inputs into most industrial development. In this paper, the physico-chemical properties of clay from Nafuta in Barkin Ladi, Plateau State, Nigeria was investigated with a view to determine its suitability for use as refractory bricks. The results show that the silica content was the highest and that of titanium the lowest. The silica ($SiO_2$) content was found to be 62.26% followed by alumina ($Al_2O_3$) 29.44%, loss on ignition (LOI) 4.30% and trace amounts of impurities oxides such as $Fe_2O_3$, $TiO_2$, CaO, MgO, $Na_2O$ and $K_2O$ were (0.43, 0.05, 0.46, 0.33, 0.13 and 2.19% respectively). The clay has a refractoriness of 1600$^oC$ and a specific gravity of 2.6. Test bricks were produced from the clay alone and blended with 5, 10 and 20% rice husk. The results of the physical properties showed that the firing shrinkage ranged from 2.5 - 7.0%, bulk density was between 1.2 and 1.80g/cm$^3$. The apparent porosity was from 21.7 to 63.9% while the cold crushing strength was between 86.9kg/cm$^2$ and 156.5 kg/cm$^2$. Generally the results showed that the clay could be used as a refractory lining materials for furnaces, kilns, incinerators, reactors etc.



**Keywords:** Physico-chemical**,** Nafuta, Clay, refractory, Porosity, Kiln, Incinerators.

## Introduction

Refractories belong to the class of ceramic materials which are employed for high temperature applications, usually above 1100$^oC$ [1]. Most refractories are made from naturally occurring high melting point oxides of $SiO_2$, $Al_2O_3$, MgO, $Cr_2O_3$, ZrO, and refractory materials include aluminosilicate, dolomite, magnesia, silica, chrome, chrome-magnesite, carbon, etc. Refractories are used in almost every industry in which heat is employed such as in metallurgical, chemical, cement, glass and petrochemicals industries. They are used for the construction and maintenance of furnaces, kilns, reactors and boilers.

The best-known refractory is the fireclay which belongs to the aluminosilicate group of refractory materials. Clays are fine particle size materials comprising of clay minerals, which are basically hydrated aluminosilicates. Clay mineral groups are kaolin, smectite, palygorskite-sepiolite, illite, chlorite, and mixed-layered clays [2]. The properties of these clays vary in their structure and composition. They also contain non-clay minerals such as quartz, feldspar, mica, calcite, dolomite etc. Ball clay, flint clay and refractory clay (also known as fireclay) are varieties of kaolin which are used extensively in the ceramics industry because of their high fusion temperatures. Kaolins are also used as fillers in the paper, rubber, textile, pharmaceutical and numerous other industries. Kaolin for refractory application must have a high $Al_2O_3$ content as well as very low impurity oxides of fluxes ($Na_2O$, $K_2O$) and colouring ($Fe_2O_3$, $Fe_2O$, $TiO_2$) agents [3].

Clays of various kinds and grades abound throughout Nigeria's sedimentary basins and on the basement. A good number of clay deposits in the country have been studied, [4, 5, 6, 7, 8]. The abundance and availability of clay and their relatively low cost guarantee their continuous utilization. In this research, Nafuta clay and its blended form with rice husk was investigated for its potentials as refractory materials.

## Materials and Methods

### Sample Preparation.

The clay sample was randomly collected from the deposit site at Nafuta in Barkin Ladi of Plateau State, North – Central Nigeria. Rice husk was collected from Gaching also in Barkin Ladi. The clay sample was dried in the sun for two weeks, crushed, ground and sieved. The rice husk was also dried, sieved and graded to suitable sizes.

### Chemical Composition Analysis

Chemical analysis of the clay was conducted using Energy Dispersive X-Ray Fluorescence Spectrometer (ED – XRF) model PW1660, XRA and the following chemical components were determined in wt %: $SiO_2$, $Al_2O_3$, $Fe_2O_3$, $TiO_2$, CaO, MgO, $Na_2O$ and $K_2O$. The loss on ignition (LOI) was determined according to the Lechler and Desiletes method [9]. This involved measuring the weight loss of a known mass of the sample after firing in furnace at 1000$^o$C for an hour and calculated thus:

$$LOI, (\%) = \frac{W_i - W_f}{W_i} \times 100 \qquad (1)$$

Where,
$W_i$ and $W_f$ are initial and final weight respectively.

### Refractoriness

The Pyrometric Cone equivalent (PCE) was used to measure the refractoriness of the clay sample [10]. This method involved using some standard cones along with the moulded cones of the test clay sample. Both the standard and the moulded test cones were mounted on a refractory plaque and placed in PCE furnace. The temperature was raised to 200$^o$C at the rate of 5$^o$C per minute, with a gradual reduction in the rate till the tip of the test cone bent touching the refractory plaque. The plaque bearing the test cone was removed from the furnace and the test cone examined when cold. Refractoriness of the test cone was the number of the standard cone that bent to the same level as the tested cone and the temperature similar to the cone number was determined from the ASTM Orton series.

### Specific Gravity

The test piece was cut from within the core of a refractory shape moulded from the clay and crushed to a size not exceeding 3mm [11]. The crushed material was then mixed and reduced to a 50g sample by cone and quartering method. The sample material obtained was dried at 110$^o$C to a constant weight, and then 10 g sample was weighed using a glass stoppered weighing bottle. A pyconometer with stopper was dried at 110$^o$C, cooled in a desiccator and its weight (Wp) was noted. The pyconometer was filled with distilled water at room temperature; with its stopper put in place, the weight ($W_1$) was noted. The test refractory sample was put in dry pyconometer, covered with the stopper and weighed (W). The stopper was removed and distilled water was added to the sample to fill the pyconometer to its half capacity. This was gently boiled for 10 minutes to avoid loss of sample due to popping. The weight ($W_2$) of the pyconometer containing the test sample and the water was recorded. The specific gravity was calculated from the relationship:

$$Specific\ Gravity\ = \frac{(W - W_p)}{(W - W_p) - (W_2 - W_1)} \qquad (2)$$

Where,
W was the weight of test sample and dry pyconometer with stopper, $W_p$ was the weight of dry pyconometer with stopper, $W_1$ and $W_2$ were the weights of pyconometer with stopper filled with distilled water and pyconometer with stopper containing the test sample and distilled water respectively.

**Brick production**

The brick samples were produced from the based clay alone and by blending the clay with 5%, 10% and 20% of rice husk. The method employed in making the bricks followed this processes sequentially: crushing, grinding, sizing, mixing/blending and forming, drying, firing, and cooling [12, 13].
The clay sample was crushed, ground and sieved to three particles size fractions referred to as the coarse, medium and fine fractions. The particles were sieved to 2000µm, 710µm and 212µm to represent the coarse, medium and fine respectively. The recommended grading type given by Thring for 60 coarse: 10 medium and 30 fines was used [1]. The clay aggregates blended in this ratio was mixed with 5% of water and the mixture moulded using a hydraulic press. This process of brick making was also done by adding to the clay-water matrix 5%, 10% and 20% rice husk sieved to suitable sizes of coarse, medium and fine as shown in Table 1. The pressed samples produced were air-dried, followed by oven dried at 110$^o$C for 8 hours and fired in a muffle furnace at 1200$^o$C. The firing was controlled at a steady rate of 5$^o$C/minute to 1200$^o$C and soaked at this temperature for 2 hours. At this firing temperature, the ceramic bond must have being developed. The specimens were then allowed to cool gradually in the furnace overnight.

**Table1:** Test Bricks Formulation

| Specimen | Batch composition (%) | |
|---|---|---|
| | Clay | Rice Husk |
| A | 100 | 0 |
| B | 95 | 5 |
| C | 90 | 10 |
| D | 80 | 20 |

**Firing shrinkage:**

The firing shrinkage was determined from the brick specimens by marking 9cm length across the diagonal with a venier caliper. The brick samples were oven dried at 110$^o$C for 5hours and fired to temperature of 1200$^o$C at a standard rate of 5$^o$C/minute. The fired bricks were allowed to cool at room temperature in a dessicator and then weighed. The firing shrinkage was calculated by measuring the dimensional changes between the dried and fired bricks thus:

$$Firing\ Shrinkage\ = \frac{L_B - L_D}{L_B}\ 100 \qquad (3)$$

Where,

$L_B$ = Dry dimension

$L_D$ = fired dimension

**Apparent porosity**

The Porosity of the fired brick samples were determined by using the boiling method [1]. Each test specimen measuring 30mm x 30mm x 20mm was cut from the fired refractory brick and then dried in an oven at 110$^o$C 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 apparent porosity was calculated using,

$$Apparent\ porosity\ = \frac{W - D}{W - S} \times 100 \qquad (5)$$

Where, W = soakedor saturatedweight
D = Dried weight
S = suspended weight

**Bulk density**

The bulk density was also determined by using the boiling method [14]. A moulded fired brick specimen measuring 30mm by 30mm by 20mm was prepared. The brick was air dried for 24 hours and oven dried at 110$^o$C to a constant weight (D). After which it was transferred to a beaker and boiled with distilled water for 2 hours to assist in releasing trapped air. It was then allowed to soak and the saturated weight freed of excess water (W) was taken. The specimen was then suspended in water using a beaker and the suspended weight (S) was taken. The bulk density was then calculated using the relationship,

$$Bulk\ density\ = \frac{D}{W - S}\rho_w \qquad (4)$$

Where, D = Dried weight
W = Saturated weight
S = suspended weight
$\rho_w$ = Density of water

**Cold Crushing Strength**

This was the load at which cracks are produced in the specimen. The test piece was cut from the fired brick in the form of cubes of about 25 mm size. The test piece was marked to indicate the direction in which forming pressure was applied and the two faces normal to this direction were prepared as bearing faces. A cardboard was placed between the platens of the press and the bearing faces of the test piece. The load was applied at a uniform rate until the test piece failed to support the load. The maximum recorded load was taken as the crushing load and the area obtained from the size of the test piece before the application of load was calculated and recorded. The cold crushing strength was determined using,

$$Cold\ crushing\ strength\ = {}^{L}/_{A} \qquad (6)$$

Where, L = Maximum load (KN)
A = Cross sectional area

## Results and Discussion

## Results

The result obtained for the chemical analysis of Nafuta clay was as shown in Table 2, and that of the physical properties of the clay and its blended form was as shown in Table 3. Table 4 showed the established standard for refractory properties of fireclay.

**Table 2:** Chemical Analysis of Nafuta Clay

| Composition | $Al_2O_3$ | $SiO_2$ | $Fe_2O_3$ | $TiO_2$ | $Na_2O$ | $K_2O$ | MgO | CaO | LOI |
|---|---|---|---|---|---|---|---|---|---|
| Percentage (wt.%) | 29.44 | 62.26 | 0.43 | 0.05 | 0.13 | 2.19 | 0.33 | 0.46 | 4.30 |

**Table 3:** Physical Properties of Fired Nafuta Clay

| Properties/specimen | A | B | C | D |
|---|---|---|---|---|
| Specific gravity | 2.6 | - | - | - |
| Refractoriness ($^oC$) | 1600 | - | - | - |
| Linear shrinkage, (%) | 2.5 | 3.0 | 5.0 | 7.0 |
| Bulk density ($g/cm^3$) | 1.8 | 1.6 | 1.5 | 1.2 |
| Apparent porosity (%) | 21.7 | 29.9 | 40.2 | 63.9 |
| CCS ($kg/cm^2$) | 156.5 | 130.4 | 104.3 | 86.9 |

**Table 4:** Established standard for refractory Properties of fire clay

| Properties/specimen | Fireclay | Insulating Fireclay |
|---|---|---|
| Specific gravity | 2.6 – 2.7* | - |
| Refractoriness ($^oC$) | 1580-1750* | - |
| Linear shrinkage, (%) | 7-9* | - |
| Bulk density ($g/cm^3$) | 1.98** | 0.85-1.5* |
| Apparent porosity (%) | 8-24* | >60** |
| Cold Crushing Strength ($kg/cm^2$) | 140-840** | 3.5-35.1** |

Source *Misra and **Chesti

## Discussion

### Chemical analysis of the sample

The analysis of the Nafuta clay sample by X-ray fluorescence in Table 2 shows that $SiO_2$ and $Al_2O_3$ are the predominant constituents, followed by the loss on ignition and $K_2O$. The clay sample is 29.44% $Al_2O_3$ content falls within the range value of 25-45% $Al_2O_3$ for fireclay refractories [14]. The higher the $Al_2O_3$ contents in clay, the higher the refractoriness. The high concentrations of $SiO_2$ (62.26%), $Al_2O_3$ (29.44%) and LOI (4.3%) suggests that the clay is a hydrated alumino-silicate type of minerals. It also suggests it is of the kaolins group which are principally composed of silica ($SiO_2$), alumina ($Al_2O_3$) and water ($H_2O$) with a chemical formula of $Al_2O_3.2SiO_2.2H_2O$.
The results also indicated the presence of oxides of impurity such as $Fe_2O_3$, $TiO_2$, $Na_2O$, CaO and MgO from minor to trace amount levels, except $K_2O$. Generally, these oxides are low but the sum total of all is within acceptable limits of clays for refractories use with a total allowable of 2-5% [14].

### Physical Properties of Test Samples

The result obtained for the physical properties of the clay and blended form is shown in Table 3. Batch A of the specimen was produced from the based clay alone while batches B, C and D were produced from the based clay blended with 5%, 10% and 20% of rice husk respectively.

The result of the specific gravity (2.6) and the refractoriness (1600$^oC$) of the clay in Table 3 both falls within the range for fireclay refractories as shown in Table 4. The relatively high refractoriness can be attributed

reasonably due to high alumina content of 29.44% and low amount of impurity oxides as their presence in high amount usually have an adverse effect on the refractoriness.
The firing shrinkage of clay is a very useful and relevant property in the production of refractory bricks. The firing shrinkage of the samples with a value range of 2.5 to 7% is also found within the allowable limit of 7-9% for fireclays bricks as shown in Table 4. However the firing shrinkage values increase with increasing rice husk. Sample A without adding rice husk has the lowest shrinkage value while sample D with 20% rice husk has the highest shrinkage value (Tables 1 and 3). The increase in firing shrinkage with increasing rice husk is because the rice husk which occupied spaces in the clay body burnt off on firing and thereby leaving open space which caused shrinkage to occur.
The porosity of sample A is the lowest (21.70%) while sample D is the highest (63.89%). This also entails that is porosity increases with increasing rice husk. This indicates that porosity can be induced in a body by addition of organic and combustible substance to the based clay. The clay acts as a bond and the rice husk gives the cellurity. The more the rice husk, the more the cellurity created and the higher the porosity as shown in Table 3. Samples A and D have porosity values within the recommended for fireclay and insulating fireclay respectively as shown in Table 4.
The bulk density decreases with increasing rice husk with sample A having the highest bulk density and the lowest in sample D (Table 3). The decrease in the bulk density with increasing rice husk could be explained with the fact that the rice husk which is a combustible matter burnt off on firing thereby making the sample to lose weight.
Cold crushing strength is the load at which cracks appear in the specimen and these values also decrease with increasing amount of the rice husk. The cold crushing strength of sample A with no rice husk is the highest and is above the minimum recommended value for fire clay while sample D with the highest amount of the rice husk has the lowest cold crushing strength as shown in Table 3. These cold crushing strength values however showed that the Nafuta clay with or without rice husk could comfortably withstand impacts at low temperatures as the cold crushing strength is an indicator of the effect of firing on ceramic bond.

## Conclusions

Nafuta clay was subjected to chemical and physical tests to investigate its potential as refractory material. The study conducted reveals that the clay has alumina content that is adequate for some refractory purposes, low amounts of the fluxing oxides and high refractoriness. The result also shows that the physical properties of the clay are within the allowable limit for fireclay refractory. The cellurity introduced by the addition of rice husk also suggested its suitability for making insulating fireclay bricks, with the purpose of preserving heat. Generally, on the basis of the physico-chemical characteristics conducted, Nafuta clay or its blended form with rice husk could be used as a back lining for the refractory bricks which would indirect or direct be in contact within the heat and the hot material inside the furnace, kilns, reactors, ladles, oven etc.

## References

[1] S. B. Hassan, “Modern Refractories: Production, Properties, Testing and Application”. Timo Commercial Printers Samaru, Zaria, first edition, 2005, pp 13-22, 28-40.

[2] S. H. Patterson and H. H. Murray; Kaolins, Refractory clay, ball clay and Halloysite in North America, Hawaii and Carribean Region, Geo. Surv. Pro; Paper No. 1306, USA pp 56.

[3] F. H. Norton; Clays, McGraw-Hill Book Co, New York, 1973, pp 67-72

[4] National Metallurgical Development Centre, (NMDC), Jos; Data Bank of Raw materials for the Metallurgical Industry, 2008, pp56-60

[5] A. Adekanmbi, “The Nigerian Steel Industry: Evaluation and Raw materials Development, A Historical Excursion (Emphasis on Ajaokuta Steel Project)”, XcessAmonqe Press Ibadan, Nigeria, first edition, 2005, pp 172-185.

[6] J. O. Borode, O.O. Onyemaobi, J.A. Omotoyinbo, “Suitability of some Nigerian Clays as Refractory Raw Materials”, Nigerian Journal of Engineering Management, Vol. 1, 2000, pp 14-18

[7] S. B. Hassan, and J. O. Adewara, "Refractory Properties of some Nigerian Clays", Nigerian Society of Engineers Technical Transaction, Vol. 29, No. 3, 1994, pp 13-19.

[8] P. S. Irabor, "Physical and Chemical Investigation on some Nigerian Kaolinite Clays for use in the Ceramics and Allied Industries", Nigerian Journal of Engineering Research and Development, Vol. 1, No. 1, 2002, pp 54-59.

[9] P. J. Lechler and M. O. Desilets; A review of the use of loss on ignition as a Measurement of total volatile in whole rock analysis, Chemical Geology, 63(13) pp 341-344.

[10] M. I. Misra, "Refractories: Their manufacture, Properties and Uses" SMT, Lakshmi Misra, Krishna colony, Ghamapur, Jabalphur, fourth edition, 1975, pp 8-13, 22-42.

[11] ASTM Standards Part 17, "Refractories, Glass, Ceramic Materials, Carbon and Graphite Products" ASTM Philadelphia, 2005,pp 7-9, 51-61.

[12] J. S. Reed, "Introduction to the Principles of Ceramic Processing", John Wiley and Sons, New York, 1986, pp 32, 154, 330.

[13] J. H. Chesters, "Refractories: Production and Properties", The Iron and Steel Institute, England, 1973, pp 3-13, 296-312.

[14] A. R. Chesti "Refractories: Manufacture, Properties and Applications" Prentice-Hall of India private Ltd, New Delhi, 1986, pp 55-63.

[15] R. W. Grimshaw; The Chemistry and Physics of Clay and Allied Ceramic Materials, 4th Edition, Revised. New York, Willey Inter Science, 1971, pp 32-45.

[16] J. P. Gilchrist, "Fuel, Furnaces and Refractories" Pergamon Press, 1977, pp 35-70