

Characterization of some clay deposits in southern zone of Ebonyi state of Nigeria and their potentials for industrial utilization

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Abstract

Characterization of some clay deposits in Southern zone of Ebonyi State of Nigeria were carried out to determine their potentials for industrial applications. Chemical and mineralogical analyses of the clay samples were determined using x-ray fluorescence spectroscope (XRF) and x-ray diffractometer (XRD) respectively. The characteristics investigated before / after sintered at various temperatures were physical appearance, loss on ignition, plasticity, linear shrinkage, apparent porosity, bulk density, modulus of rupture, water absorption, thermal shock resistance and estimated refractoriness. The results obtained indicated that the clay samples are mostly kaolinitic clay deposits and chemically composed of mainly silica ranging 42.24 % to 61.21% and alumina ranging 15.11 % to 35.41%. The results of chemical and physical characteristics examined confirmed that the clay samples are suitable for industrial applications such as paper, paint, fertilizer, refractory bricks, ceramic tiles, porcelain insulator product e.t.c. Additionally, exploitation of two of the clay samples for porcelain insulator and dense refractory brick signified that excellent qualities of 33KVA porcelain insulators and dense refractory bricks of intercontinental standard can be fashioned locally with 100% local content. However, if these clay deposits can be utilized for industrial applications, it will go a long way in sinking the huge amount depleted in importation of many ceramic products and creating employment opportunity.

Keywords: Clay; Characterization; Refractoriness; Plasticity Index; Porosity

1. Introduction

Clays are numerous fine earth particles products of weathering and disintegration of feldspathic and granite rocks. They are anhydrous multifarious compounds of alumina and silica that exist in different proportions with various amounts of impurities like iron, organic matter and residual minerals [1,2]. It can also be referred to as a naturally occurring material which is fine granule in nature and commonly plastic when mixed with water and hardens when sintered [3]. However, categorization of clays for industrial applications is very vital. The different ratios to which the major components are bonded influence the clay minerals inherent properties and successive application [4]. Moreover, properties such as adsorptivity, bulk density, plasticity, shrinkages behaviour, refractoriness and thermal shock resistance are some of the technical characteristic of curiosity during characterization [5].

There are numerous types of clays depending on the areas of formation or location of the clay deposit. **Fire clay** is usually located in association with coal seams. Coal itself is fossilized remains of foliage, which grew millions of years ago. They are moderately free from fluxes and consequently consisting basically of alumina (Al_2O_3) and silica (SiO_2). Iron oxide is usually present ($\pm 0.1\%$) in it and can be tolerated as long as it does not affect the refractoriness of its product [6,7]. **Kaolin or China clays** are white burning clays, generally of low plasticity and high refractoriness. A high

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proportion of these are “kaolinite” ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) jointly with a substantial amount of impurities-free silica, quartz, iron oxide, titania etc [6,8,9]. **Hallosite Bearing Clays** which have the mineral hallosite, bear a resemblance to the china clays in physical appearance with in some properties but are extra plastic and turn out a denser fired body [7,8]. **Ball Clay** is the kaolintic sedimentary clay that usually consists of 20%-80% kaolinite, 10%-25% mica, 6%-65% sand (quartz). They are commonly used to impact plasticity and rheological stability during shaping process of many ceramics articles. It has property of opacity and fusion point that aids to lower the fusion point of the composite and also have iron oxide (red) which behaves as a flux [10]. **Sillimanite, Kyanite and Andalusite** is a group of related minerals, all with a formula, $3[\text{Al}_2\text{O}_3 \cdot \text{SiO}_2]$. On calcining, they behave as kaolinite and convert to mullite, cristobalite and glass but the proportion of mullite is higher [6]. **Diaspore, Bauxite and Gibbsite** are hydrated minerals with the formula, $\text{Al}_2\text{O}_3 \cdot \text{XH}_2\text{O}$ in which X is 1 for diaspore and 3 for Gibbsite. Bauxite is a mixture of the other two and X is approximately equal to 2. Bauxite is the most commonly used of the three and it may be added to the refractory brick batch in the uncalcined or moderately calcined state in order to effect combination with free silica and promote mullite formation or it may be used after very high temperature of calcination to give corundum [6].

The use of clays in general depends on their properties. Clays (Kaolinite) can be used in clay polymer/organic interactions, as is applied in paints and inks, where Kaolin is employed as an inert colloidal pigment [11]. It is also used in paper industry both as filling agent and as a coating agent. Printing inks tend to adhere better to Kaolin treated paper surface [11,12]. Both the grain size and shape of clays are useful properties in the production of various types of plastic and rubber products e.g. automobile tires where kaolinite is frequently employed as filler. It is used as raw material in paper making, rubber, plastic, paints, pharmaceuticals, soap, refectories and tiles, cement, fertilizer, textile, insecticides, toothpaste etc. Medically, it is used for production of artificial bone, as absorbent and in anti-diarrhea formulated [12,13,14].

A lot of researches have been carried out on clays in this regard for use as a medium for the removal of heavy metal pollutants and other unwanted substances from the environment such as industrial waste effluents [15]. Also, it is widely used for the production of electrical porcelain insulator and other equipments such as, circuit breaker, cut out switch component for power bushing weather shed and suspension high voltage insulator [16]. Odewale, et al., [17] developed a standard 33 KVA porcelain insulator using Nsu clay and other local raw materials. The research indicated that good quality 33KVA porcelain insulator of international standard can be produced locally with 100% local content. Production of porous firebrick from mixtures of clay and recycled refractory waste with addition of expanded perlite [18]. Also, Olokode et al., [19] investigated the suitability of using Ilese Awo clays and cow dung to produce insulating firebrick and to determine the optimal ratio of the constituent which gave a positive result. Other researchers on utilization of clay shows that some ceramic tiles were manufactured using clay and milled glass materials [20] while others are made of nearly 100% clay minerals. Also, Elekhame, et al., [21] affirmed in his research that clays are the major raw materials for sanitary wares production.

Clay minerals are among the most abundant raw materials that have been underutilized probably due to lack of knowledge of their properties. It is a standard practice that the use, exploration or commercialization of any raw material depends on the characteristics of the raw material in question. The composition of a given clay accounts almost entirely for its chemical and physical properties, it leads to its utilizations or applications. It has become necessary to characterize clay samples from different locations around various Local Government Area of Ebonyi South Zone of Ebonyi State with a view of sourcing and identifying the most suitable clays for different industrial applications. This research aims at identifying and sourcing efficient, low cost clay materials for industrial utilization. The characterization of particular clay will determine or guide on the area(s) of its application and commercial utilization.

2. Materials and methods

The Ogbu Edda clay sample was collected from Ogbu Edda community and Nguzu Edda clay was collected from Nguzu Edda community, both in Afikpo south Local Government Area of Ebonyi State. Amasiri clay and Ozizza clay were collected from Amasiri community and Ozizza community both in Afikpo North Local Government Area of Ebonyi State. While the Ishiagu clay sample was collected from Amata Ishiagu community in Ivo Local Government Area of Ebonyi State. Also, Okposi clay sample was collected from court area of Okposi community in Ohazara Local Government Area of Ebonyi State. At the same time, Isieke Oshiri clay sample was collected from Isieke Oshiri community in Onicha Local Government Area of Ebonyi State, all in Nigeria. Samples of clay were collected at random in different clay deposits around these regions in the community to obtain clay sample representatives from there.

2.1. Determination of the Chemical and Mineralogical Compositions of the Clay Samples

The collected clay samples were analysed using X-ray fluorescence spectroscope (XRF) to determine oxide compositions. The results in concentration of weight percent oxide were achieved.

The clay samples were analysed using X-ray diffraction patterns (XRD) to determine the oxide mineral present in the different clays (mineralogical composition).

2.2. Determination of the Physical Properties of the Clay Samples

2.2.1. Visual Inspection / Observation

The inspection / observation of all the clay samples were done to determine their physical appearances immediately after sample collection. The process was repeated after the sintering of test pieces from all the clay samples at 950°C, 1050°C, 1150°C and 1250°C.

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

The percentage Loss on Ignition of the clay samples were determined by measuring the weight loss of a known mass of the clay samples after heating in a furnace at 1000°C for 1hr 30mins. The loss on Ignition of the clay samples were calculated using this relation:

$$\text{Loss on ignition (LOI)} = \left(\frac{W_i - W_f}{W_i} \right) 100\%$$

Where W_i and W_f are initial and final weight respectively.

2.2.3. Determination of the plasticity index

Plasticity index is the range of water content required to be add to clay before it becomes plastic. However, Plasticity index (PI) is liquid limit (LL) minus plasticity limit (PL). Therefore, to establish the plasticity index, liquid limit and the plastic limit must be known.

2.2.4. Determination of the liquid limits

The Casagrande apparatus method was used in this research. 200g of the clay sample was sieved using 425µm sieve, air dried, and carefully mixed. A little distilled water was added to the sample and mixed on a sheet glass. The cup of the apparatus was half filled with the wet clay and leveled off. Grooving tool was then used to cut 2mm groove from the sample. The handle of the apparatus was rotated at a stable rate, which activate the cam, causing the cup to lift 10mm and then drop onto the base. The number of blows required to close the gap over 13mm was recorded. A fraction of the tested sample was removed and positioned for moisture content determination (at 105°C). Plastic flow caused the groove to close, however, the groove was considered closed as soon as two parts of the sample came into contact at the base of the groove. The test was repeated two more times, utilizing a little more water for each test. The process was repeated for all the clay samples.

2.2.5. Determination of the plastic limit

In this test, 20g of the prepared sample as in the liquid limit test was used to form a clay rod of about 80mm long and 3mm diameter used. Enough water to make it satisfactorily plastic was added to the sample, mixed on the glass plate and rolled into a ball shape. The clay ball was then rolled out in-between the hand and the glass to form a strand (thread). The strand was alienated into two parts as soon as small cracks began to become visible. One part was formed into a strand, rolled until the diameter of the strand was reduced to 3mm from 6mm. The rod diameter helped out in determining the strand diameter. The sample is said to be at its plastic limit as soon as it crumbled at a strand (thread) diameter of 3mm. Thereafter, a fragment of the strand was removed and subjected to water content determination (at 105°C). The test was repeated two more time and average was determined. The process was repeated for all the clay samples.

2.2.6. Linear Shrinkage

At wet stage, a mark (line) with sharp object were given to five prepared test pieces from each clay samples at the centre point of the top side of the test pieces (a measured length). The changes in length of the marks (line) were determined after drying and sintering to present the linear shrinkage of the pieces.

$$\% \text{ drying shrinkage} = \frac{\text{wet length} - \text{dry length}}{\text{wet length}} \times 100$$

$$\% \text{ firing shrinkage} = \frac{\text{dry length} - \text{fired length}}{\text{dry length}} \times 100$$

$$\% \text{ Linear shrinkage} = \frac{\text{wet length} - \text{fired length}}{\text{wet length}} \times 100$$

2.2.7. Apparent Porosity and Bulk Density.

Five test pieces (60mmx40mmx30mm) from each clay sample were prepared and used to carry out this test. The pieces were weighed (W_1) after proper drying in oven. The pieces were removed and the specimens were soaked in water. Saturated specimens were weighed in air (W_2) and in water (W_3). The weight of the absorbed liquid ($W_2 - W_1$) was used to realize the volume since density of water is 1g/cm^3 . The value also stands for volume of the open pores in cubic centimetres. The total volume of a specimen is ($W_3 - W_1$). The apparent porosity and bulk density were determined using equation below equations.

$$\% \text{ Apparent porosity} = \frac{W_2 - W_1}{W_2 - W_3} \times 100$$

$$\text{Bulk density} = \frac{W_1}{W_2 - W_3} \times 100$$

2.2.8. Flexural Strength or Modulus of Rupture (MOR)

In this test, a rectangular bar specimens of 100m x14.5mm x11mm were produced and dried at 110°C , sintered at various temperatures (950°C , 1050°C , 1150°C and 1250°C) before cooled in a desiccator. A transverse strength tester was used to break the specimens and the forces required to cause the crack on the specimens were recorded. The M.O.R was evaluated using the expression below;

$$\text{M.O.R} = \frac{3Pl}{2bh^2}$$

Where P = force, l = Distance between support, b = breadth and h = height of the test piece.

2.2.9. Water Absorption Test

Five test pieces (as in M.O.R) were used in this test. It was done by a 24hrs immersion in cold water as recommended by Standard Organization of Nigeria (SON). The test pieces were pre-conditioned by drying in a ventilated oven at 110°C until they maintain a constant mass. They were then cooled to ambient temperature and weighed to note their initial weight (w_1). The test pieces were immersed in cold water at room temperature for 24hrs and removed. All trace of water was cleaned off with dry towel and the weights of the test pieces (w_2) were recorded. The percentage water absorption that is the weight gain was then evaluated using the equation:

$$\% \text{ water absorption} = \frac{\text{saturated weight} - \text{dry weight}}{\text{dry weight}} \times 100\%$$

2.2.10. Determination of Refractoriness

The refractoriness of the clay samples were estimated using Shuen's formula.

$$K = \frac{360 + Al_2O_3 - RO}{0.228}$$

Where K = Refractoriness ($^\circ\text{C}$),

Al_2O_3 = Alumina Content in the clay

RO = Sum of all the oxides beside SiO_2 in the clay (or materials)

360 and 0.228 are constants.

2.2.11. Spalling Count Test or Thermal Stress Resistance Test

Five test pieces size of 60mmx40mmx30mm were used. The samples were heated in an electric kiln, at about 1150°C and soaked for 15 minutes. The hot test pieces were then picked out of the kiln and plunge into water of room temperature one after the other, Observation for any crack were made on the pieces and returned to the kiln. The same process was repeated until the samples cracked. Thermal shock resistance is the number of cycles required to cause obvious crack on the sample. The process was repeated for all the clay samples.

2.3. Utilization of Ogbu Edda Clay and Nguzu Edda Clay for Industrial Application

Ogbu Edda clay was used with other raw materials (feldspar, quartz and talc sourced locally) in various body compositions to produce 33KVA porcelain insulator. The insulators were shaped using jigger jolley machine and sintered at 1400°C via front loading, J W Ratchliff, P U 131 brand of electric kiln. Also, Nguzu Edda clay was used with grog in varying percentages to produce dense refractory bricks. The bricks were uniaxially compacted using hydraulic press under a pressure of 30 MPa for the rectangular-shaped specimens and sintered at 1400°C via front loading, J W Ratchliff, P U 131 brand of electric kiln. The technological properties of the 33KVA porcelain insulators and dense refractory bricks were measured after sintering steps.

3. Results and discussion

Table 1 Result of Chemical compositions of the clay samples by X-ray fluorescence spectroscopy (XRF).

| Parameters | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | TiO ₂ | MgO | K ₂ O | ZnO | P ₂ O ₅ | Na ₂ O | MnO | ZrO ₂ | Other oxides | Total |
|-------------------|------------------|--------------------------------|--------------------------------|------|------------------|------|------------------|------|-------------------------------|-------------------|------|------------------|--------------|-------|
| Ogbu Edda clay | 54.99 | 35.41 | 2.15 | 0.18 | 4.41 | 0.14 | 1.31 | 0.01 | - | 0.17 | 0.02 | 0.21 | 0.99 | 100 |
| NguzuEdda clay | 53.80 | 34.89 | 5.57 | 0.11 | 2.60 | 0.11 | 0.57 | 0.03 | - | 0.09 | 0.03 | 0.26 | 1.94 | 100 |
| Ozizzaclay | 56.84 | 22.92 | 2.99 | 0.22 | 1.64 | 1.79 | 6.14 | 0.01 | 1.12 | 0.92 | 0.03 | 0.45 | 4.93 | 100 |
| Amasiriclay | 57.26 | 28.84 | 1.13 | 3.03 | 2.35 | 1.45 | 0.49 | 0.02 | - | 0.29 | 0.04 | 0.26 | 4.84 | 100 |
| Ishiaguclay | 42.24 | 15.11 | 22.07 | 7.22 | 4.52 | - | 1.71 | 0.04 | 5.47 | - | 0.16 | 0.22 | 0.81 | 100 |
| Okposiclay | 52.96 | 29.87 | 3.25 | 1.94 | 2.72 | 1.43 | 0.92 | 0.05 | 1.09 | 0.38 | 0.13 | 0.47 | 4.97 | 100 |
| IsiekeOshiri clay | 61.21 | 19.95 | 3.78 | 1.30 | 2.52 | 1.50 | 2.16 | 0.07 | 2.47 | 1.42 | 0.18 | 0.76 | 4.03 | 100 |

The results of chemical analysis of the clay samples as shown in Table 1 indicated that all the clays had alumina (Al₂O₃) contents ranging from 15.11 % to 35.41% with silica (SiO₂) contents of 42.24 % to 61.21%. The alumina contents are in agreement with Aderibigbe, [22], which reported that the main refractory clay deposits in Nigeria are within the alumina-silicate raw material (kaolin and fireclay) deposits with alumina content of less than 45%. Moreover, the alumina contents are within 23% to 36% recommended for insulator production [14,23,24] except that of Ozizza, Ishiagu and Isieke Oshiri clays which are below the range. Also, the percentage SiO₂ contents in the clay minerals are within the acceptable range of 40% and above for classic clay precious for porcelain insulator and refractory production [6,24,25]. Furthermore, the chemical composition of the clays measure up favourably with Grimshaw's composition specifications for clays utilized in some industrial applications [7,26,]. Except those of Ozizza, Ishiagu and Isieke Oshiri clays which had Al₂O₃ contents below 25%, this can be improved by addition of other raw materials. Therefore, the clay could be utilized in ceramic, paper, paint, fertilizer and refractory industries.

The result of mineralogical compositions in Table 2 as determined by X-ray diffraction (XRD) revealed that the clay samples had kaolinite (35% to 67%) as their main constituents except that of Ishiagu clay with quartz (47%) as its main constituent. Other mineral constituents identified comprise of Muscovite, illite, chlorite, montmorillonite, Orthoclase, haematite and Albite (Table. 2). This indicated that high percentage of these clay samples are "kaolinitic clay deposits" (Al₂O₃.2SiO₂.2H₂O) together with a considerable amount of impurities-free silica, iron oxide, titania etc [6,8,9].

Table 2 Result of Mineralogical analysis of the clays by X-ray diffraction patterns (XRD).

| Principal mineral (%) | Ogbu Edda clay | Nguzu Edda clay | Ozizza clay | Amasiri clay | Ishiagu clay | Okposi clay | Isieke Oshiri clay |
|-----------------------|----------------|-----------------|-------------|--------------|--------------|-------------|--------------------|
| Kaolinite-1A | 35 | 58 | 37 | 51 | - | 67 | 53 |
| Quartz | 26 | 24 | 29 | 30 | 47 | 13 | 32 |
| Muscovite | 2.7 | 5.2 | 2.3 | 4 | 32 | - | - |
| Hematite | - | 2.9 | - | 3.4 | - | - | - |
| Illite | 14 | 2.8 | 12 | 3.2 | - | 4.6 | 2.5 |
| Albite | 3.7 | 7 | 2.6 | 8.3 | 14 | - | - |
| Orthoclase /Feldspar | 18 | - | 17 | - | 5 | 2 | 5 |
| Chlorite | - | - | - | - | 2 | 6 | 5.3 |
| Montmorillonite | - | - | - | - | - | 3.6 | - |

Table 3 Result of visual appearance of clays

| CLAY SAMPLE | Raw sample | 950°C | 1050°C | 1150°C | 1250°C |
|--------------------|---------------|-------------|-------------|-------------|-------------|
| Ogbu Edda clay | Milky | Gray | Gray | Gray | Gray |
| Nguzu Edda clay | Mottled white | Light Cream | Light Cream | Light Cream | Light Cream |
| Ozizza clay | Deep Ash | Ash White | Ash White | Ash White | Ash White |
| Amasiri clay | Light Gray | Cream | Cream | Cream | Cream |
| Ishiagu clay | Deep Gray | Milky | Milky | Milky | Milky |
| Okposi clay | Black | White | White | White | White |
| Isieke Oshiri clay | Milky | Cream | Cream | Cream | Cream |

Table 4 Result of loss on Ignition (LOI) of clay samples

| CLAY SAMPLE | Ogbu Edda clay | Nguzu Edda clay | Ozizza clay | Amasiri clay | Ishiagu clay | Okposi clay | Isieke Oshiri clay |
|-----------------|----------------|-----------------|-------------|--------------|--------------|-------------|--------------------|
| % LOI at 1000°C | 10.82 | 10.17 | 9.67 | 11.72 | 11.97 | 9.30 | 10.20 |

It was noticed in visual observation that the clay samples possess various colours from milky, mottled white, deep/Light gray/ash to black colours at raw form. After sintering (950 °C – 1250°C), the colours changed to more attractive colours of ash white / white, light cream / cream to milky colours (Table. 3). Also, the percentage losses on ignition (LOI) of the clay samples from Ogbu Edda clay to Isieke Oshiri clay had 10.82%, 10.17%, 9.67%, 11.72%, 11.97%, 9.30% and 10.20% (Table. 4). Their percentage losses on ignition (LOI) are within the acceptable range of 5% - 14% as stipulated [7,26]

Table 5 Liquid Limit (LL) Result

| Test | 1 | 2 | 3 |
|--|------|------|------|
| Container number | A | B | C |
| Number of blows (N) | 10 | 20 | 40 |
| Container M (g) | 21.4 | 21.5 | 21.1 |
| Container plus wet clay M_w (g) | 40.5 | 42.1 | 38.3 |
| Container plus dry clay M_d (g) | 32.8 | 34.1 | 31.9 |
| Dry clay = $(M_d - M)$ | 11.4 | 12.6 | 10.8 |
| Water loss = $(M_w - M_d)$ | 7.7 | 8.0 | 6.4 |
| Water Content (w) = $\frac{M_w - M_d}{M_d - M} \times 100$ | 67.5 | 63.5 | 59.3 |

Table 6 Plastic Limit (PL) Result

| Test | 1 | 2 |
|--|------|------|
| Container number | A | B |
| Container M (g) | 17.8 | 17.1 |
| Container plus wet clay M_w (g) | 31.5 | 31.9 |
| Container plus dry clay M_d (g) | 27.6 | 27.8 |
| Dry clay = $(M_d - M)$ | 9.8 | 10.7 |
| Water loss = $(M_w - M_d)$ | 3.9 | 4.1 |
| Water Content $w = \frac{M_w - M_d}{M_d - M} \times 100$ | 39.8 | 38.3 |
| Mean | | 39.1 |

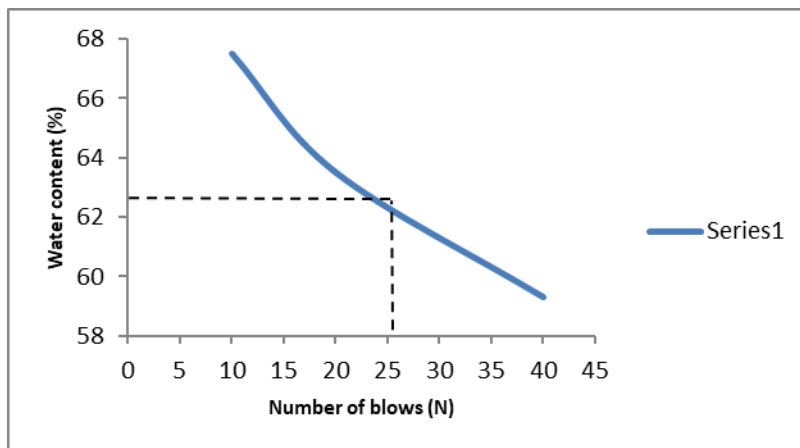


Figure 1 Water content (w) as a function of Number of blows (N)

Table 7 Plastic Index

| Details | Liquid limit (LL)% | Plastic Limit (PL) | Plasticity index |
|----------------|--------------------|--------------------|------------------|
| Ogbu Edda clay | 62.4 | 39.1 | 23.3 |

Table 8 Result of Plastic Index of clay samples

| CLAY SAMPLE | Ogbu Edda clay | Nguzu Edda clay | Ozizza clay | Amasiri clay | Ishiagu clay | Okposi clay | Isieke Oshiri clay |
|------------------------|----------------|-----------------|-------------|--------------|--------------|-------------|--------------------|
| Plastic Index (PI) (%) | 23.3 | 25.9 | 24 | 24.6 | 28 | 31 | 23 |

The Liquid limit (LL) and plastic limit (PL) results are exposed in Tables 5 and 6 correspondingly. Figure 1 illustrates the plot of water content (W) as a function of number blows (N), from these the liquid limit (LL) and hence plasticity index (PI) values shown on Table 8 were derived. The clay samples from Ogbu Edda clay to Isieke Oshiri clay had plasticity index values of 23.3%, 25.9%, 24%, 24.6%, 28%, 31% and 23% (Table. 8). These values are within the recommended value of 10-30% for ceramic clays as suggested by RMRDC [27] and Chukwudi, [8]. The values also fall within the range of 10% - 60% recommended for ceramic clays [9,26,28].

Table 9 Result of Linear Shrinkage of clay samples

| CLAY SAMPLE | 105°C (%) | 950°C (%) | 1050°C (%) | 1150°C (%) | 1250°C (%) |
|--------------------|-----------|-----------|------------|------------|------------|
| Ogbu Edda clay | 3.01 | 7.90 | 8.70 | 9.30 | 10 |
| Nguzu Edda clay | 2.50 | 4.60 | 5.10 | 6.50 | 7.40 |
| Ozizza clay | 2.02 | 4.11 | 5.28 | 6.59 | 7.81 |
| Amasiri clay | 2.12 | 7.83 | 10.26 | 10.39 | 11.13 |
| Ishiagu clay | 2.04 | 4.30 | 5.58 | 6.79 | 8.01 |
| Okposi clay | 2.01 | 7.76 | 8.77 | 9.94 | 10.01 |
| Isieke Oshiri clay | 2.10 | 4.61 | 4.99 | 5.32 | 5.61 |

The linear shrinkage of the clay samples investigated exposed that from Ogbu Edda clay to Isieke Oshiri clay had 10%, 7.40%, 7.81%, 11.13%, 8.01%, 10.01% and 5.61% values at 1250°C (Table. 9). The clay samples were subjected to five different ranges of heat treatments (105 °C, 950°C, 1050°C, 1150°C and 1250°C). It was revealed that the linear shrinkage of the clay samples increases progressively with the temperature (Table. 9). This may occur as a result of the percentage losses on ignition (LOI) of the clays samples (Table. 3), high loss on ignition of clay sample usually leads to high linear shrinkage of the clay. It may crop up probably due to the development that usually occurs along with the changes of low quartz to high quartz and the high quartz to high cristobalite polymorphic modifications that happened while sintering between 573 °C and 1250°C [17]. Also, the more a clay sample is bonds together during sintering, the less porous and denser it is and the more shrinks it becomes [7]. Majority of the clay samples fell within the recommended linear shrinkage range of 7-10% for refractory clays [27,29] while some fell outside it. Also, the obtained values of the result fell within the recommended range of 2-10% for clays used for porcelain insulator production [14,30] while some fell outside the range.

The result of the apparent porosity obtained specified that the clay samples from Ogbu Edda clay to Isieke Oshiri clay had 32.44%, 31.06%, 24.12%, 33.21%, 17.31%, 26.20% and 30.01% values at 1250°C (Table. 10). The clay samples were exposed to four different sintering temperatures (950°C, 1050°C, 1150°C and 1250°C). It was discovered that the apparent porosity of the clay samples decreases progressively as the temperature increases (Table. 10). This indicated that at high sintering temperature, thermally enthused material moves into the pores / holes which lead to reduction in porosity. Also, the formation of vitreous phase at elevated temperature could initiate reduction in porosity [7,14,31].

The apparent porosity values of three clay samples obtained are within the acceptable range (10-30%) suggested for refractory clays [29,32,33] while some fell outside the range.

Table 10 Result of Apparent Porosity of clay samples

| CLAY SAMPLE | 950°C (%) | 1050°C (%) | 1150°C (%) | 1250°C (%) |
|--------------------|-----------|------------|------------|------------|
| Ogbu Edda clay | 38.69 | 33.69 | 33.32 | 32.44 |
| Nguzu Edda clay | 36.71 | 32.91 | 32.22 | 31.06 |
| Ozizza clay | 29.16 | 25.98 | 25.51 | 24.12 |
| Amasiri clay | 38.94 | 34.55 | 34.28 | 33.21 |
| Ishiagu clay | 20.78 | 19.12 | 18.89 | 17.31 |
| Okposi clay | 29.28 | 28.34 | 27.12 | 26.20 |
| Isieke Oshiri clay | 33.04 | 32.91 | 31.17 | 30.01 |

Table 11 Result of Bulk Density of clay samples

| CLAY SAMPLE | 950°C (g/cm ³) | 1050°C (g/cm ³) | 1150°C (g/cm ³) | 1250°C (g/cm ³) |
|--------------------|----------------------------|-----------------------------|-----------------------------|-----------------------------|
| Ogbu Edda clay | 2.65 | 2.21 | 2.22 | 2.22 |
| Nguzu Edda clay | 2.12 | 1.89 | 1.92 | 1.92 |
| Ozizza clay | 2.24 | 1.78 | 1.88 | 1.86 |
| Amasiri clay | 3.00 | 2.77 | 2.79 | 2.79 |
| Ishiagu clay | 2.08 | 1.71 | 1.78 | 1.76 |
| Okposi clay | 2.32 | 1.89 | 1.92 | 1.91 |
| Isieke Oshiri clay | 2.39 | 2.06 | 2.09 | 2.10 |

Table 12 Result of transverse strength or modulus of rupture (MOR) of clay samples in MPa.

| CLAY SAMPLE | 950°C (MPa) | 1050°C (MPa) | 1150°C (MPa) | 1250°C (MPa) |
|--------------------|-------------|--------------|--------------|--------------|
| Ogbu Edda clay | 10.4 | 11.13 | 11.15 | 12.24 |
| Nguzu Edda clay | 12 | 13 | 13.16 | 14 |
| Ozizza clay | 13.87 | 14.97 | 15.07 | 16.70 |
| Amasiri clay | 10 | 11.81 | 12 | 13.13 |
| Ishiagu clay | 14.16 | 15.96 | 16.51 | 18.64 |
| Okposi clay | 13.01 | 14.12 | 15 | 16.02 |
| Isieke Oshiri clay | 9.92 | 10.81 | 11.29 | 12.91 |

The result of the investigation shows that the clay samples (from Ogbu Edda clay to Isieke Oshiri clay) had bulk density of 2.22g/cm³, 1.92 g/cm³, 1.86 g/cm³, 2.79 g/cm³, 1.76 g/cm³, 1.91 g/cm³ and 2.10 g/cm³ values at 1250°C (Table. 11). The clay samples were exposed to four different sintering temperatures (950°C, 1050°C, 1150°C and 1250°C) and it was revealed that the bulk density of the clay samples decreases as the temperature increases but not steady (Table.

10). At high temperature, densification usually came up which is important in the lessening of porosity and rise in bulk density. This may be attributed to the development of the vitreous phase which initiates the stalemate of the pore in the sintered clay samples and increasing the bonding strength [7,14]. The bulk density values fall approximately within the suggested minimum of 2g/cm^3 for porcelain body [17,24,34]; $1.7\text{g/cm}^3 - 2.1\text{g/cm}^3$ for dense refractory bricks [35] and range of $0.8\text{g/cm}^3 - 2.9\text{g/cm}^3$ [36].

The modulus of rupture (MOR) result of the clay samples from Ogbu Edda clay to Isieke Oshiri clay signified that they had 12.24MPa, 14MPa, 16.70MPa, 13.13MPa, 18.64MPa, 16.02MPa and 12.91MPa values at 1250°C (Table. 12). The clay samples were exposed to four different sintering temperatures ($950^\circ\text{C} - 1250^\circ\text{C}$) and it was discovered that the modulus of rupture (MOR) values of the clay samples increases progressively with the temperature (Table. 12). The moderate modulus of rupture values may be attributed to the development of the glassy phase which offers the bonding potency of the sintered clay samples. Moreover, the presences of CaO, K₂O and Na₂O in the chemical compositions (Table. 1) of the clay samples are enough to boost the progression of vitreous phase growth [14]. In addition, the closer to the sintering point a clay sample is fired, the more obvious becomes the sintering at the temperature [7] and as a result lessening porosity, increasing density and linear shrinkage with increase in modulus of rupture (MOR) values of the clay samples. The modulus of rupture (MOR) values obtained were within the acceptable range when considering the international recommended minimum value of 10MPa as stated by IS 1445 [37] and 15MPa for porcelain insulators [34,38,39]. Also, they are within the range of 9.8 - 68.8MPa recommended for fireclay bricks [40] and are higher than the recommended minimum of 5MPa [29].

Table 13 Result of water absorption of clay samples

| CLAY SAMPLE | 950°C (%) | 1050°C (%) | 1150°C (%) | 1250°C (%) |
|--------------------|-----------|------------|------------|------------|
| Ogbu Edda clay | 33.54 | 28.89 | 27.39 | 26.58 |
| Nguzu Edda clay | 25.87 | 23.31 | 22.53 | 21.40 |
| Ozizza clay | 11.03 | 12.02 | 12.99 | 13.98 |
| Amasiri clay | 32.50 | 27.83 | 26.19 | 25.38 |
| Ishiagu clay | 17.84 | 16.12 | 15.89 | 15.31 |
| Okposi clay | 24.28 | 21.64 | 20.16 | 19.80 |
| Isieke Oshiri clay | 30.07 | 28.96 | 27.67 | 26.91 |

The result of water absorption of the clay samples investigated indicated that from Ogbu Edda clay to Isieke Oshiri clay had 26.58%, 21.40%, 13.98%, 25.38%, 15.31%, 19.80% and 26.91% values at 1250°C (Table. 13). It was exposed that the water absorption values of the clay samples decreases progressively with increase in temperature (Table. 13). This may be as a result of the development of the glassy phase at elevated temperature that leads to the impasse of the pores in the sintered clay samples. Moreover, the nearer the budding temperature of a clay sample is sintered, the conspicuous becomes the sintering at the temperature [7,14], which led to reduction in porosity, rising bulk density and decline water absorption.

Table 14 Result of estimated refractoriness of clay samples using shuen's formular.

| CLAY SAMPLE | Ogbu Edda clay | Nguzu Edda clay | Ozizza clay | Amasiri clay | Ishiagu clay | Okposi clay | Isieke Oshiri clay |
|---|----------------|-----------------|-------------|--------------|--------------|-------------|--------------------|
| Estimated using shuen's formular ($^\circ\text{C}$) | 1692.15 | 1682.37 | 1590.70 | 1644.47 | 1458.16 | 1634.65 | 1583.82 |

The results of estimated refractoriness of the clay samples using shuen's formula designated that from Ogbu Edda clay to Isieke Oshiri clay had refractoriness of 1692.15°C , 1682.37°C , 1590.70°C , 1644.47°C , 1458.16°C , 1634.65°C and 1583.82°C (Table. 14). The result demonstrates that the refractoriness of the clay samples are relatively high. This may be attributed to moderate to high percentage of the alumina content and high fusing temperature of SiO₂ content in the clay samples chemical compositions (Table. 1). It is a known fact that temperature increases as alumina content

increases. All the clay samples met the essential refractoriness for refractory bricks above cone 08 (990°C) i.e minimum of 1000°C [41,42].

Table 15 Spalling Count Test or Thermal Stress Resistance Test

| CLAY SAMPLE | Ogbu Edda clay | Nguzu Edda clay | Ozizza clay | Amasiri clay | Ishiagu clay | Okposi clay | Isieke Oshiri clay |
|-----------------|----------------|-----------------|-------------|--------------|--------------|-------------|--------------------|
| Number of Cycle | 27 | 26 | 19 | 28 | 12 | 23 | 25 |

The result of the Spalling count cycle or thermal stress resistance of the clay samples investigated indicated that from Ogbu Edda clay to Isieke Oshiri clay had 27, 26, 19, 28, 12, 23 and 25 number of cycles (Table. 14). It was revealed that the higher the porosity the higher the number of spalling count cycles (Table. 10 and 14) and the lower the bulk density, the lower the thermal conductivity of the sintered clay samples [43]. The thermal conductivity is not rely only on their porosity, but it is also depends on the shape and size of the hole / pore with chemical and mineralogical composition of the clay body [44]. The thermal stress resistance values are considered satisfactory except that of Ozizza and Ishiaguclays (19 and 12) which fell below the recommended range is 20 - 30 cycles [8,29,45].

However, the chemical composition of clay raw material plays a vital role in the properties of the end products and the percentages of each raw material in the body composition are important. There is correlation between thermal conductivity and strength, at the same time other physical properties, like abrasion, permeability and mechanical strength are frequently interrelated to the density and porosity of the product. Additionally, porosity has effect on both thermal conductivity and mechanical strength. Besides, as density improves, spalling resistance may turn downward, it is very imperative to glance for combinations of properties that stick to success of the product's application.

After utilization of Ogbu Edda clay for 33KVA porcelain insulator product, it was discovered in the technical properties of the products that as the percentage content of Ogbu Edda clay increases which brought about the decrease in percentage of feldspar in the porcelain recipe, led to increase in the linear shrinkage, water absorption, appearance porosity, bulk density, crushing strength and flash over voltage with reduction in thermal stress resistance of the porcelain insulators and vice versa. Furthermore, some samples provided suitable result when considering the properties stated compared with international standard. Also, it was observed in the dense refractory bricks produced that the higher the grog content of the dense bricks, the lower the linear shrinkage, bulk density and compressive strength while the higher the porosity, spalling count test and refractoriness of the bricks and vice versa. Nonetheless, some samples of bricks produced gave the most favourable result when considering the moderate percentage linear shrinkage, higher bulk density, compressive strength and refractoriness with standard spalling count cycle weighed against with international standard. This exposed that high-quality of 33KVA porcelain insulators and dense refractory bricks of global standard can be manufactured locally with 100% local content.

4. Conclusion

The clay samples are mostly kaolinitic clay deposits and their chemical compositions indicated that they can be used for industrial applications when compared with the compositions of other clays that are used for specific industrial applications (ceramic, paper, paint, fertilizer, refractory and pharmaceutical industries). Also, almost all the investigated properties gave results that are acceptable for refractory bricks, ceramic tiles, porcelain insulator product e.t.c. Furthermore, utilization of two of the clay samples for porcelain insulator and dense refractory brick revealed that high-quality of 33KVA porcelain insulators and dense refractory bricks of international standard can be manufactured locally with 100% local content. If these clay deposits are exploited and harnessed, it will go a long way in reducing the enormous amount depleted in importation of many ceramic products.

Compliance with ethical standards

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Statement of ethical approval

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