



## Evaluation of Some Physico-chemical and Mineralogical Properties of Clay Mineral Deposits in Ihievbe, Owan East Local Government Area, Edo State, Nigeria

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### ABSTRACT

The chemical, physical and mineralogical composition of clay mineral deposits at Ihievbe in Owan East Local Government Area of Edo State, Nigeria were evaluated using standard methods. The chemical assay revealed the predominance of silica (41.85-42.82%), alumina (20.17-20.42%), iron oxide (11.33-13.75%), sodium oxide (5.09-12.04%), loss on ignition (3.93-4.67%) and moisture (1.07%). This indicates that the deposit consists largely of aluminosilicate minerals. The mineralogical studies revealed the presence of montmorillonite, kaolinite, halloysite, muscovite, palygorskite as clay minerals present. Other minerals present were kyanite, naotocite, quartz, goethite and ramsdellite. The evaluated physical properties showed that the clay minerals had moderate swelling characteristics, medium plasticity, good thermal stability and refractoriness above 1200°C. However, the apparent porosity and air permeability values were slightly above permissible limits for refractory clays. Thus the clay mineral deposits assayed possess characteristics to enable them to be employed in high temperature operations.

**Keywords:** Clay mineral, Plasticity, Refractoriness, Thermal stability

### INTRODUCTION

Clay minerals are of diverse importance based on their different unique properties. Some industries such as the foundries and ceramics employ the use of clay on account of its thermal stability, while others such as petroleum, rubber, paint and pharmaceutical industries utilize it based on its ability to swell and become sticky when it absorbs water acting as sealant, filler, colourant etc, while in agriculture and environmental remediation, its importance are based on its capacity to serve as reservoir of exchangeable ions which is used to improve soil fertility or purification of polluted soil or water through ion exchange. Some clays are consumed directly because of the health benefits (Gomes, 2018). The cement industry utilizes it as a raw material for the supply of alumina and silica (Odo and Nwajagu, 2003; Onyeobi *et al.*, 2013, Shermeen and Petra, 2017, Uchida, 2000, Ogeleka *et al.*, 2017). The multifarious applications of clay based on its properties make it imperative to undertake the study of any clay mineral deposit as studies have shown that no two clay mineral deposits are the same both in physical or chemical composition based on the fact that clay minerals are secondary weathering products which are formed from the disintegration of parent rocks and also subject to in situ alterations due to climatic conditions including erosion (Joussein *et al.*, 2005; Rashidi and Seilsepour, 2008). Nigeria is blessed with clay

mineral deposits as its proven reserve is estimated at several billion of tonnes (Lori *et al.*, 2007; RMRDC, 1990). While efforts have been made to characterize some of the clay mineral deposits, large amount of these clay mineral deposits remain uncharacterized (Irabor, 2002; Elueze *et al.*, 1999; Irabor and Ohenhen, 2018; Irabor and Okunkpolor, 2020; Lori *et al.*, 2007). Clay defined broadly as naturally occurring material composed of fine-grained minerals which is generally plastic at appropriate water content, hardens when dried or fired. Although clay usually contains phyllosilicates, it may contain other materials which imparts plasticity and hardness when dried or fired (Guggenheim and Martin, 1995; Akhievbulu *et al.*, 2010). They contain aluminosilicate minerals, which are arranged either in octahedral and or tetrahedral geometry to form individual sheets interlayered with cations and are grouped as kaolin, smectite, palygorskite, saponite, illite and mixed clays (Jock *et al.*, 2013, Laine *et al.*, 2017). The arrangement or composition of the octahedral and tetrahedral sheets is largely responsible for both the physical and chemical characteristics of clay mineral (Irabor *et al.*, 2021). This study is aimed at undertaking the physical, chemical and mineralogical characterization of a clay mineral deposit in Ihievbe in Owan East Local Government of Edo State, Nigeria with a view to ascertain its thermal and plastic properties

## MATERIALS AND METHODS

### Sampling:

The samples were collected on profile basis in the study site in Ihievbe, Owan East Local Government Area of Edo State, Nigeria between latitudes 7°. 006' 91''N and longitude 6°. 005' 22''E. The profile was determined by colour variation as described by Onyeobi and Imeokparia, (1994). Fifteen (15) samples were collected per depth using digger, spade and trowel all made of stainless steel. The samples were packaged in plastic bag, labelled, and air-dried in the laboratory. Determination of physical properties of the clay minerals such as moisture content, loss on ignition, atterberg limits and linear shrinkage, refractoriness, thermal shock resistance, bulk density were carried out as described by (Irabor and Ohenhen, 2018).

### MOISTURE CONTENT DETERMINATION

The moisture content was determined on dry weight basis. The test clay sample (5.0 g) was oven dried at a temperature of 105-110°C for three hours and weighed at one hour interval after cooling in a desiccator until a constant weight was obtained. The loss in weight was computed and expressed in terms of percentage (equation 1).

$$\% \text{ Moisture content} = \frac{\text{Loss in weight (g)}}{\text{weight of sample (g)}} \times 100 \quad 1$$

### LOSS ON IGNITION (L. O. I.)

The clay sample (5.0 g) was heated in a muffle furnace at 400°C for 4 hours after which it was cooled in desiccators and weighed. The difference in weight gave the loss of ignition. It is expressed in percentage terms (equation 2).

$$\% \text{ Loss on ignition} = \frac{\text{Loss in weight (g)}}{\text{weight of sample (g)}} \times 100 \quad 2$$

### ATTERBERG LIMITS DETERMINATION

#### Liquid Limit

The air-dried clay samples (120.0 g) from thoroughly mixed portion of material passing 425 microns sieve were obtained. Distilled water was added to the clay sample in a mixing disc to form uniform paste. A portion of the paste was placed in the cup of liquid limit device and spread into portions with few strokes of spatula and spread into depth of 1 cm at the point of maximum thickness. The clay sample in the cup was divided by the firm strokes of the grooving tool along the diameter through the centre line so those clean sharp grooves of proper dimensions were formed. The cup was lifted and dropped by turning crack at the rate of 2 revolutions/seconds until the two halves of clay sample came in contact with each other for a length of about 1cm by flow only. The number of blows required to cause the groove close for about 1cm was recorded. A representative portion of clay was taken from the cup for moisture content determination the test was repeated with different moisture contents at least three more times for

blows between 10-40. A flow curve was obtained by plotting a graph of moisture content (on Y-axis) and number of blows (on X-axis) The moisture content corresponding to 25 drops (blows) was read, which represented the liquid limit.

### Plastic Limit (PL) and Plasticity Index (PI)

The sieved clay sample (15.0g) was wet with water until it became plastic. The mix was rolled on a glass plate with the hand into thread of about 1.3mm in diameter until the thread showed sign of crumbling. The rolled soil was weighed and placed in an oven for 24hours after which the moisture content was determined. This process was repeated three times and the average % moisture content was determined. The values obtained were recorded as plastic limit to nearest whole number. The plasticity index was then calculated by the difference between % liquid and the % plastic limits .

### LINEAR SHRINKAGE

The sieved clay sample was wetted by addition of water and then placed in a shrinkage mould of dimension 140mm by 12.5 mm and the top smoothed with the aid of a spatula. The mould was placed in an oven set at a temperature range of 105-110°C for 24 hours. It was allowed to cool to room temperature after which the length of the oven-dried sample was measured and recorded. The difference in length was recorded (equation 3).

$$\text{Linear shrinkage \%} = \frac{\text{change in length}}{\text{original length}} \times 100 \quad 3$$

### BULK DENSITY

The test pieces were prepared and air-dried for 24hours, the clay samples were later oven dried at 110°C for 24 hours, heated to 1100°C, cooled in desiccator and weighed to nearest 0.001 g (**D**), after which the specimens were transferred to a beaker filled with water and heated for 30minutes to assist in releasing trapped air. The sample was cooled and soaked weight (**W**) taken. The sample was then suspended in water using a beaker placed on a balance and suspended weight (**S**) was taken, the bulk density was calculated from equation 4;

$$\text{Bulk Density} = \frac{D}{W-S} \times \rho \text{ (g/cm}^3\text{)} \quad 4$$

Where *D*= Dried weight, *W*= Soaked weight, *S*= Suspended weight,  $\rho$  = Density of water

### PERMEABILITY TO AIR

Test sample were prepared to specification of 5.08 cm diameter and 5.08 cm height from a standard rammer. The test pieces were air-dried for 24 hours and then dried at 110°C for 12 hours in an oven. Thereafter, 2000cm<sup>3</sup> of water was allowed to pass through the sample from the jar containing water. The time taken for 2000 cm<sup>3</sup> of water to displace equal volume of air through the test piece

was recorded. The pressure difference between the surfaces was measured by a manometer. Permeability was calculated from equation 5;

$$P_A = \frac{V \times h}{A \times p \times t} \quad 5$$

Where  $P_A$  = Permeability number

$V$  = Volume of air

$h$  = Height of sample

$A$  = Cross sectional area of specimen

$p$  = Pressure of air in cm of water

$t$  = Time in minutes

### APPARENT POROSITY

Disk samples were prepared and air dried for 24 hours. The samples were then oven dried at 110°C, the pieces were fired at 1100°C, cooled and then transferred into desiccator and weighed to the nearest 0.001 g (D). The samples were then transferred into a 250 ml beaker in an empty vacuum desiccators; water was then introduced into the beaker until the test pieces were completely immersed. The samples were allowed to soak in boiled water for 30 minutes being agitated from time to time to assist in releasing trapped air bubbles. The samples were transferred into empty desiccator to cool. The soaked weight ( $W$ ) was taken. The samples were then weighed suspended in water using a beaker placed on a digital weighing balance and this gave suspended weight ( $S$ ). The apparent porosity was calculated using the equation 6;

$$\text{Apparent porosity} = \frac{W-D}{W-S} \times 100 \quad 6$$

Where  $W$  = Soaked weight,  $D$  = Dried weight,  $S$  = Suspended weight

### Chemical Assay:

The chemical compositions of the mineral deposit were determined on dry samples by method described by (Irabor and Iwu, 2011; Irabor and Okeke, 2018). This was done by taking some quantity of the test sample, heated to 105 – 110°C for one hour to remove moisture, cooled in a desiccator and thereafter kept in a sealed container. The test sample (1.0g) was weighed into a beaker and digested in Aqua – regia. The determination of the chemical components of the samples were carried out using Atomic Absorption Spectrophotometer (Bulk Scientific 210VGP) and flame Photometer (model Sherwood 410) and the total silica content was determined by the standard method as described (Irabor and Okunkpolor, 2020).

### Mineralogical Analysis

The samples were pulverized to fine particles ( $\leq 2 \mu\text{m}$ ) as described by (Irabor and Iwu, 2011) and were subjected to X – ray diffraction using GBC Enhanced Mini Material Analyzer (EMMA) X-ray Diffractometer with Cu,  $K\alpha$

radiation source. The generator operating conditions were 40 KV and 5 SmA. The d-values obtained were compared with the standard X-ray powder diffraction file published by the Joint Committee on the Powder Diffraction Standards (JCPDS, 1980) for the identification of the different mineral species and Pei-Yuan(1977).

### RESULTS AND DISCUSSION

The results of the chemical, physical and mineralogical analysis of the Ihievbe clay mineral deposits are presented in Tables 1, 2 and 3 respectively. The chemical composition of the clay mineral deposit showed that it is made up of alumino-silicate minerals (Moore and Reynolds, 1997). Whereas kaolinite, montmorillonite, kyanite and quartz were found in both sites of the clay mineral deposit studied, halloysite, muscovite and ramsdellite were present in the site1 while goethite, naotocite were present at the site 2. Montmorillonite being an expanding (2:1) clay mineral would have influenced the swelling and shrinking properties of the clay mineral deposit (Segad *et al.*, 2010). Kaolinite is a (1:1) clay mineral which has low swelling, shrinking and high thermal characteristics all of which have contributed to the thermal behaviour of the clay mineral deposit (Deer *et al.*, 1992). Quartz is made up of silica oxide and it is known to cause reduction in plasticity, shrinkage and thermal properties (Singh *et al.*, 2019). Halloysite is a (1:1) kaolinite type of mineral which is a weathering product caused by the hydrothermal alteration of aluminosilicate mineral. It is often found intermixed with kaolinite, dickite and montmorillonite and possesses good thermal stability (Joussein *et al.*, 2005; Wilson and Keeling, 2016; Singer *et al.*, 2004). The presence of halloysite which is thermally stable would have had positive contribution to the thermal behaviour of the clay mineral deposit. Kyanite which is often used as an index mineral to define and trace a metamorphic zone that was subject to particular degree of metamorphism is particularly stable to heat (Winter and Ghose, 1979; Nesse, 2000) and therefore would have contributed to the thermal stability of the clay mineral deposit. Muscovite, a non-expanding silicate mineral was found present in site1. The refractory nature of muscovite and its array of colours has been established (Kalita and Wary, 2015). Therefore, its presence must have also contributed to both colour and the thermal stability of the clay mineral deposit characteristics. The mineral neotocite is a (1:1) phyllosilicate mineral though expanding contains manganese and iron silicate which exhibits variation in colour from black to brown (Emsley, 1991). The presence of neotocite and goethite a monohydrated iron oxide (Schwartzmann, 1988) largely contributed to the colouration of the clay mineral deposit. The liquid limit of the deposits (38-42%) showed that the clay has good swelling properties (Mitchell and Soga,

1993). The value of the plastic index (16-19%) showed that the clay mineral deposit fell within the medium plastic clay based on classification in terms of Atterberg Plastic Index (API) (15-30%) on classification of clay minerals (Seed *et al.*, 1997). The linear shrinkage of the deposits was between (7-9%) which fell within the recommended limit (4-10%) for refractory clays (Omowumi, 2000). The values of the apparent porosity (28-35%) slightly exceeded the recommended standard values (20-30%). The thermal shock resistance of

the clay mineral deposits (18-30 cycles) demonstrate the ability of clay minerals to withstand thermal shock (Onyemobi, 2002; Yami and Umaru, 2007). The refractoriness of the clay minerals deposits exceeded 1200°C. The combined properties of the clay mineral deposit showed how much the non-expanding (1:1) clay minerals present in the deposits influenced their overall behaviour especially with respect to thermal stability and plastic behaviour.

**Table 1: The Results of the Chemical Assay of Ihievbe Clay Mineral Deposit**

	Site 1	Site 2
Moisture( %)	1.07± 0.06	1.07± 0.09
L.O.I (%)	4.67 ± 0.25	3.93± 0.19
Al <sub>2</sub> O <sub>3</sub> (%)	20.42 ± 0.31	20.17± 0.38
SiO <sub>2</sub> (%)	42.82 ± 0.56	41.85 ± 0.66
Fe <sub>2</sub> O <sub>3</sub> (%)	13.75 ± 0.41	11.33 ± 0.12
K <sub>2</sub> O (%)	0.15 ± 0.01	0.17 ± 0.02
Na <sub>2</sub> O (%)	5.09 ± 0.23	12.04 ± 0.27
MgO (%)	0.06 ± 0.01	0.05 ± 0.01
CaO (%)	0.05 ± 0.01	0.05± 0.01

**Table 2: The Results of Analysis of the Physical Properties of Ihievbe Clay Mineral Deposit**

	Site 1	Site 2
Liquid limit %	38	42
Plastic limit %	22	23
Plastic index %	16	19
Linear shrinkage %	7	9
Bulk density ( g/cm <sup>3</sup> )	3.15	2.63
Apparent porosity(%)	35	28
Permeability (millidacies)	105	86
Thermal shock resistance (cycles)	18	30
Refractoriness	> 1200°C	>1200°C
Colour	Yellow	Brown

**Table 3: The Results of the Mineralogical Assay of Ihievbe Clay Mineral Deposit**

Mineral	d-value	Site 1	Site 2
Kaolinite Al <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub>	7.17, 3.58,1.49	Present	Present
Montmorillonite	4.48,3.34,2.49	Present	Present
Na <sub>3</sub> (AlMg) <sub>2</sub> Si <sub>4</sub> O <sub>10</sub> XH <sub>2</sub> O			
Halloysite Al <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (OH) <sub>4</sub> .2H <sub>2</sub> O	4.36,3.35,1.67	Present	Not Detected
Quartz SiO <sub>2</sub>	3.34,4.26,1.82	Present	Present
Muscovite KAl <sub>2</sub> (Si <sub>3</sub> Al)O <sub>10</sub> (OH) <sub>2</sub>	3.36,4.49,2.57	Present	Not Detected
Palygoskite( MgAl) <sub>5</sub> (SiAl) <sub>8</sub> O <sub>20</sub> (OH) <sub>2</sub> .8H <sub>2</sub> O	4.47,4.26	Present	Not Detected
Kyanite Al <sub>2</sub> SiO <sub>5</sub>	1.38,3.35,3.18,1.96	Present	Present
Naotocite(Mn,Mg,Fe)SiO <sub>3</sub> .H <sub>2</sub> O	1.36,3.36,1.54	Not Detected	Present
Goethite Fe <sub>2</sub> O <sub>3</sub> .OH	4.18,2.45	Present	Present
Ramsdelite	1.66,2.55,1.47	Present	

## CONCLUSION

The clay mineral deposits at Ihievbe have been studied. The results showed that the two clay mineral deposits had kaolinite, montmorillonite and kyanite as common aluminosilicate minerals present, whereas, muscovite and halloysite were present in site1, the minerals neotocite and goethite

were present in site 2. On the whole, the deposits consisted of mixed clays whose combined effect gave the deposits good thermal stability and could be used where the combination of colour, swelling properties and thermal stability are desirable.

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