



## ANALYTICAL EVALUATION OF THE THERMAL PROPERTIES OF EHANDIAGU AND UKPOR CLAY BLENDS FOR INDUSTRIAL APPLICATION

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### ABSTRACT

Analytical evaluation of the thermal properties of Ehandiagu and Ukpore Clay blends for industrial application was undertaken in this work. The clay samples were collected by manual method of mining, with the help of a digger and a shovel. The collected raw clay samples were dried in air and crushed to a particle size of 0.325mm using a locally fabricated pulverizing machine and soaked in a plastic

container containing about 20 liters of water. The filtrate was sun dried for three days followed by oven drying at 100°C for three hours. Chemical analysis was carried out at National Steel Raw Materials Exploration Agency (NSRMEA), Kaduna, with the aid of a Mini Pal 4 version (PW 4030) compact energy dispersive X-ray spectrometer. The clay samples were blended in the ratios of 80:20, 40:60, 60:40 and 20:80 designated as EU1, EU2, EU3 and EU4 respectively. E and U represent Ehandiagu and Ukpore in that order. Apparent porosity, bulk density and modulus of rupture of the clay samples were determined at 900°C, 1000°C, 1100°C and 1200°C in an electric kiln. The results showed that EU1 clay blend (80:20) had highest modulus of rupture of 23.58 at 1200°C and also maintained the highest values of apparent porosity at all service temperature. Also, the apparent porosity of EU1 at the studied service temperatures was between 20-30% which is within the standard range

required for the production of fireclay and siliceous fireclays. Therefore, this clay blend can be used for the production of fireclay and siliceous fireclays.

## INTRODUCTION

Clay is composed of silica ( $\text{SiO}_2$ ), Alumina ( $\text{Al}_2\text{O}_3$ ) and water ( $\text{H}_2\text{O}$ ) plus appreciable concentration of oxides of iron, alkali and alkaline earth, and contains groups of crystalline substances known as clay minerals such as quartz, feldspar and mica Folaranmi, (2009). Although, clay is inorganic in nature, it is also complex and its composition lies mainly on geographical location.

As was stated by Nwosu *et al.*, (2013) although, clay minerals share a basic set of structural and chemical characteristic however, each clay mineral has its own unique set of properties that determine how it will interact with other chemical species. This variation in both chemistry and structure, therefore, accounts for their applications in extremely diverse fields. The properties of clay units Nnuka and Enejor (2001) stated depend on the mineralogical compositions of the clays used to manufacture the unit, the manufacturing process and the firing temperature. Nnuka and Enejor (2001), hinted further that the percentage of the minerals oxides ( $\text{Fe}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{CaO}$  etc) and the presence of the alkali metals oxides ( $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$ , etc) determine their suitability and application for various purposes. Idenyi and Nwajagu (2003) hinted that clay has been reported to be abundant in soils, sedimentary rocks and hydrothermal deposits. However, Lopez-Galindo *et al.*, (2007) argued that this view is often misconceived with that of clay and therefore, opined that clay mineral refers to a family (Phyllosilicates) which consists of hydrated Alumino-silicates having a considerable amount of the elements; Mg, K, Ca, Na, Fe and at times Ti, Mn or Li. According to Aliyu *et al.*, (2013) clays are basically classified into two basic types with respect to their geological origin, primary clay (Kaolins) and secondary clay. Aliyu *et al.*, (2013) further stated that there are natural forces and various geological upheavals that induced various impurities in the form of minerals. It is a variation of these impurities in the basic formula that accounts for the different types of clay, and consequently, of clay bodies, Aliyu *et al.*, (2013) concluded. While Malu *et al.*, (2013) opined that Clay materials are basically divided into three groups, Lopez-Galindo *et al.*, (2007) and Nwoye, (2010) stated that the first group contain mainly Kaolinites; the second group are those that contain mainly Montmorillonite and the third group are intermediate product of disintegration of mica into kaolin. Nwoye, (2003) had attributed that the grain size and distribution of clays tend to have effect on their physical

properties and technological properties such as binding ability, shrinkage and plasticity. The various characteristic properties of most clay products are believed to be the consequence of impurity additions, sintering (firing procedure), and manufacturing processes Nweke, and Ugwu (2007).

Nigeria is endowed with abundant clay deposits with differing properties probably owing to geological differences Kefas *et al.*, (2007). There is an abundance of clay deposit in South East State Nigeria. Despite the abundance of clay in Nigeria, Kefas *et al.*, (2007) were of the view that it is rarely exploited for major uses pointing out that clay is mainly used for making traditional pots and bricks. Corroborating the work of Kefas *et al.*, (2007), Nwajagu (1995), stated that though, most Nigerian clay deposits remain unidentified, however, some of those identified have not been systematically studied. Malu *et al.*, (2013) noted that the chemical constituents of clays determine their usage.

Clay product derived from the weathering and hydrothermal reactions of rocks is a versatile industrial mineral that has amazing variety of uses and application as in petroleum cracking, cosmetic base, paper chalk, ceramics porcelain, dinner works, architectural tiles, pharmaceutical industries Ogbebor *et al.* (2010). The presence of  $\text{Fe}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$  (mineral oxide) in clay sample Kefas, (2007) observed determines its uses in bricks, floors tiles and paper making. The refractory potential of clay as was expressed by Malu *et al.*, (2013) is as a result of the presence of  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  and  $\text{CaO}$  (Alkali metal oxides) therefore, making clays suitable for ceramic products. Lopez-Galindo *et al.*, (2007) confirmed clay as a potential raw material in the pharmaceutical and cosmetics industries, though, insisted such clay materials have variable composition. Arisa, (1997) had earlier stated that clay has enormous potential in the ceramic industry as was also upheld by Malu *et al.*, (2013); Arisa, (1997) however, concluded that this depends greatly on the composition, grain size, grain distribution, grain structure and pores. Therefore, to expand the application of clays for industrial purposes samples of clays were obtained from Ukpör in Anambra State and Ehandiagu in Enugu State for blending to harness their industrial potentials and suitability for developing industrial products.

## 2.0 MATERIALS AND METHODS

### 2.1 Sample collection

Clay samples for this work were collected from Ehandiagu and Ukpör clay deposits in sacks by manual method of mining, with the help of a digger and a shovel. The clay samples were

collected from already existing open cast mine sites in a depth of about 35cm. The collected clay samples were in lump form.

**2.2 Sample Processing and Preparation** The collected raw clay samples were dried in air and crushed to a particle size of 0.325mm using a locally fabricated pulverizing machine. The crushed clay samples were soaked in a plastic container containing about 20 liters of water. The clay/water mixture was stirred vigorously to ensure proper dissolution and allowed to settle for four days. The dissolved clay/water mixture was later filtered to remove unwanted materials. The filtrate was sieved further using a mesh sieve of size 0.075 mm so as to obtain finer particles. The filtrate was allowed to settle for another four days after which excess water was decanted off. The resulting clay slip was dried in the sun for three days followed by oven drying at 100°C for three hours. The processed dry clay was pulverized and sieved again. The two clay samples were mixed with water and moulded using different mould sizes with respect to the tests to be carried out. The clay samples i.e. Ehandiagu and Ukor were later blended at the ratios 80:20, 60:40, 40:60 and 20:80.

**Table 2.1: Batch composition of Ehandiagu and Ukor clays.**

| Sample | Ehandiagu clay % | Ukor clay % |
|--------|------------------|-------------|
| EU 1   | 80               | 20          |
| EU 2   | 60               | 40          |
| EU 3   | 40               | 60          |
| EU 4   | 20               | 80          |

### 2.3 Chemical Analysis using XRF Technique

Representative samples of the clays were analyzed to determine the chemical composition (% SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, etc.). The samples were analyzed using a Mini Pal 4 version (PW 4030) compact energy dispersive X-ray spectrometer. The analysis was done at National Steel Raw Material Exploitation Agency, Kaduna. The samples to be analyzed were weighed and ground to powder using an agate mortar into a particle sizes within the range of 125µm and less. Pellets of 19mm in diameter were prepared by carefully mixing small portions of an organic liquid as binder (PVC dissolved in toluene) and processed with a hydraulic process to 10 tons. Each of the pellets was put in a plastic holder to ensure no interaction between the primary X-rays emerging from the X-ray tube window and the plastic holder. The holder and specimen were loaded into the specimen chamber of the spectrometer and the instrument operated at 30kV and 1mA for 10mins. The primary X-rays would excite secondary

emissions characteristic of the elements being analyzed and the spectrum analyzed to determine the concentration of the elements in the sample.

#### 2.4 Apparent Porosity

The apparent porosity test gives the fractional void volume of a fired body. This was determined by drying the test specimens at 110<sup>0</sup>C for 24 hours to ensure total moisture loss and fired at 900<sup>0</sup>C 1000<sup>0</sup>C, 1100<sup>0</sup>C and 1200<sup>0</sup>C in an electric kiln. The fired specimens were weighed in air and the value recorded was W<sub>1</sub>. The samples were immersed in water. After 24 hours, the samples were removed, re-weighed and the value was recorded as W<sub>2</sub>.

The dry weight in air (W<sub>da</sub>), the weight soaked in water (W<sub>sw</sub>) and the saturated weight (W<sub>sa</sub>) were used to calculate the apparent porosity, of each test specimen using the following relation:

$$\text{Apparent porosity} = \frac{W_{sa} - W_{da}}{W_{sa} - W_{sw}} \times \frac{100}{1} \dots\dots\dots 2.1$$

#### 2.4 Determination of Bulk Density

Bulk density is the mass per unit volume of the materials ignoring the volume occupied by pores. It depends upon the true specific gravity and the porosity Adams, (1979).

The bulk density of the blend was determined for each test specimen using the results of the apparent porosity test and was given as the ratio of the true weight and the bulk volume of test specimen.

$$\text{Bulk density (Bd)} = \frac{W_{da}}{W_{sa} - W_{sw}} \times \frac{100}{1} \dots\dots\dots 2.2$$

#### 2.5 Determination of modulus of rupture

This is a mechanical parameter which quantifies the ability of a ceramic material to resist deformation under load. The ASTM C133 – 97 (2008) is the standard test method for the determination of cold crushing strength and MOR of ceramic materials but not a measure of performance at elevated temperature. In this test a 3-point loading or bending jig was used where a test tile of 10x4x1cm will be tested until it fails. The pressure at which the test piece fails will be recorded and the M.O.R will be evaluated using the expression below;

$$\text{M.O.R} = \frac{3FL}{2bh^2} \dots\dots\dots 2.3$$

Where F=force

L=length between knife edges.

B=breadth of brick.

H=height of brick.

### 3. RESULTS AND DISCUSSIONS

#### 3.1 XRF Result Analysis

Table 3.1 shows the result of the chemical analysis on Ehandiagu and Ukpokor clays using X-ray fluorescence (XRF). From table 3.1, it can be observed that silica ( $\text{SiO}_2$ ) and alumina ( $\text{Al}_2\text{O}_3$ ) constitute the major composition of the clays with other metal oxides present in smaller amounts. While Ehandiagu clay sample had the highest  $\text{SiO}_2$  content of 70.14%, Ukpokor clay sample had the highest  $\text{Al}_2\text{O}_3$  content of 22.55%. As was noted by Abubakar et al., (2014),  $\text{Al}_2\text{O}_3$  determines the refractory characteristic of clay; therefore, it can be deduced from Table 3.1 that Ukpokor clay has better refractoriness than Ehandiagu clay. The  $\text{Fe}_2\text{O}_3$  contents of the two clays 0.72%, and 2.71% for Ehandiagu clay and Ukpokor clay respectively were found to be high and above the standard required for ceramics (0.5 – 1.2%), refractory bricks (0.5 – 2.4%), glass (2-3%) and paper (0.3 – 0.6%) but below the requirement for paper production (13.4 – 13.7%) which corroborated the work of Chukwu et al. (2017). Such high levels of iron oxide Abubakar et al., (2014) opined usually give a reddish colour to the clay body when fired making it suitable for some ceramic products such as flower vase which requires such colouration.

The presence of alkali oxides like CaO,  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  in reasonable amounts in Ukpokor clay shows good thermal ability during firing at low temperatures. The Loss on Ignition (LOI) of clay Olokode et al., (2013) argued accounts for the water vapour from de-hydroxylation reactions in the clay minerals, carbonate decomposition into  $\text{CO}_2$  and oxides as well as burning out of organic matter or other impurities present in the clay. The Loss on ignition (LOI) of Ehandiagu and Ukpokor clays are 13.60 and 10.12 respectively.

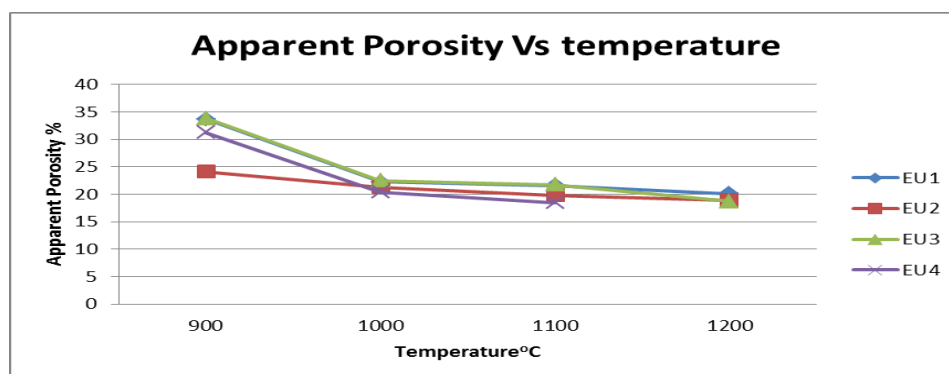
**Table 3.1: Results of XRF Chemical analysis of Ehandiagu and Ukpokor clay samples.**

| Oxides                  | Ehandiagu | Ukpokor |
|-------------------------|-----------|---------|
| $\text{SiO}_2$          | 69.171    | 65.142  |
| $\text{Al}_2\text{O}_3$ | 21.304    | 22.555  |
| CaO                     | 0.763     | 0.711   |
| MgO                     | 1.501     | 1.241   |
| $\text{Na}_2\text{O}$   | 0.581     | 0.329   |

|                   |       |       |
|-------------------|-------|-------|
| Fe <sub>2</sub> O | 0.832 | 2.52  |
| K <sub>2</sub> O  | 0.076 | 0.310 |
| MnO               | 0.003 | 0.410 |
| TiO <sub>2</sub>  | 1.703 | 2.512 |
| LOI               | 12.95 | 9.68  |

### 3.2 Result of Apparent Porosity

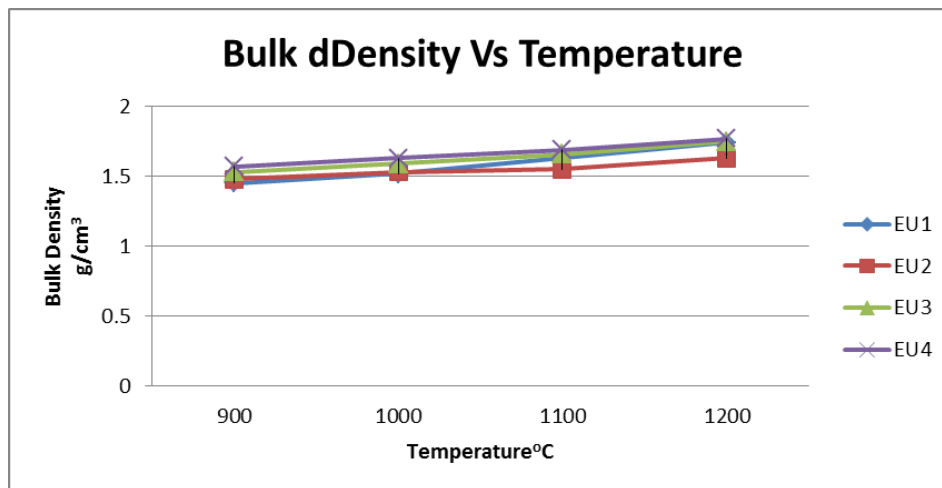
The apparent porosity of the clay blends decreased as firing temperature increased from 900°C to 1200°C. Although, the decrease in apparent porosity was observed for the four clay blends of EU1 to EU4, however, it was also observed that EU1 had the least difference in percentage value of 40.26% between 900°C and 1200°C while EU3 had the highest value of 44.73% within the same temperature range. Chester (1973) and Omowumi (2001), had hinted that 20-30% and (>23.7%) standard range of apparent porosity as suitable for production of fireclay and siliceous fireclays respectively. Only EU1 satisfied this requirement across the firing temperatures of 900°C to 1200°C adopted in this work. This signifies that this clay blend can be used for the production of siliceous fireclay at very high temperatures. While EU3 could not satisfy this requirement beyond 1100°C, this cannot be said of EU2 and EU4 which could only satisfy this at 1000°C.



### 3.3 Result of Bulk Density

Bulk density of a clay material is regarded as the mass per unit volume of the material ignoring the volume occupied by pores. In most clay materials, bulk density usually increases with rising firing temperature. The same can be said of these clay blends where similar trend was observed. Etukudoh *et al.*, (2016) had opined that the progressive increase in bulk density with firing temperature in clay materials implies that the clay becomes more compact and dense is expected to have a progressive increase in strength. The highest bulk density value of 1.77 g/cm<sup>3</sup> was recorded at 1200°C by EU4. Generally, the highest bulk density of the blends occurred at 1200°C. Aliyu *et al.*, (2013) and Thring (1962) had reported the

standard bulk density values of clay samples to be within the range of 1.08-1.97 g/cm<sup>3</sup>. All the clay blends in this work had bulk density values within this range.

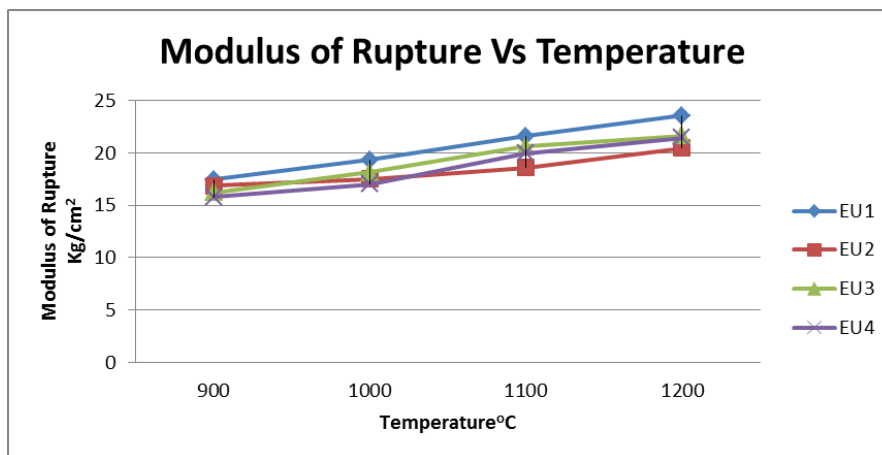


### 3.4 Result of Modulus of Rupture

The modulus of rupture is the flexural breaking strength (in kg/cm<sup>2</sup>) of a refractory, measured at room temperature. The modulus of rupture of the clay blends increased with increase in temperature. Nweke and Ugwu (2007) had observed this trend in Onueke, Ede – Achara), and Iboko clay samples. While Etukudoh et al., (2016) attributed this increase to clay body becoming more compact and rigid as temperature increases. Kefas et al., (2007) viewed the increase as a result of bond formation in the glassy phase of the clay body.

The highest value of 23.58kg/cm<sup>2</sup> was observed at the maximum service temperature of 1200°C for EU1. The clay blends maintained a progressive increase in modulus of rupture as the firing temperature increased. Also, EU1 had the highest modulus of rupture values across the firing temperatures of 900°C to 1200°C. Also, modulus of rupture value obtained at all temperatures was within the wide range of 1.4 to 105 kg/cm<sup>2</sup> as was stated by Kefas et al., (2007) generally required for the manufacture of a wide variety of clay products.





| Clay blend | Bulk density(g/cm <sup>3</sup> ) temperatures °C |      |      |      | Apparent Porosity(%) temperatures °C |       |       |       | Modulus of rupture (kg/cm <sup>2</sup> ) temperatures °C |       |       |       |
|------------|--|------|------|------|--------------------------------------|-------|-------|-------|--|-------|-------|-------|
|            | 900  | 1000 | 1100 | 1200 | 900                                  | 1000  | 1100  | 1200  | 900  | 1000  | 1100  | 1200  |
| EU1        | 1.45   | 1.52 | 1.63 | 1.74 | 33.68                                | 22.52 | 21.58 | 20.12 | 17.46  | 19.34 | 21.60 | 23.58 |
| EU2        | 1.48   | 1.53 | 1.55 | 1.63 | 34.12                                | 21.30 | 19.82 | 18.88 | 16.86  | 17.48 | 18.58 | 20.46 |
| EU3        | 1.53   | 1.59 | 1.66 | 1.75 | 33.54                                | 22.50 | 21.69 | 18.70 | 16.22  | 18.16 | 20.62 | 21.64 |
| EU4        | 1.57   | 1.63 | 1.69 | 1.77 | 31.25                                | 20.38 | 18.48 | 17.64 | 15.80  | 17.02 | 19.96 | 21.42 |

#### 4. CONCLUSION

The following conclusion was drawn from this work:

- Clay can be blended in a biaxial body composition to enhance thermal properties.
- EU1 (80:20) can be used for the production of siliceous fireclay and fireclay very high temperatures.
- The modulus of rupture of the clay blend was highest at maximum service temperature of 1200°C for EU1 (80:20).

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