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EHA-NDIAGU CLAY DEPOSITS: PHYSIC-CHEMICAL CHARACTERIZATION FOR INDUSTRIAL REFRACTORY APPLICATIONS.

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ABSTRACT

This research work evaluated the characterization of Eha-Ndiagu clay in Enugu State, South-East Nigeria for its industrial potentials. The chemical analysis was performed at National Steel Raw Materials Exploration Agency (NSRMEA), Kaduna, using a Perkin Elmer Atomic Absorption Spectrophotometer (AAS) while the physical properties were investigated following American Society for Testing and Material (ASTM) stipulated standards. The physical properties determined were apparent porosity, bulk density, linear shrinkage, apparent density, modulus of rupture, plasticity index, Atterberg Limit and water absorption capacity. The result of the chemical composition analysis showed that the, the clay contains mainly Silica Oxide (SiO2) 56.71%, Aluminum Oxide (Al2O3) 21.05% and Iron oxide (Fe2O3) 5.31% with other oxides such as MgO (1.50%), K2O (1.92%) and TiO_2 (1.03%) in appreciable amounts. The physical analysis showed a variation in the linear shrinkage (3.35 to 7.88%), total shrinkage (5.06 to 9.46%), apparent porosity (41.02 to 32.57%), bulk density (1.52 to 1.76 g/cm3), water absorption (15.22 to 8.9%) and modulus of rupture (12.86 to $24.78 kgF/cm^2$) with increase in firing temperature from 900^oC to 1200^oC. The Atterberg Limit result showed that the clay has high plasticity. The overall analysis of the results showed that Eha-Ndiagu clay deposit is siliceous in nature and of the alumino-silicate refractories that may be classified as kaolinitic fireclay. This result therefore, showed that Eha-Ndiagu clay has good industrial potentials and can be useful in the manufacture of ceramics, high melting clays, refractory, bricks, tiles and in the paper and

paint industries but with the incorporation of additives that will help to obtain the desired properties.

INTRODUCTION

There is a growing research interest in clay nationally and globally as an industrial raw material. Research properties investigated mostly borders on areas of the researcher's interest. As at thirties and up to sixties, in the USA and Europe, extensive work on identification and application of clays abound Grim et al. (1935) and Nwajagu (1995). The use of any engineering material for intended purpose is largely dependent on its inherent or tailored properties. Clay is no exception to this and is among class of engineering materials which properties can be tailored to suite particular applications. Clays according to Ajayi, and Agagu (1981) are valuable resources in agriculture, engineering and structural ceramic industries for the manufacture of domestic and commercial products. Ademila and Adebanjo (2017) stated that clay bodies are widely distributed on the Precambrian basement complex of Nigeria. Velde (1995) viewed clays as geological materials having particle size of less than 2 microns and the family of minerals have similar chemical compositions and common crystal structural characteristics. Other minerals which may impart plasticity may also be contained in clays Bakker (1993).

Utilization of most clay minerals in desired application Aramide et al., (2014) argued is a function of the physical and chemical properties of the particular clay mineral and dependent on its structure and composition. The structure and composition of kaolins, smectites, and palygorskite and sepiolite Aramide et al., (2014) continued are very different even though they each have octahedral and tetrahedral sheets as their basic building blocks. The arrangement and composition of these octahedral and tetrahedral sheets Murray (1999) contended account for major and minor differences in the physical and chemical properties of kaolin, smectites and palygorskite.

Although, clay minerals share fundamental set of structural and chemical characteristic, however, Nwosu et al., (2013) are of the view that each clay mineral has its own unique set of properties that determine how it will interact with other chemical species. The variation in both chemistry and structure among the clays Nwosu et al., (2013) hinted leads to their applications in extremely diverse fields.

Though Nwosu et al., (2013) stated that specific clay minerals are identified by several techniques including thermal differential analysis, scanning electron microscope, infrared spectrometry and X-ray diffraction, Newman (1987) identified chemical analysis as an essential step to establishing the nature of minerals Newman (1987).

Generally, impurities and abundance are of major concern in the evaluation of clay samples as commercial raw materials.

Characteristic properties of most clay products Nweke and Egwu (2007) believe are as a consequence of impurity additions, sintering (firing procedure), and manufacturing processes.

The characterization of clay is mainly carried out with a number of experimental approaches in order to investigate all the relevant features.

Over the years, studies on the characterization and properties of clays deposits in different parts of Nigeria for useful information on their applicability has been undertaken by any researchers Onwuachi-Iheagwara (2012), Ahmed and Onaji (1986) and Nuka and Enejor (2001). As a result, such clays as mayo-belwa, nsu, ukpor, afuze, usen, uzalla clay, kankara, adiabo and dabagi have been characterized and found useful applications for industrial purposes and ceramics Kefas et al., (2007) and Abubakar et al., (2014).

In the southern part of Nigeria, Amaefule, (1990) stated that most regions are richly blessed with natural resources like clay and these clay-based materials occur both in the plain and river rime areas.

This research work aims to determine the physical and chemical composition of Eha-Ndiagu clay for possible Industrial applications. Eha-Ndiagu is located in Isi-uzo local government of Enugu state, South-East Nigeria.

2.0 MATERIALS AND METHODS

2.1 Sample collection and pretreatment

Ehandiagu clay was the major material used in this research work. The clay Sample was collected from Eha-Ndiagu clay deposit in sacks by manual method of mining, using digger and shovel. The clay sample was collected from an existing open cast mine sites indicating that the clay was being used for local activities. The clay was in lumps of about 5-7cm. The collected clay sample was soaked in excess water for three days, stirred vigorously to ensure proper dissolution of any deleterious materials present in them and also to form slurry. The slurry was then sieved to remove the deleterious materials and other foreign substances. The filtrate was then allowed to settle for three days after which excess water was decanted. The clay was then sundried and oven dried at 100 ^{0}C for 24 hours. The dried clay sample was pulverized in a locally fabricated grinder and sieved to an average particle size of 100μm.

Plate 1: Sample of Eha-Ndiagu clay.

Plate 2: Test bar of Eha-Ndiagu clay.

2.2 Molding of the Test Pieces

The clay was then molded into rectangular shapes using metallic moulds. The surface of the moulds was coated with lubricants to prevent the test pieces from sticking to the surface and avoid cracking of the test pieces.

2.3 Determination of Modulus of Rupture

This is a mechanical parameter which quantifies the ability of a ceramic material to resist deformation under load in observance of ASTM C133 – 97. Five long rectangular test pieces were made and air dried for 7 days after which they were oven dried at 105 ^{0}C until a constant weight was obtained. Four of the pieces were fired to their respective temperatures of 900^{o}C, 1000^{o}C, 1100^{o}C and 1200^{o}C in an electric kiln (Fulham Pottery). A 3-point loading or bending jig was used where a test tile 10x4x1cm was tested until it failed. The electrical transversal strength machine was used to determine the breaking load, P (Kg). A vernier caliper was used to determine the distance between supports L (cm) of the transversal machine. The height, H (cm) and the width, B (cm) of the broken pieces were determined and the average value obtained from the two broken parts was recorded.

The pressure at which the test piece failed was recorded and the M.O.R was evaluated using the expression below;

$$\text{M.O.R} = \frac{3FL}{2bh^2} \text{ ………………………………..2.1 Kefas et al., (2007):}$$

Where F=force
L=length between knife edges.
B=breadth of brick.
H=height of brick.

2.4 Determination of Plasticity Index

These tests were carried out in accordance with ASTM D423 – 66 (Re-approved 1972) for the determination of atterberg limits using the Casangrade apparatus and accessories. The moisture contents determined from the liquid limit (LL) and plastic limit (PL) tests were used to compute the plasticity index, PI. The difference between the LL and PL gives the PI for each of the clays, to the nearest whole number.

$$\text{(PL) - (PL) = PI ………………………………………..2.2}$$

2.5 Determination of Shrinkage

The shrinkage was determined considering both the dried and fired shrinkage. The drying shrinkage is an index of the body to withstand cracking or retain shape and size after firing. It also indicates the degree of plasticity of the mixture. After molding the rectangular test pieces, a vernier caliper was used to insert a 10 cm mark on each of them and was recorded as the original length Lo (cm). The test samples were air dried for 10 days and oven (cabinet) at 105 0C until a constant weight was obtained. The shrinkage from the 10 cm mark was determined and recorded as the dried length, Ld (cm). The dried samples were fired to their respective temperatures of 900^{o}C, 1000^{o}C, 1100^{o}C and 1200^{0}C with each temperature corresponding to a particular test piece. The shrinkage of the test pieces from the 10 cm mark were then determined and recorded as the fired length, Lf (cm).

Dry Shrinkage (%) = 100[Lo - Ld]/Lo …………… 2.3

Linear Shrinkage (%) = 100[Ld - Lf]/ Ld …………2.4

Total Shrinkage (%) = 100[Lo – Lf]/ Lo ………… 2.5

2.6 Bulk Density, Apparent Porosity and Water Absorption Capacity

The bulk density accounts for the overall weight coming upon the foundation of a refractory structure. The apparent porosity test gives the fractional void volume of a fired body while the water absorption test is an index of the ability of the ware to take up moisture or liquid. These tests were carried out by drying the test specimens at 110^0C for 24 hours to ensure total moisture loss and fired at 1000^0C, 1100^0C and 1200^0C in an electric kiln. The fired specimens were weighed in air and the value recorded was W_1. The samples were immersed in water. After 24 hours, the samples were removed and re-weighed; the value was recorded as W_2. Each of the samples were attached to a string and suspended in water in a beaker. The water was allowed to boil for 30 minutes. The boiled water was allowed to cool and the weight soaked and suspended in water was taken as W3. The water of absorption was calculated using this formula;

Water absorption = $\frac{W_2 - W_1}{W_1} \times \frac{100}{1}$ ………………………2.6

The apparent porosity, apparent density and bulk density were calculated using the following formulae:

Apparent Porosity (%) = 100 $[M_2 - M_1]/[M_2 - M_3]$ ………… 2.7

Apparent Density (g/cm3) = $M_1/[M_1 - M_3]$ ………………….. 2.8

Bulk Density (g/cm3) = $M_1/[M_2 - M_3]$ ……………………….. 2.9

2.7 Chemical Analysis using AAS

The samples were analyzed for major element oxides (SiO2, Al2O3, Fe2O3, MgO, CaO, Na2O, K2O and MnO) at National Steel Raw Materials Exploration Agency (NSRMEA), Kaduna, using a Perkin Elmer Atomic Absorption Spectrophotometer (AAS). P2O5 and TiO2 were determined by a colorimetric method while loss on Ignition (IL) was obtained by heating in a furnace to a temperature of 1000^oC for thirty minutes.

3.0 RESULTS AND DISCUSSION

3.1Chemical Analysis

The result of the chemical analysis on Ehandiagu, clay using Atomic Absorption Spectrum (AAS) is shown in Table 4.1. It can be observed from Table 4.1that silica (SiO_2) and alumina (Al_2O_3) constitute the major composition of the clays with other metal oxides present in smaller amounts. While the Ehandiagu clay SiO_2 content is 56.71%, the AL_2O_3 content is 21.05%. The silica content of the clay satisfied the clay requirement for refractory bricks (>51.7%) and high melting clays (53-73%) as was reported by Chester (1973). However, the silica level was also found to be lower than the requirement for glass (80-90%) but higher than that for paper (45.0 – 45%) and paint (45.3 -47.9%) in line with the work of Grimshow (1971). The alumina composition of the clay did not satisfy the requirement for the manufacture of ceramics (26%), Recfractory bricks (25 – 44%), paper (33.5 – 36.1%), paint (37.9 – 38.4%) although, they met the standard required for production of glass (12 – 17%) Grimshow (1971). The percentage composition of alumina in clay is a strong indication of its refractoriness and the higher the amount of alumina the more refractory the clay is Abubakar et al., (2014) stated. The Fe_2O_3 content of Ehandiagu clay of 5.31%, was 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). Furthermore, such high levels of iron oxide 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 Abubakar et al., (2014).

The presence of alkali oxides like CaO, K_2O and Na_2O in reasonable amounts in clay shows good thermal ability during firing at low temperatures, hence, reducing the vitrification temperature and refractoriness of the clay as Maiti and Kumar (1992) observed. This however cannot be said of Ehandiagu clay in which the alkalis (K2O, Na2O) as well as CaO, MgO and MnO occurred in relatively insignificant proportions in the range 0.11 to 0.53% and is indicative of the high degree of weathering, under tropical conditions, from which the clay bodies resulted Onyeobi et al., (2013). Also, the presence of these oxides in clay Maiti and Kumar (1992) noted acts as mild fluxes, as they combine with the oxides of silica and alumina on firing to form eutectics and so reduce the vitrificaion temperature and refractoriness of the clay. The Loss on Ignition (LOI) of clay accounts for the water vapour from dehydroxylation reactions in the clay minerals, carbonate decomposition into CO_2 and oxides as well as burning out of organic matter or other impurities present in the clay Olokode et al., (2013). The Loss on ignition (LOI) of Ehandiagu clay is 13.60. This is within the standard for manufacture of ceramics (>8.18%), refractory bricks (8 – 18%) and high melting clays (5 -14%) Grimshow, (1971) and also indicate that the clay sample has low carbonaceous matter and higher mineral matter contents as Malu et al., (2013) stated. TiO_2 content is greater than 1% in Ehandiagu clay.

Table 1: Result of Chemical Analysis using AAS.

Oxides	Ehandiagu
SiO_2	56.71
Al_2O_3	21.05
CaO	0.34
MgO	1.50
Na_2O	0.38
Fe_2O	5.31
K_2O	1.92
MnO	0.82
TiO_2	1.03
LOI	13.60

3.2 Atterberg Limit

The Atterberg Limit indicates the liquid limits (LL), plastic limits (PL) and plasticity index (PI) of the clay samples. Atterberg limits (liquid limit, plasticity limit, plasticity index) Ojo et al., (2017) stated are the properties that can reflect the amount and type of clay in clay samples. They help in understanding the industrial behaviour of kaolin clays (Spagnoli and Sridharan, 2013) pointed out. From table 3.1, it can be seen that the liquid limits of Ehandiagu clay sample is 66%. Whitlow (1995) noted that liquid limit less than 30% indicates low plasticity, between 35% and 50% indicates intermediate plasticity, between 50% and 70% high plasticity, between 70% and 90% indicates very high plasticity and greater than 90% indicates extremely high plasticity. On this basis, it can be said Ehandiagu clay sample is highly plastic.

Declan, and Paul (2003) stipulated that the liquid limit of clay materials used for barriers lining should be less than 90%. The determined liquid limit of the studied clay is less than 90%. Thus the clay is expected to exhibit low hydraulic conductivity and will be suitable for use as barrier soils.

The plastic limit of Ehandiagu clay sample was 35%, as indicated in Table 1. Grimshaw (1971) prescribed a plastic limit range of 10 – 60% for clays suitable for use in ceramic production. This implies that Ehandiagu clay sample met this requirement. Also, the technological evaluation of kaolin with moderate range of plasticity Ojo et al., (2017) explained shows that such clay deposit could be exploited commercially for use in the ceramics industry. The deposits can be adjudged suitable for white ware and sanitary ware when compared with the kaolin industry specifications of NAFCON (1985).

The value of plasticity index (an indicator of clay plasticity) for Ehandiagu sample is 31%. Rowe et al., (1995) indicated that plasticity index of clay must be greater than 7% for the clay to be suitable for use as barrier materials. Ehandiagu clay sample has a high liquid limit (66%), high plastic limit (35%) and a high plasticity index (31%) indicating a high swelling and high compressibility characteristics. Murray et al., (1992) suggested a notable increase in permeability where materials have a plasticity index of 12% or less, hence the clay materials under consideration with plasticity index greater than 12% can be considered suitable for barrier soil as notable increase in permeability is not expected.

3.3 Moisture Content

Ademila and Adebanjo (2017) stated that moisture content determination is important in understanding the engineering properties of clay soil for construction and industrial use. It is a function of the void ratios and the specific gravities of the samples. Banaszak (2013) using kaolin clay, illite clay and electrotechnical porcelain showed how the strength of dried materials may vary with moisture content and changes that may occur during the drying process. Ojo et al., (2017) suggested a specific level of moisture content at which clay is easily workable; below which the molded clay body will crack. The moisture content Ehandiagu clay sample was 24% as indicated in Table 1. Although it is not a constant property of clay soils Ademila and Adebanjo (2017) contended, the values obtained are consistent with the fines contents of the clays. The high moisture content obtained therefore conforms to the generally accepted high porosity and low permeability properties of clay Ademila and Adebanjo (2017).

3.4 Apparent Porosity (%)

The apparent porosity according to Ugwuoke and Amalu (2017) is a measure of the effective open pores spaces in a refractory into which molten metal, slag, fluxes, vapours etc can penetrate and thus contribute to the structure’s degradation. As was stated by Chester (1973) the standard values range for porosity is 20-30%. Gupta (2008) noted that low percentage of apparent porosity enhances gas entrapment in the refractory which adversely affects its life span when in operation. The apparent porosity for the clay sample was determined at 900°C, 1000°C, 1100°C and 1200°C. The apparent porosity of the clay sample decreased as temperature increased, as could be observed in Fig.1, with a peak value of 34.69% at 1100°C. The percentage apparent porosity decrease as the temperature increased indicates more closure of the clay pores. This also agreed with work of Etukudoh et al., (2016) who maintained that the decrease is due to the increase in shrinkage with increasing temperature which resulted in the.

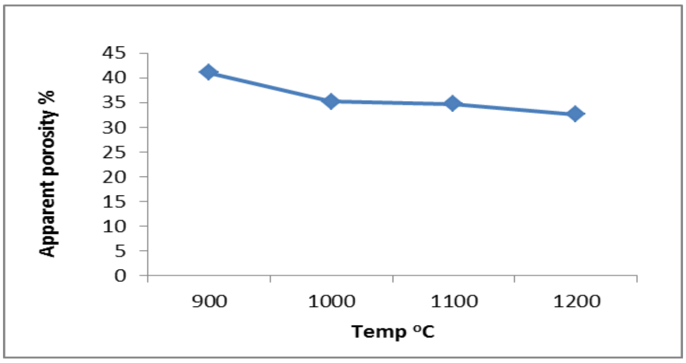

Fig. 1: Effect of Temperature on Apparent Porosity.

Coming together and closure of the pores of the clay body. Ehandiagu clay samples could be utilized in the production of insulating materials by the addition of some carbonaceous materials to help improve its porosity and insulating properties Etukudoh et al., (2016) hinted. Also, in line with the work of Omowumi (2001), where the standard range of 20-30% and (>23.7%) apparent porosity for production of fireclay and siliceous fireclays respectively was recommended, Ehandiagu clay sample therefore may be used for fireclay and siliceous fireclays production.

3.5 Bulk density (g/cm^3)

Bulk density is the mass per unit volume of the materials ignoring the volume occupied by pores and as Adams (1979) stated depends upon the true specific gravity and the porosity. The bulk density for the e clay sample was determined at 900°C, 1000°C, 1100°C and 1200°C. From Fig. 2, the bulk density increased as temperature increased. This is in line with the works of Etukudoh et al., (2016) where bulk density increased with increase in temperature and Ugwuoke and Amalu (2017) in which the highest bulk density 1.83g/cm^3 of a clay sample was obtained at a temperature of 1300°C. This as Etukudoh et al., (2016) opined implies that the clay becomes more compact and dense is expected to have a progressive increase in strength of the clay body. Ehandiagu clay sample had the highest bulk density values of 1.76 (g/cm^3) at 1200^{o}C. This is within the range of 1.08-1.97 g/cm3as reported by Aliyu et al., (2013) and Thring (1962). The high values of bulk density observed therefore can be attributed to low percentage of porosity of the sample. This makes Eha-Ndiagu clay suitable for siliceous fireclays as reported by Omowumi (2001) and fireclays according to Abdullahi, and Samaila (2007).

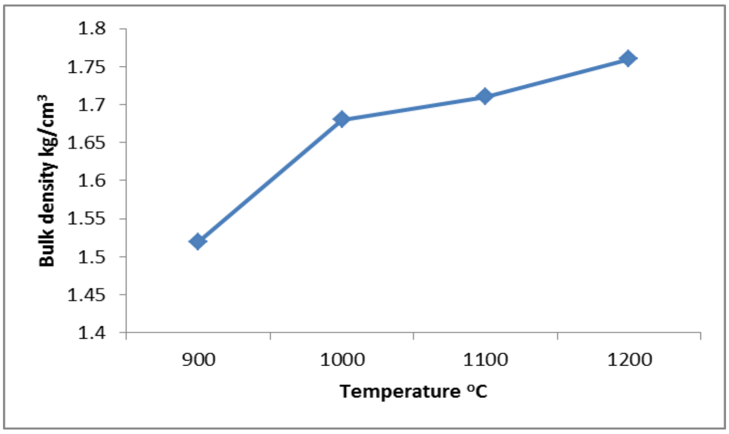

Fig. 2: Effect of Temperature on Bulk Density.

3.6 Water absorption

A decrease in water absorption with increase in firing temperature of the clay sample was observed as recorded in Fig 3. With increase in the firing temperature of the clay sample from 900^{o}C to 1200 ^{0}C the water absorption decreased from 15.22% to 8.90%. This decrease in water absorption Etukudoh et al., (2016) argued may be attributed to an increase in shrinkage and decrease in porosity of the clay body with increase in firing temperature.

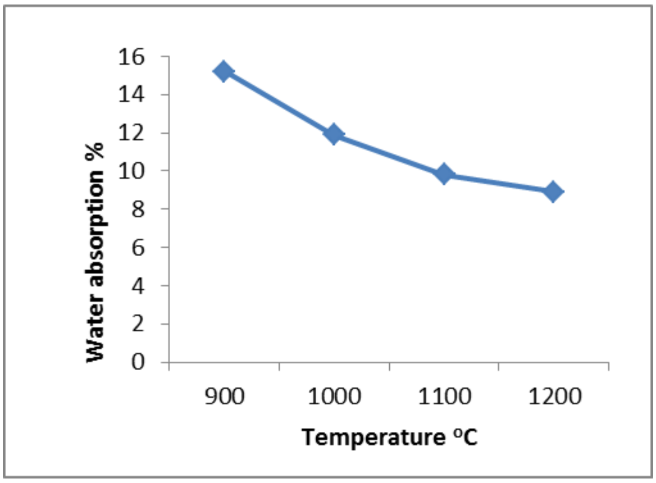

Fig. 3: Effect of Temperature on Water Absorption.

3.8 Modulus of rupture

The modulus of rupture is the flexural breaking strength (in kg/cm2) of a refractory, measured at room temperature Ugwuoke and Amalu (2017). The modulus of rupture increased with increase in temperature Fig. 4 and corroborates the work of Nweke and Ugwu (2007) where such trend was observed in Onueke, Ede – Achara), and Iboko clay samples. Etukudoh et al., (2016) attributed this increase to the clay body becoming more compact and rigid as temperature increases and the clay body shrinks. Kefas et al., (2007) viewed the increase as a result of bond formation in the glassy phase of the clay body. Also, the modulus of rupture value obtained at all temperatures was within the wide range of 1.4 to 105 kgF/cm^2 as was stated by Kefas et al., (2007) generally required for the manufacture of a wide variety of products.

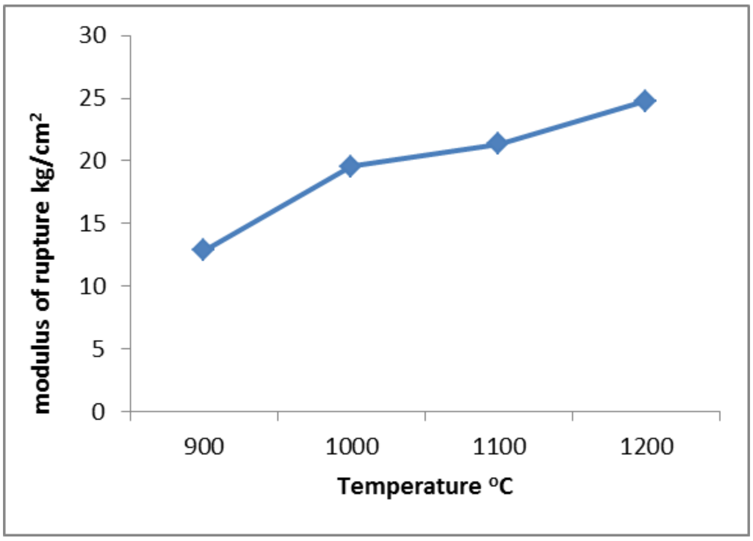

Fig. 4: Effect of Temperature on Modulus of Rupture.

3.9 Linear shrinkage (%)

Ugwuoke and Amalu (2017) stated that shrinkage represents the firing efficiency of clay samples. The linear shrinkage of Eha-Ndiagu clay sample increased with increase in firing temperature. Although Omowumi (2001) quoted a recommended shrinkage value range of 4-10%, Chester (1973) however, recommended linear shrinkage range of 7-10% for refractory clays. Hence, Abolarin et al., (2004) suggested that lower shrinkage values were more desirable in order that the clay will be less susceptible to volume change. The shrinkage value of the clay sample fell within the recommended range. Etukudoh et al., (2016) also reported an increase in the linear shrinkage of the clay with increase in firing temperature from 900^{o}C to 1200 ^{0}C and attributed it to the removal of certain components in the clay body with increase in temperature resulting in the sintering and subsequently vitrification of the clay body.

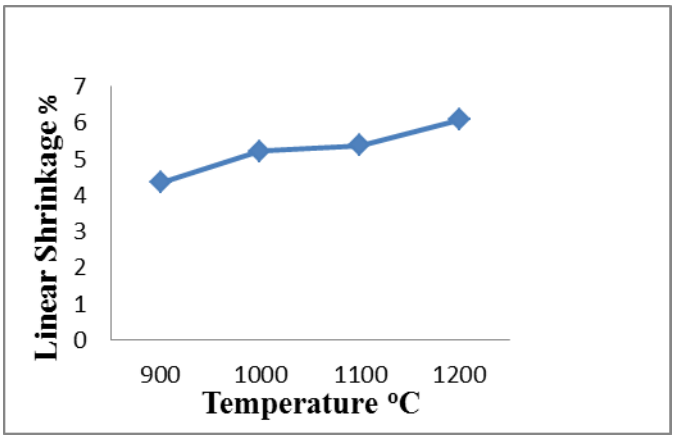

Fig. 5: Effect of Temperature on Linear Shrinkage.

The implication is that as the temperature is increased the clay body tends to compress leading to decrease in porosity in some cases. As can be seen from Fig. 5, the linear shrinkage of Eha-Ndiagu clay sample at the studied firing temperature range of 900°C (4.35%) to 1200°C (6.08%) was within the range of 4-10% reported by Omowumi (2001) and meets the requirement for fireclays as was stated by Abubakar et al., (2014) and the standard value range of 7-10% for alumino-silicates and kaolinites reported by Zubeiru (1997).

Table 2:

Property Description	Ehandiagu			
	900°C	1000°C	1100°C	1200°C
Apparent Porosity	41.02	35.17	34.69	32.57
Modulus of rupture	12.86	19.57	21.34	24.78
Water absorption	15.22	11.88	9.80	8.90
Bulk density	1.52	1.68	1.71	1.76
Linear shrinkage (%)	4.35	5.22	5.38	6.08

4. CONCLUSION

This research work successfully characterized Eha-Ndiagu clay as a potential industrial raw material considering its physical and chemical properties. The result showed that Eha-Ndiagu clay contains mainly silica (SiO_2) and alumina (Al_2O_3) with reasonable quantity of iron oxide (Fe_2O_3). The presence of alkali oxides in the clay was also observed. The high strength and high plasticity of the clay is an indication of its usefulness in the manufacture of a variety of products. Eha-Ndiagu clay may therefore be useful in the manufacture of ceramics, high melting clays, refractory, bricks, tiles and in the paper and paint industries but with the incorporation of additives that will help to obtain the desired properties.

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