ASSESSMENT OF THE REFRACTORY AND PLASTIC PROPERTIES OF CLAY MINERAL DEPOSITS IN UHONMORA, OWAN WEST LOCAL GOVERNMENT AREA IN EDO STATE, NIGERIA.

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ABSTRACT

In order to assess the plastic and thermal behaviour of the clay mineral deposits at Uhonmora, Edo State, Nigeria, the chemical, mineralogical and physical properties were evaluated. The results of the study showed that the deposits consisted of phyllosilicate minerals such as kaolinite, illite and clinochlore; other mineral present were quartz, sanidine high, geonalite, diaspare and and lepidocrocite. The clay minerals exhibited moderate swelling characteristics and medium plasticity; the linear shrinkage and apparent porosity were within the accepted values for refractory clays. It is reddish-brown colour on account of the presence of coloured iron mineral lepidocrocite. The clay minerals also exhibited good thermal shock resistance and refractoriness above 1200°C. The properties of the clay mineral deposits were reflection of the combined effects of the properties of the clay minerals and other non-clay mineral constituents. The study revealed that the clay mineral deposits studied had good refractory characteristics and can be used at operating temperature conditions above 1200°C.

Key words : phyllosilicates, swelling, plasticity, porosity and thermal stability

INTRODUCTION

The uses of clay in wide variety of both domestic and industrial applications such as pottery, ceramics, textile, paper, foundries, paint, insecticides, fillers, adhesives and in treatment of ailments have been reported and documented over the centuries[1-2]. Due to its wide range of industrial applications and the different requirements of the different industries, the compositions and nature of the clay mineral deposit are very critical both for its quality and commercial values [3-4]. Several types of clays with varied mineralogical, chemical composition and physical properties exists and have been reported in Nigeria [5-7]. However, many clay minerals deposit remain unidentified and uncharacterized [8-9]. Clays are naturally occurring materials composed of fined grained minerals with particle size less than 2 microns. When mixed with water, it becomes plastic and hardens when dried or fired [10-11]. It consists of hydrous aluminosilicate minerals which are arranged in well-ordered patterns comprising of octahedral and tetrahedral geometry which results in packed layers and sheets [12-13]. It is
either classified as (1:1) when it contains one tetrahedral silica sheet and one octahedral alumina sheet or (2:1) when there are two silica units to one octahedral alumina unit with the octahedral unit sandwiched between the silica sheets [14]. Very often, it contains other non-clay compounds such as alkali and alkaline earth metals, silicate minerals and non-aluminosilicate compounds all of which influences both the physical, mechanical and chemical behaviour of the clay materials [15]. The very important physical properties of clay are swelling and plastic behaviour when it absorbs water. Thus, clays are generally classified as expanding or non-expanding clays depending on the degree of swelling [16]. One of the major properties of clay which recommends it for application in ceramic and foundry industries is the capacity to withstand high temperature operations. Some clays are refractory while others are not [17]. These refractory properties are intrinsically linked to the clay mineral composition. The aim of this study is to evaluate the composition and properties of the Uhonmora clay mineral deposits and to assess both the swelling and refractory characteristics.

**Sampling**

The samples were collected on profile basis in the study sites in Uhonmora, Owan West Local Government Area, Edo State, Nigeria between Latitude 6°52′1″ N Longitude 5°56′54″ E. The profile was determined by colour variation [18]. A minimum of fifteen (15) samples were collected per site. The samples were labelled and air-dried in the laboratory. The 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 in accordance with standard method [19].

**Moisture content determination**

The moisture content was determined on a 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.

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

**Loss on ignition**

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.

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

**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, trimmed to a depth of 1cm 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 that clean sharp groove 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 (15.0 g) 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 24 hours after which the moisture content was determined. This process was repeated three times and the average %moisture content was determined. The values obtained was 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 140 mm by 12.5 mm and the top smoothened 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 oved-dried sample was measured and recorded. The difference in length was recorded.

\[ \text{Linear shrinkage %} = \frac{\text{change in length}}{\text{original length}} \times 100 \quad \ldots 3 \]
**Bulk density**

The test pieces were prepared and air-dried for 24 hours, 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 30 minutes to assist in releasing trapped air. The specimen was cooled and soaked weight (W) taken. The specimen 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 below:

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

Where:

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

**Permeability to air**

Test samples 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. 2000 cm$^3$ of water was allowed to pass through the sample from the jar containing water. The time taken for 2000 cm$^3$ 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 the equation below:

$$P_A = \frac{V \times h}{A \times P \times t}$$

Where

- $P_A$ = Permeability number
- V = Volume of air
- h = Height of specimen
- 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 specimens were then transferred into a 250 ml beaker in an empty vacuum desiccators; water was then introduced into the beaker until the test places were completely immersed. The specimens were allowed to soak in boiled water for 30 minutes being agitated from time to time to assist in releasing trapped air bubbles. The specimen was transferred into empty desiccator to cool. The soaked weight (W) was taken. The specimen was 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 below:

\[
\text{Apparent porosity} = \frac{W - D}{W - S} \times 100 \ldots \ldots 6
\]

Where:

- W = Soaked weight,
- D = Dried weight,
- S = Suspended weight

**Chemical Assay**

The chemical compositions of the mineral deposit were determined on dry samples obtained by taking some quantity of the test sample, heated to 105-110°C for one hour to remove moisture, cooled in a dessicator 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) in accordance with prescribed method[20] and the total silica content was determined by the standard method[21].

**Mineralogical Analysis**

The samples were pulverized to fine particles (≤2µm) and were subjected to X-ray diffraction using GBC Enhanced Mini Material Analyzer (EMMA) X-ray Diffractometer with Cu, Kα radiation source. The generator operating conditions were 40KV and 5SmA. The Diffractograms obtained were compared with the standard X-ray powder diffraction file published by the Joint Committee on the Powder Diffraction Standards for the identification of the different mineral species [22-23].
RESULTS AND DISCUSSION

Table 1: The Chemical Assay of the Clay Mineral Deposits in Uhonmora

<table>
<thead>
<tr>
<th>% Composition</th>
<th>Site 1</th>
<th>Site 2</th>
<th>Site 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>0.53 ± 0.09</td>
<td>0.61 ± 0.05</td>
<td>0.72 ± 0.02</td>
</tr>
<tr>
<td>Loss on Ignition ( L.O.I)</td>
<td>3.20 ± 0.12</td>
<td>4.27 ± 0.20</td>
<td>3.80 ± 0.40</td>
</tr>
<tr>
<td>Al2O3</td>
<td>26.75 ± 0.82</td>
<td>28.92 ± 0.76</td>
<td>27.54 ± 0.90</td>
</tr>
<tr>
<td>Fe2O3</td>
<td>7.42 ± 0.42</td>
<td>3.51 ± 0.60</td>
<td>6.89 ± 0.23</td>
</tr>
<tr>
<td>SiO2</td>
<td>50.32 ± 0.52</td>
<td>51.57 ± 0.61</td>
<td>53.72 ± 0.62</td>
</tr>
<tr>
<td>CaO</td>
<td>0.070± 0.01</td>
<td>0.09 ± 0.01</td>
<td>0.08 ± 0.02</td>
</tr>
<tr>
<td>Na2O</td>
<td>5.15 ± 0.32</td>
<td>4.51 ± 0.22</td>
<td>4.08 ± 0.31</td>
</tr>
<tr>
<td>K2O</td>
<td>0.20± 0.01</td>
<td>0.58 ± 0.01</td>
<td>0.76 ± 0.21</td>
</tr>
<tr>
<td>MgO</td>
<td>0.12 ± 0.01</td>
<td>0.37 ±0.01</td>
<td>0.42 ± 0.03</td>
</tr>
</tbody>
</table>

Table 2: Physical Properties of the Clay Mineral Deposits in Uhonmora

<table>
<thead>
<tr>
<th></th>
<th>Site 1</th>
<th>Site 2</th>
<th>Site 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquid limit %</td>
<td>33</td>
<td>30</td>
<td>33</td>
</tr>
<tr>
<td>Plastic limit%</td>
<td>9</td>
<td>11</td>
<td>14</td>
</tr>
<tr>
<td>Plasticity index%</td>
<td>24</td>
<td>19</td>
<td>14</td>
</tr>
<tr>
<td>Linear shrinkage%</td>
<td>24</td>
<td>19</td>
<td>19</td>
</tr>
<tr>
<td>Bulk density (g/cm³)</td>
<td>2.98</td>
<td>2.34</td>
<td>2.85</td>
</tr>
<tr>
<td>Apparent porosity</td>
<td>23</td>
<td>24</td>
<td>28</td>
</tr>
<tr>
<td>Air permeability ( milidacies)</td>
<td>78</td>
<td>92</td>
<td>88</td>
</tr>
<tr>
<td>Thermal Shock resistance ( Cycles)</td>
<td>26</td>
<td>24</td>
<td>30</td>
</tr>
<tr>
<td>Refractoriness</td>
<td>&gt;1200°C</td>
<td>&gt;1200°C</td>
<td>&gt;1200°C</td>
</tr>
</tbody>
</table>
Table 3: The Mineralogical Composition of the Clay Mineral Deposits in Uhonmora

<table>
<thead>
<tr>
<th>Mineral</th>
<th>d-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolinite Al$_2$Si$_2$O$_5$(OH)$_4$</td>
<td>7.19, 3.58, 1.49</td>
</tr>
<tr>
<td>Clinochlore (MgAl)$_6$(SiAl)$<em>4$O$</em>{10}$(OH)$_8$</td>
<td>7.16, 3.58, 2.45</td>
</tr>
<tr>
<td>Illite K$<em>{0.7}$Al$</em>{2.1}$(SiAl)$<em>4$O$</em>{10}$(OH)$_2$</td>
<td>3.35, 2.58, 4.49</td>
</tr>
<tr>
<td>Quartz SiO$_2$</td>
<td>3.34, 2.58, 1.82</td>
</tr>
<tr>
<td>Diaspore Al$_2$O$_3$.H$_2$O</td>
<td>2.13, 3.99, 2.32</td>
</tr>
<tr>
<td>Sanidine High KAlSi$_3$O$_8$</td>
<td>3.33, 4.24, 3.79</td>
</tr>
<tr>
<td>Geonalite (Fe,Mn)Si$_2$O$_5$(OH)$_4$</td>
<td>2.47, 7.22, 3.58</td>
</tr>
<tr>
<td>Lepidocrocite FeO.OH</td>
<td>2.47, 6.26, 3.29</td>
</tr>
</tbody>
</table>

The results of the chemical analysis of the Uhunmora clay are contained in tables 1, 2 and 3. The chemical composition revealed that the moisture content (0.53-0.72%), loss on ignition (3.20-4.27%), Al$_2$O$_3$ (26.75-28.92%), Fe$_2$O$_3$ (3.51-7.42%), SiO$_2$ (50.32-53.72%), CaO (0.07-0.09%), Na$_2$O (4.08-5.15%) and K$_2$O (0.20-0.76) were present. The results of the physical properties as presented in Table 2 showed that the liquid limit (30-33%), plastic limit (9-14%), plastic index (19-24%), bulk density (2.34-2.98 g/cm$^3$), apparent porosity (23-28%), air permeability (78-92 millidacies), thermal shock resistance (24-30 cycles) and refractoriness was greater than 1200°C. The mineralogical studies as contained in Table 3 showed that deposits contained the following minerals kaolinite (Al$_2$Si$_2$O$_5$(OH)$_4$), clinochlore(MgAl)$_6$(SiAl)$_4$O$_{10}$(OH)$_8$ , illite (K$_{0.7}$ Al$_{2.1}$(SiAl)$_4$O$_{10}$(OH)$_2$ , quartz (SiO$_2$), diaspore (Al$_2$O$_3$.H$_2$O), sanidine High(KAlSi$_3$O$_8$), geonalite ((Fe,Mn)Si$_2$O$_5$(OH)$_4$) and lepidocrocite (FeO.OH). The chemical composition indicates high content of silica and alumina which showed that it is an aluminosilicate mineral deposit [24]. The moisture content was very low compared to the loss on ignition which indicated that the lost water was more of the chemically bound water molecule in the clay structure. The clay mineral clinochlore is a member of the chlorite group of minerals which is a secondary mineral found in hydrothermal alteration of intermediate aluminosilicate containing magnesium end rich member [25]. It is a 1:1 aluminosilicate mineral which is an expanding clay [26]. Another important clay mineral found in the deposit sites was kaolinite which is a 1:1 phyllosilicate, with characteristic non-expanding, low swelling and shrinking properties as well as high thermal...
resistance [27]. The observable low shrinkage, high thermal resistance and thermal stability of the clay would have been greatly enhanced by the presence of kaolinite. The clay mineral illite was present. It is a non-expanding clay mineral which can also be formed by the hydrothermal conversion of kaolinite, has higher ability to absorb water than kaolinite and may have contributed to the swelling properties of the clay [28-29]. The clay mineral deposit was found to contain diaspore, a monohydrate aluminium oxide mineral whose refractory properties has been well established and documented [30]. The presence of this mineral would have largely contributed to the thermal stability and refractoriness of the clay mineral. The iron oxide existed as lepidocrocite, a monohydrate form of the oxide which is reddish-brown in colour and could be partly responsible for the colouration of the clay mineral deposit [31]. The k-feldspar mineral sanidine high was detected. This mineral is a precursor to the formation of kaolinite through hydrothermal weathering processes [32]. The value of the liquid limit (average 32%) showed that the clay is not a high swelling clay [33] and the value of the plasticity index (19-25%) showed that the clay mineral deposit fell within the medium plastic clay based on the classification in terms of Atterberg Plastic Index (API) (15-30%) based on the classification of the clay mineral [34]. The values obtained for the plasticity index were similar to those obtained in previously reported study [35], but there was disparity with respect to the liquid limit. The linear shrinkage of the studied clay deposits (average 8%) fell within the the recommended limit (4-10%) for refractory clays. The apparent porosity values were within the acceptable limit for refractory clays (20-30%), the average value of permeability (86 millidacies) of the clay mineral was within the accepted range (25-90 millidacies). The values of the bulk density (2.34-2.98g/cm³) however exceeded the internationally permissible limit (1.7-2.1g/cm³) and the thermal shock resistance met the standard set for refractory clays [36]. The refractoriness of the clay exceeded 1200°C. This demonstrates that the clay can be used for high temperature operations.

CONCLUSION

The clay mineral deposit at Uhonmora has been evaluated for its chemical, physical and mineralogical properties in order to access its refractory behaviour. It was found to consist of alumino-silicate minerals. Mineralogically, it comprised essentially of kaolinite, illite and clinochlore clay minerals. Other minerals present were quartz, diaspore, lepidocrocite, sanidine high and geonalite. The combined effect of the minerals resulted in the clay exhibiting medium plastic behaviour, good thermal shock resistance and refractoriness above 1200°C.
REFERENCES


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