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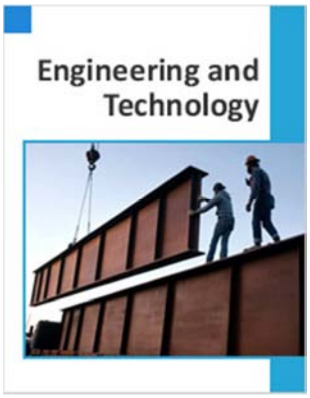

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Analysis and Categorization of Akparavini and Okwuboyere Clay Deposits in Biase LGA of Cross River State-Nigeria

Ovat Friday Aje*, Bisong Maria-Kaka Achu

Department of Mechanical Engineering, Cross River University of Technology, Calabar, Nigeria

Email address
fraijoe@yahoo.com (O. F. Aje), mkbisongachu1@gmail.com (B. Maria-Kaka A.)
*Corresponding author

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Abstract
Apart from petroleum and gas exploration, mining and exploitation of Nigerian mineral resources have not received sufficient attention. In this present study, the analysis and categorization of Akparavini and Okwuboyere clays for industrial applications was carried out. Samples from these locations were collected and analysed for their physical and chemical properties to determine their suitability for industrial applications. The experimental result of the chemical analysis of the clays showed silica (50.09%) and alumina (20.09%) as the major constituents while other metal oxides such as Fe_2O_3 (7.97%), CaO (1.01%), K_2O (2.56%) and Na_2O (0.95%) were present in appreciable quantities for Akparavin and silica (57.92%) and alumina (29.21%) as the major constituents while other metal oxides such as Fe_2O_3 (6.48%), CaO (0.81%), K_2O (2.11%) and Na_2O (0.88%) for Okwuboyere respectively. There was also variation in the physical properties of the clays up to 1600°C. The result of this study showed that Akparavini and Okwuboyere clays have good industrial potentials and can be used in the manufacture of ceramics, high melting clays, refractory, bricks, tiles and colour vase. This can help reduce the bulk of clay minerals imported into Nigeria.

1. Introduction

Researches have been carried out to give adequate and satisfactory data as regards the properties which will enable our local clay to be used in the manufacture of refractory bricks used by industries in Nigeria. The researches made are mostly in the Northern and Western parts of the country. In other regions like the South East and the South-South, there have been little or no research made. This may be due to either lack of interested persons in the discipline or inadequate funds to support the efforts made.

Clay deposits have been identified in many regions in Nigeria though with differing properties probably owing to geological differences [1]. Furthermore several studies have been done on clay soils around Nigeria. They include Mayo-Belwa in Adamawa [1], Kachia, Kafanchan, Wusasa in Kaduna [2]; Sheda, Abajiand Karimu in Abuja [2]; Olokoro, Ukpor, Otamiri and Nsu in South eastern Nigeria [3]; Abakiliki in Ebonyi [4]; clay deposits in Ekiti, south-west Nigeria [6] and Ugbegun clay deposit in Edo central Nigeria [5] amongst others.

It has been found that refractory materials have properties which determine their applications; they have both physical and chemical properties. The chemical properties help determine the physical properties, so that by altering the chemical properties we obtain different values of physical properties, and then different applications. The diversification on steel products and their cleanliness requirements in recent years have increased the demand for high quality refractory. Steel making requires high temperatures of the order 1600°C. High quality refractory at a cheaper cost is the main requirement because refractory adds to the cost of products [7].

Refractory materials are made from clay and are a set of materials that retain their strengths at high temperatures. They are non-metallic materials having those physical and chemical properties that make them applicable to structures or as components of systems that are exposed to high temperature applications. A material is refractory if it has a very high melting point in addition to its physical and chemical properties that makes it suitable for use in kilns, furnaces, reactors and other high temperature vessels [8]. According to them, refractory materials are materials capable of withstanding high temperatures, and that high quality refractory materials resist high temperature fluctuations between 1000°C and 1500°C and are also good thermal and electrical insulators.

It was observed by [9], that the raw materials for the production of various types of refractory materials are kaolinite ($Al_2O_3.2SO_2.2H_2O$), Chromites ($FeCr_2O_3$), Magnesite $MgCO_3$ and various types of clays. He also noted that other additives such as sawdust, graphite and some binders are available locally. Nnuka and Enejor, [10] studied the characteristics of Nigerian clays and discovered that the Otukpo clay can withstand a furnace temperature of about 1710°C which compared favorably with imported refractories. Amuda [11], in an earlier research on the characterization and evaluation of refractory properties of some clay deposits in Southwest Nigeria, reported that the clays displayed reasonable refractory properties that compared favorably with standard values and recommended a blend of these clays for furnace lining. Meanwhile, [12] determined the moulding properties of locally available clays for casting operations and discovered that the Barkinladi and Alkaleri clay samples were suitable for construction of furnaces and furnace lining.

From findings made by [13], on the analysis and characterization ofsome selected North-west clay deposits showed that the clays could be used for production of ceramic, basic refractory, mortar lining, kilns and also as refractory bricks for furnace lining.

In terms of chemical composition, refractories composed of either single or multi-component inorganic compounds with non-metallic elements [14]. They are classified as either acid refractories, basic refractories and/or neutral refractories. The Acid refractories are a set of refractories used where slag and atmosphere are acidic. They cannot be used under basic conditions [6]. Irabor [15], noted that the main raw materials used for the production of these acid refractories are silica, ZrO_2 and alumino-silicate; typical refractories are fire clay, quartz and silica. Basic refractories are produced from a composition of dead-burnt magnesite, dolomite, chrome ore. The applications of various refractories and concluded that Magnesite, chrome combinations have good resistance to chemical action of basic slag, have good mechanical strength and volume stability at high temperatures, chrome-magnesite refractories used for side walls of soaking pits, magnesite with varying amounts of carbon has excellent resistance to chemical attack by steel-making slag [7].

Neutral refractories are chemically stable to both acids and bases and are manufactured from Al_2O_3, Cr_2O_3 and Carbon [8].

Physically refractory materials are either bricks or monolithic. Shaped refractories are in the form of bricks of some standard dimensions [16]. Special shapes with required dimensions are hand-molded and are used for particular kilns and furnaces. Ramming refractories are in loose dry form with graded particle size and are often mixed with water for use. Investigation [17], revealed that castable refractory materials contain binders such as aluminate cement which imparts hydraulic setting properties when mixed with water; the materials are installed by casting and are also known as refracting concretes. Onyeji [18], on the analysis and characterization of Nyikangbe clay deposit noted that, mortars are finely ground refractory materials which become plastic when mixed with water and are used to fill gaps created by a deformed shell, and to make walls gas-tight to prevent slag penetration.

Bricks are joined with mortars to provide a structure. Plastic refractories are packed in moisture-proof packing and pickings are opened at the time of use. Plastic refractories have high resistance to corrosion. Study by [19], revealed that monolithic refractories are replacing conventional brick refractories in steel making and other metal extraction industries. He gave the main advantages of monolithic refractories to include greater volume stability, better spalling tendency, elimination of joints compared with brick lining, and can be installed in hot standby mode.

2. Materials and Methods

2.1. Sample Collection

Clay samples were collected from different locations of Akparavini and Okwuboyere, in the Southern Senatorial district of Cross River State. The samples were randomly collected from different points at the depth of 50cm.

2.2. Sample Preparation

Samples of about 3kg were collected and air-dried for five (5) days. Samples were finely crushed to achieve homogeneity of the particles sizes. The ground samples were sieved with a mesh of 1.13µm. The ground and sieved samples were mixed with some quantity of water to a pastry form. The pastry sample was moulded (70x50x50mm) into bricks and compacted with a hydraulic press according to

ISO standards. The different clay samples were subjected to both physical and chemical tests. The equipment used during the test included Son Holland-Telex 59388 Furnace, Oven, Navigator N3B110 Ohaus Electronic weighing machine, crusher, bell jar, dessicator, X-ray Spectrometer. These equipment can be found in the laboratory of Bao Yao Huan Jian Iron and Steel Company, Export Processing Zone (EPZ) Calabar, Cross River State-Nigeria.

2.3. Physical Analysis

2.3.1. Cold Crushing Strength

Test pieces measuring measuring 75mm by 50mm were prepared and are-dried for 24 hours thereafter transferred to a furnace and heated for a period of 6 hours and at a temperature of 1600°C. After the heating process they were removed from the furnace and allowed to cool at room temperature and each piece was in turn place in a crusher. Load was applied axially and continually until the test piece failed. The procedure was repeated for other test pieces. The respective loads at which each test piece failed were recorded. The cold crushing strength (CCS) was calculated from the equation (1).

$$\text{CCS} = P/A \tag{1}$$

Where A = Area of specimen
P = Load applied

2.3.2. Thermal Shock Resistance

75mm × 50mm ×50mm test pieces from the different sources were inserted in a furnace maintained at 1600°C for a period of about 10 minutes. Thereafter, they were removed and allowed to cool in a dessicator for 10 minutes and then returned back to the furnace (still at 1600°C) for another 10 minutes. The process was repeated until the pieces were comfortably deformed when a small force was applied. Then the number of heating and cooling cycles for each of the specimen was recorded.

2.3.3. Linear Shrinkage

The samples were prepared and their original lengths of 75mm recorded. They were dried in the air for 24 hours and then dried in an oven at 110°C for 24 hours. They were then transferred to a furnace maintained at 1600°C and heated for a period of 6 hours. They were then brought out by means of pair of tongs and allowed to cool in a dessicator. The linear shrinkage was determined using equation (2).

$$\text{Linear Shrinkage} = \frac{L_D - L_F}{L_D} \times 100 \tag{2}$$

where L_D = Dried or original length
L_F = Fired length

2.3.4. Bulk Density

After air-drying for 24 hours, the test pieces were then put in an oven air dried at a temperature of 110°C for 6 hours. They were allowed to cool and weighed using weighing balance and their dried weights (D) were recorded in turn. They were then transferred to a beaker and heated for 40 minutes and cooled, the soaked weight was recorded. Water was put in another beaker and each of the test pieces in turn suspended in the water so that their suspended weights was recorded. The equation for calculating bulk density is given in equation (3).

$$\text{Bulk Density} = \frac{D\rho_W}{W - S} \tag{3}$$

Where D = Dried weight
ρ_w = Density of water
W = soaked weight
S = suspended weight

2.3.5. Apparent Porosity

Test pieces from the different locations were prepared and allowed to dry in the air for a period of 24 hours before transferring them to the oven and drying for another 24 hours at 110°C. They were thereafter transferred to a furnace and fired to a temperature of 1600°C, after which they were removed by pair of tongs and allowed to cool in open air before weighing them in a dessicator and noted the dry weight (D_W). Bubbles were observed as the pores in the samples were filled with water. The bubbles were made to escape through periodic agitation and after about 40 minutes, and then weighed in the dessicator so that their soaked weight(S_W) were noted. Also, by means of beaker placed on a balance, the specimens were weighed suspended in water and the suspended weight (W) was taken. The apparent porosity for each location was determined from equation (4).

$$\text{Apparent porosity} = \frac{S_W - D_W}{S_W - W} \times 100 \tag{4}$$

where S_w = Soaked weight
D_W = dried weight
W = suspended weight

2.3.6. Permeability

After preparing various samples, they were air-dried for 24 hours and thereafter oven dried at 120^{0}C for 12 hours. 2000cm^3 of air held in a bell jar was forced to pass through the specimen. At this time, air entering the specimen was equal to that leaving the specimen. The pressure difference was read by a manometer, and noted. Also, the time taken for the 2000cm^3 of air to pass through the specimen was recorded. The respective permeabilities were calculated from equation (5).

$$\text{Permeability number, } P_n = \frac{Vh}{APt} \tag{5}$$

Where P_n = permeability number
V = Volume of air, cm^3
H = height of specimen, cm
A = Cross sectional area of specimen, cm^2
P = pressure of air in cm of water, N/cm^2
T = time, minutes

2.3.7. Modulus of Rupture (MOR)

Different test pieces of clay bar, 75mm × 50x50mm dimension were prepared, the dry samples were moistened and mixed to a workable state. The wedged samples was cast in wooden mold, coated with thin film of machine engine oil. The bars were (temperature marked) then charged into an electric furnace separately along with American standard pyrometric cones of refractoriness 1600°C and fired for approximately ten hours, removed from the furnace and allowed to cool. Each batch of bars were broken at the center bending on a Denison strength testing machine at 7.0 span and MOR was calculated from the expression in equation (6).

$$\mathrm{MOR} = \frac{3PL}{2bh} \tag{6}$$

Where p = Breaking Load in kgf, L = Distance between support, b = Breath, h = Height.

2.3.8. Modulus of Plasticity (MOP)

The molded clay was deformed by dropping onto it from a fixed height of a flat-headed plunger of known weight. The distance traveled was read from the graduated scale. The modulus of plasticity (MOP) for the clay sample was obtained from the expression (7);

$$\mathrm{MOP} = \frac{\mathrm{OriginalHeight}}{\mathrm{DeformedHeight}} \tag{7}$$

Also, the percentage making moisture for the clay samples were obtained from the expression (8);

$$\%\ \mathrm{Making\ Moisture} = \frac{\mathrm{WetWeight} - \mathrm{DryWeight}}{\mathrm{NetWeight}} \tag{8}$$

2.3.9. Loss on Ignition (L.O.I)

The weight of an empty porcelain crucible was determined and recorded as M_1 (g), 3.0 g of the dried ground clay was added and the weight of the crucible and clay was determined, M_2 (g). The samples were then ignited in the laboratory kiln at 1600°C. After the cooling of the samples, the weight of the crucible and samples after ignition were determined, M_3 (g). The loss on Ignition was then calculated using equation (9).

$$\text{Loss of ignition} = \frac{M_2 - M_3}{M_2 - M_1} \times 100 \tag{9}$$

Where, M_1 = Mass of dried porcelain crucible M_2 = Mass of sample clay and porcelain crucible

M_3 = Mass of heated clay sample and porcelain crucible

2.4. Chemical Analysis

The Chemical Analysis of the clay samples was carried out by means of x-ray Fluorescence Spectrometer (XRFS). The technique was used to determine the concentrations of elements present in the various samples. The samples were ground and sieved to produce 75µm particle size. Thereafter, 2grams of the sieved samples was intimately mixed with 5grams of lithium tetraborate binder ($Li_2B_4O_7$) anhydrous solution that acts as a fluxing agent. It was thereafter pressed to a pellet in a mould and then dried in oven at 120°C for 20 minutes to remove the absorbed moisture and then kept inside a desicator. The spectrometer was turned on and allowed to warm up to stabilize the optics and x-ray tube. The samples were placed in turn in the machine and the elemental concentrations present in the samples were displayed on a monitor. The result of the analysis is as given in Table 2.

3. Results and Discussion

3.1. Results

The results obtained from the various tests carried out for physical and chemical properties are shown in Table 1 and Table 2.

Table 1. *Physical Properties of Akparavini and Okwuboyere clays from Biase LGA of Cross River State.*

Properties	Okwuboyere	Akparavuni
Cold Crushing Strength (MN/m²	21.46	21.84
Thermal shock Resistance (Cycles)	24.00	20.00
Linear Shrinkage (%)	8.10	7.81
Bulk Density (g/ cm³	2.36	2.90
Apparent Porosity (%)	24.56	17.24
Permeability (%)	65.00	75.00
Modulus of rupture KgF/cm²	45.10	48.00
Modulus of plasticity Kgf/cm³	3.57	3.42

Table 2. *Chemical Composition of clay samples from Akparavini and Okwuboyere clays in Biase LGA, Cross River State.*

Elements/Compound	Akparavuni	Okwuboyere
Al_2SO_3	20.090	29.212
SiO_2	50.089	57.922
MgO	0.113	0.131
CaO	1.012	0.812
Fe_2O_3	7.971	1.880
K_2O	2.561	2.120
Cl	0.029	0.015
Co	0.013	0.017
MnO	0.108	0.055
Cr_2O_3	0.037	0.055
Na_2O	0.950	0.881
Ni	0.071	0.088
Cu	0.147	0.036
P_2O_5	0.059	0.057
SO_3	0.026	0.038
Sr	0.025	0.029
TiO_2	1.587	1.532

3.2. Discussion

Linear shrinkage

The linear shrinkage is an indicator of the firing efficiency of the clay samples. Okwuboyere, and Akparavuni have high values of linear shrinkage as 8.10% and 7.73% respectively, but falls within the accepted range of 7-10% as noted by [9]. He also noted that higher values of linear shrinkage may result in warping and cracking of the brick which may cause loss of heat in the furnace. However, [12], pointed out that lower values were more desirable as this means the clay is less susceptible to volume change. That means the values obtained from Akparavuni having shrinkages of 4.46% will make for very good refractory materials.

Bulk density

The bulk density is a measure of the weight of a given volume of refractory and is often considered in conjunction with porosity. In comparison to clays from other regions of Nigeria, these are quite dense when compared with values obtained from Gur and Yamarkumi clays (2.06-2.11 g/cm^3), Plateau and Bauchi clays (1.94-2.04 g/cm^3), as respectively reported by [1] and [12]. Ito clays are more likely to shrink with their respectively reported bulk densities of 1.64 g/cm^3 and 1.73 g/cm^3.

Apparent porosity

The values for Okwuboyere and Akparavini fall within the internationally accepted range of 20-30% as was noted by [9].

Thermal shock resistance

The thermal shock resistance determines the ability of the material to withstand repeated heating and cooling cycles. Okwuboyere, Akparavuni, Idere and Ito have values lower than the recommended range of 25-30cycles.

Cold crushing strength

This is a measure of the ability of the clay to withstand abrasion and loading. Values that are recommended are above 18MN/m^2 and of the areas investigated, Okwuboyere and Akparavuni have all met the standard. In other regions, [8], reported an impressive 26.5 MN/m^2 for Nsu clay, while [22], reported 18.065 MN/m^2 and 16.620MN/m^2 for Nyikangbe and Beji clays.

Permeability

Is the measure of the ability of the clays to prevent gases and liquids from penetrating the The permeability numbers of all the samples fall within the standard range of 25-90%. given by [9]. Refractories under the influence of liquids and gases should be impervious, as this will help to eliminate leakage of gases and penetration of liquids through the walls of the furnace.

Chemical composition

The experimental results of the chemical analysis of the clays showed silica (50.09%) and alumina (20.09%) as the major constituents while other metal oxides such as Fe_2O_3 (7.97%), CaO (1.01%), K_2O (2.56%) and Na_2O (0.95%) were present in appreciable quantities for Akparavin and silica (57.92%) and alumina (29.21%) as the major constituents while other metal oxides such as Fe_2O_3 (6.48%), CaO (0.81%), K_2O (2.11%) and Na_2O (0.88%) for Okwuboyere respectively. It was observed that Okwuboyere and Akparavini clay samples as reported in Table 2 are more kaolinitic. The presence of Fe_2O_3 contributed to the reddish colour of both clay samples from grey. This colour changed after the firing process when carbonaceous materials and the iron began to oxidize which agrees with [21]. The proportion of elemental composition of iron in clays determines the thermal conductivity potential of such materials, hence [20], suggested that any such fire clays can be used as refractories. However, with the use of the XRFS equipment for the chemical analysis, other compounds and elements were found to exist in the samples tested though in very low percentages as shown in Table 2.

4. Conclusion

This study has shown that the Mbayion clay sample has high silica and Alumina content which makes it kaolinite clay. Furthermore the moderately lower content of alkali oxides (CaO, MgO, K_2O and Na_2O) in the clay qualifies it for use as refractory materials Kilns lining and for the manufacture of floor tiles and bricks making.

These potentials therefore qualify the exploration of these clay deposits for Industrial applications by the Cross River State government for economic development and job creation for our teeming graduates in the state and the nation at large.

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