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CHARACTERIZATION OF UJOGBA CLAY DEPOSITS IN EDO STATE, NIGERIA FOR REFRACTORY APPLICATIONS

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ABSTRACT

Ujogba clay deposits in Esan Land, Edo State of Nigeria were characterized for refractory and other applications. The characteristics investigated were mineralogical composition, chemical composition, linear shrinkage, apparent porosity, bulk density, cold crushing strength and density. The physical property tests were carried out in accordance with ASTM C71. Results obtained showed that the Ujogba clays are largely kaolinite and chemically composed of mainly silica (68.93 %) and alumina (24.61 %). These oxides appreciated to 71.04 % (silica) and 19.63 % (alumina) in the fired product; yet the ratio of alumina to silica remained unchanged. The physical property such as refractory property examination confirmed that the clays belong to the fireclay class and are useful for refractory and ceramic applications.

Keywords: Characterization, refractory applications, Linear shrinkage, Apparent porosity, Bulk density, Cold crushing strength, Density

INTRODUCTION

Refractory materials are non-metallic, inorganic substances, mainly mixtures of oxides, which are capable of withstanding very high temperature conditions without losing their chemical and mechanical integrity. The common oxides found in refractory mixtures are oxides of silicon and aluminium. Naturally, silica (SiO₂) and alumina (Al₂O₃) are the main constituents of all alumino-silicate minerals, particularly clays. The amount of alumina in a clay mineral determines its potential use as an alumino-silicate refractory raw material. Kaolinites have been found to possess the highest content of alumina (up to 39.50 % by weight), as revealed by the chemical formula, Al₂O₃.2SiO₂.2H₂O [1]. Refractories are indispensable materials used in high temperature applications such as metal extraction/refining, sanitary ware, table ware, wall and tiles, construction materials, electric porcelains insulator, for materials glazing and metallurgical heat treatments, foundry melting practices and power generation [2].

The refractory need of Nigeria; a developing industrial nation is potentially enormous. It was estimated that the Ajaokuta Steel Company and Delta Steel Company will, at full capacity, respectively require 43,503 and 25,000 tones/year of fireclay refractories for their activities and these products are sourced from abroad [3,4]. However, small-scale industries in Nnewi and elsewhere in the country have recently embarked on the fabrication of spare parts. The spare parts are fabricated using high temperature furnaces (foundry melting furnaces and heat-treatment furnaces) that require refractories as linings. Most of the refractories consumed in this country are sourced from abroad whereas there are many clay deposits in Nigeria that could be used as raw materials for refractory fabrication, one of such is the Ujogba clay in



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Edo State [5-6]. Report in literature reveals that in 1987 alone, Nigeria imported 27 million metric tons of refractories. The country expends a lot of foreign exchange for refractory importation. Yet, a lot of clay deposits abound in the country, which can be developed to meet our local needs. Earlier works in literature on various Nigerian clay deposits have shown that many of them are richer in silica content than in alumina [7].

Many of those deposits can be processed to make them suitable for use as refractory materials. Therefore, the development of our local materials for the production of refractories to meet local industrial and technological requirements which can later serve as means of foreign earnings is not only justified but imperative especially during this period when Federal Government of Nigeria is embarking on economic diversification due to current met-down from oil sector.

With that regards, this research project is a contribution in the characterization of refractory materials. The evaluation of some refractory characteristics of kaolinite clay deposits in Edo State is a focus of the present study. The properties examined are plasticity index, linear shrinkage, apparent porosity, bulk density, cold crushing strength, thermal shock, and refractoriness (softening point); in addition to chemical and mineralogical analyses of the clays [8, 9].

MATERIALS AND METHODS

Materials used in this research work are Ujogba clays obtained at two different sites A and B in Igweben L.G.A, Edo State, Nigeria as shown in Plate 1.

Major equipment used includes: Hydraulic press and electric furnace.

a. Hydraulic press specification:

WEBER-HYDRAULIC AC-8800 Vibrorg, Denmark TYPE: P-16H CAPACITY: 16t Serial No: 29580

b. Electric furnace:

NABERTHERM MORE THAN HEAT 30-3000⁰C



Plate 1. Clay photographs obtained at different sites A and B



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Each of the clay samples was soaked in water for 24 hours and classified using 100 µm sieve. The clay slurry obtained was allowed to settle. The settled clay was poured into a plaster of Paris (POP) box after decantation of the upper liquid layer. Wet clay obtained after moisture absorption by the POP was oven dried at 100 °C for hours. The dry clay lump was crushed with a hammer mill machine (model: 000T, PUISSANE: 1,5KV, NO: 13634), into granulated particles size of less than 2 mm, before finally pulverized into powders using ball milling machine (model: 87002.....Limoges-France, type machine: A50....43) in accordance with [10-15]. The powders were sieved using 100 µm mesh size sieve shaken with a vibro sieve shaker (model Fritsch GmbH, D-55743 Idar-oberstein, Germany). The clay powders were uniaxially compacted in a 3cm diametric and 7 cm high cylindrical die using hydraulic press under 50 MPa pressure. The pressure was sustained overnight while the sample was inside the die. Prior to compaction, the die has been coated inside with the lubricant/parting agent for ease of sample removal after compaction process. Thereafter, the sample was carefully removed from the die and oven dried at 100°C for 48 hours. The dried samples were heated to 900°C in a controlled atmosphere of electric furnace (Thermolyte 46200) at 5°C/min. They were held at this temperature for 1 hour after which the temperature was increased to 1000°C and also held for 1 hour. The heating was continued until 1200°C at 100°C interval the samples were held at the temperature for a period of 1 hour. Table 1 below presents the heating regimes and four different groups of samples heat treated at 900°C, 1000°C, 1100°C and 1200°C were presented in Plate 2.



Plate 2. Produced clay refractories at different firing temperatures

Standard order	Holding temperature (°C)	Holding time (hour)
1	900	1
2	1000	1
3	1100	1
4	1200	1

Table 1. Firing Temperature

Prior to processing/fabrication of the brick, pre-washing and post-washing chemical analyses were carried out on the clay samples to determine the effect of washing on their chemical and mineralogical compositions with regards to alkali oxides such as Na₂O and K₂O in the clays. The color of the fired bricks was observed after furnace cooled to room temperature. The linear shrinkage (average), apparent porosity, bulk density, cold crushing strength, thermal shock resistance and thermal conductivity property tests were conducted on the produced fired bricks. The chemical and mineralogical analyses were carried out using



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atomic absorption spectrophotometer, model UNICAM 929. The quantity of SiO_2 and Al_2O_3 in the fired samples was analyzed in accordance with [16-17]. The amount of chemically combined or constitutional water otherwise called loss on ignition was estimated using the formula in Equation 1.

Loss on Ignition (LOI) =
$$\frac{m_i - m_f}{m_i} x100$$
 1

where m_i is the weight of the crucible and brick before firing and m_f is the weight after firing.

Porosity of the brick sample was determined by soaking the bricks in a bath of oil and water maintained at 90 ± 10 °C and left for 24 hours after which the samples were removed from the bath. Mass of the samples before and after soaking were determined and recorded. Porosity of the samples was determined using a formula in Equation 2; where m₁ is the mass of the brick before soaking, m₂ mass of the brick after soaking, b is the density of the liquid mix and V is the volume of the sample.

$$Porosity(p) = \frac{m_1 - m_2}{\flat V} x100$$

However, apparent porosity of each brick sample was also determined in accordance with ASTM C 20-8a. 50 x 50 x 40 mm³ brick sample was used. The dry weight in air (W_{da}), the weight soaked in water (W_{sw}) and the saturated weight (W_{sa}) of each sample were determined. The apparent porosity, Pa and bulk density of each brick were calculated using formulae in Equations 3-4.

$$P_a = \frac{W_{sa} - W_{da}}{W_{sa} - W_{sw}} x \ 100$$

Bulk density (Db) = True eight bulk volume =
$$\frac{W_{da}}{W_{sa} - W_{sw}}$$
 4

The shrinkage properties of the bricks were determined by measuring both the green and fired dimensions of the 50 x 50 x 40 mm³ bricks, using a Vernier caliper. All three sides were measured and the average linear shrinkage was calculated. Linear shrinkage of each side was calculated as a percentage of the original green dimension as shown in Equation 5. l_g and l_f are the green and fired lengths of the brick respectively.

$$Linear shrinkage = \frac{l_g - l_f}{l_g} x \, 100$$

The compressive property test was conducted on the brick samples using Tensometric Universal Testing Machine. 3cm diametric and 7 cm long bricks were loaded gradually in compression until the bricks failed to offer further resistance to deformation which was indicated by the brick fracture. The maximum load reached prior to brick fracture was recorded.



RESULTS AND DISCUSSION

Percentage composition of various compounds in each of the clay sample and their mineralogical profiles are presented in Table 2.

S/N	Chemical Composition			Constituents			
	Compounds	Site A	Site B	Minerals	Site A	Site B	
		%			%		
1	SiO ₂	53.93	61.04	Kaolinite ¹	65	52	
2	Al ₂ O ₃	24.61	19.63	Free Quartz	12	31	
3	Fe ₂ O ₃	3.21	3.78	Illite ²	4.3	2.4	
4	MgO	1.34	1.51	Chlorite ³	6.1	5.2	
5	CaO	0.35	0.32	Montmonilonite ⁴	3.5		
6	Na ₂ O	0.28	0.46	Feldspar	3	4	
7	K ₂ O	1.91	2.18				
8	$L.O.I(H_2O)$	9.2	10.11	Clay Content [1+2+3+4]	78.9	59.6	
9	Al ₂ O ₃ /SiO ₂	0.46	0.32				
	Ratio						

Table 2.	Chemical	and M	lineralo	gical co	mpositions	of both	clays
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Porosities and bulk densities of the produced bricks

Figure 1-4 display the porosities and bulk densities of the fired bricks produced from clay obtained from sites A and B. It is observed that the porosity varies inversely with the bulk density. Clay bricks from site A has percentage porosities ranging from 19.68 to 23.37 % while that from site B has a porosity range from 19.7 to 29.77 %. Their respective average bulk densities vary from 1.03 g/cm³ to 2.09 g/cm³. The porosity values of the brick samples from sites A and B are in line with recommended standard range (10 –30%) for fireclay refractories in literature [18]. Also, the bulk density values fall approximately within the range of 1.7 - 2.1 g/cm³ for dense firebricks as found in literature [19].



Figure 1. Porosity of clay brick obtained from site A





Figure 2. Porosity of clay brick obtained from site B



Figure 3. Bulk density of clay brick obtained from site A



Figure 4. Bulk density of clay brick obtained from site B

Linear shrinkage and apparent porosity

Figure 5-8 present the linear shrinkages and apparent porosities of the clay bricks from sites A and B respectively. It is observed that there is a slight decrease in the linear shrinkages as the firing temperatures increased from 900 to 1200°C. This decrease agrees with a decrease in the apparent porosities of the



samples. As the firing temperature increases, moisture within the brick changes into vapour and diffuse out of the brick, thereby creating vacant sites within the clay. Clay particles then migrate to occupy the vacant sites. This results in the shrinkage of the clay molecules and collapse of the void within the brick. At the point of intersection of clay grains, necking occurs leading to the fusion of clay grains. This is termed grain consolidation which leads to the formation of the bond which increases density (Figures 9-10) of the bricks due to reduced porosity within the volume of the bricks.







Figure 6. Linear shrinkage of the clay brick from site B



Figure 7. Apparent porosity of the clay brick from site A





Figure 8. Apparent porosity of the clay brick from site B



Figure 9. Density of the clay brick from site A



Figure 10. Density of the clay brick from site B



Compressive Strength

Figures 11-14 present the cold crushing strength and Young's modulus of the produced clay bricks from sites A and B. For both samples, there is an increment in the cold crushing strength as the firing temperature increase. This increase can be attributable to reduced porosities and increased densities with an increment in firing temperatures. The compressive strengths of the bricks obtained at 1200°C for sites A and B are 1.53 and 2.00 MPa which fall within the recommended range (1.0-68.8 MPa) for strength at peak in literature [14]. Cold crushing strength is a useful indicator of the ability of a refractory to withstand handling and impact at low temperatures. The higher the efficiency of firing, the denser the product samples and the higher is the cold crushing strength. The Young's Modulus values in Figures 13-14 fall within the range of 18.15 to 109 MPa fireclay bricks [18, 19].







Figure 12. Compressive strength of clay brick from site B





Figure 13. Compressive strength of clay brick from site A



Figure 14. Compressive strength of clay brick from site B

CONCLUSION

In this research work, Ujogba site A and B clays have been characterized to ascertain their potentials for ceramic and refractory applications. The results obtained show they meet the criteria for use as refractory/ceramic raw materials in all the characteristics investigated, viz.: chemical/mineralogical composition, shrinkage, porosity, bulk density, cold crushing strength, apparent porosity and density. The clays can be used as fireclay refractories. It is recommended that in using the clays for refractory applications, the high alkali content must be reduced by leaching (soaking and washing) and more inert additive (up to 44 % chamotte instead of the 25 % used) should be added for better performance of Ujogba clay as refractory materials.

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