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# The Mineralogical, and XRD Analyses Characterized Clay and Clay Minerals of Bida

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**Abstract**

The detail physical, chemical and surface properties of clays mainly depend on their mineralogical compositions. The main objective of this research was to study the mineralogical and chemical composition of clay obtain from a clay deposit in Bida Niger State. The clay sample was collected from a deposit in Minna, Nigeria. It was cleaned, soaked, dried, crushed and sieved then moulded to some definite shapes depending on the type of test carried out on the various clay samples.The qualitative mineralogical phase identification was performed by X-ray diffraction (XRD) in powder samples using an Analytical Empyrean, diffraction DY674. In determining the chemical constituents of the clay samples in terms of the individual oxides; the analysis of the samples was carried out. The mineralogical composition of clay, 20.854° of intensity 18.7% and 26.634° of intensity 100.0% was observed to be quartz minerals, while the mineralogical composition of clay, 12.318° of intensity 100.0% and 19.801° of intensity 80.0% was observed to be kaolinite minerals. It was observed that the high abundance of quartz minerals in the clay deposits sample shows that the clay have high silica content, and are bound to be strongly acidic as the silica provides several sites for the hydroxyl groups or water molecules to bind. These results reveal that the dominant clay mineral in the clay deposits at Bida is kaolinite.It is not surprising that clays from these areas have been used for pottery, ceramic, bricks and tiles.

Key words: Kaolinite, Mineralogy, Chemical Composition, X-Ray Diffraction

## Introduction

Clay is a very fine grained, unconsolidated rock matter, which is plastic when wet, but becomes hard and stony when heated. When mixed with water, it becomes plastic and mouldable and becomes hard again on drying and firing. It has its origin in natural processes, mostly complex weathering, transported and deposited by sedimentation within geological periods. Clay is composed of silica ($SiO_2$), Alumina ($Al_2O_3$) and water ($H_2O$) plus appreciable concentration of oxides of iron, alkali and alkaline earth, and contains groups of crystalline substances known as clay minerals such as quartz, feldspar and mica. Clay is used in various fields such as geology, chemistry, soil science, engineering and ceramic technology (Folaranmi, 2009).

Earlier researches have done some work on various refractory clay deposits in Nigeria and have found them suitable for use in metallurgical industries with some additives (Maluet al., 2018; Abubakar et al, 2014; Abuhet al., 2014). To this end, the society has increased the various steel products coming out of the steel plant of Ovwian-Aladja, Osogbo, Jos, Katsina and Ajaokuta.Clay has phyllosilicate minerals that impart plasticity

to clay and which harden upon firing or drying (Grace and Seun, 2016; Guggenheim and Martin, 1993). Clays are broadly categorised as kaolinites and smectites (Mark, 2010; Abuhet al., 2014). The kaolinites include kaolinite, halloysite, and dickite(Longstaffe, 1981) and the smectites include montmorillonite, nontronite, saponite and hectorite(Bailey and Brindley, 1979)

In a research by Ovat and Bisong (2017), clay samples from Cross River State (Idere and Ito) were characterized. The results of the physical and chemical properties of both Idere and Ito clay samples showed that, these clays can be used for brick making, floor tiles and stoneware.several studies have been done on clay soils around Nigeria. They include Mayo-Belwa in Adamawa (Kefas et al., 2007), Kachia, Kafanchan, Wusasa in Kaduna (Manukaji,2013); Sheda, Abaji and Karimu in Abuja (Manukaji,2013); Olokoro, Ukpor, Otamiri and Nsu in South eastern Nigeria (Nwoye, 2010), Abakiliki in Ebonyi (Nweke et al.,2007) and Ugbegun clay deposit in Edo central Nigeria (Ogbebor et al.,2010) amongst others.

Clay materials are basically divided into three groups; Those that contain mainly Kaolinites which are white, grayish-white or slightly colored becoming darker and plastic when moistened with water (Lopez-Galindo et al., 2007 and Nwoye, 2010).The second group are those that contain mainly Montmorillonite and the third group of clays are the intermediate product of disintegration of mica into kaolin (Nwoye, 2010).The presence of smectite in clays and clay minerals is an indication that the clay can be used as fullers or bleaching earths and the presence of kaolinites gives chance of getting clays for ceramics and pottery works (Bailey and Brindley, 1979).The presence of $Fe_2O_3$, MgO, CaO, $Na_2O$ (mineral oxide) in clay sample determines its uses in bricks, floors tiles and paper making (Kefas et al, 2007). While the presence of $Na_2O$, $K_2O$ and CaO (Alkali metal oxides) help to indicate refractory potential hence making clays suitable for ceramic products. Clay has also been applied in pharmacy and cosmetics though such clay materials have variable composition (Lopez-Galindo, 2007).

As deposits of clay raw material are widelydistributed in Nigeria, In order to determine the profitabilityof utilizing clay from a particular deposit for any application,it is of paramount importance to examine the microstructuralmorphology, determine the mineralogicalcomposition andanalyses the various available phases in such clay deposit(Giray *et al.*, 2013; Nweke and Egwu, 2007). To ascertain the viability and industrial importance, this research work was set out to study the mineralogical and chemical composition of clay mineral obtained from a clay deposit in Bida, Niger State.

**Materials and Method**

The materials used in this work were clay (obtained from Bida, Niger State) and water, and the equipment included Oven, Weighing balance, Sieve shaker and sieve pan, Venier calliper, Thermometer (mercury in glass), and X-ray diffraction machine.

**Methodology**

The clay was characterized in terms of its mineralogical and chemical composition.

**Determination of Mineralogical Composition**

The qualitative mineralogical phase identification was performed by X-ray diffraction (XRD) in powder samples using an Analytical Empyrean, diffraction DY674, a copper anode material manufactured by panalytical, Holland. The samples was pulverized (grind to fine powder) using arget pulverizing machine. The ground samples was ensured to press 150 micro mesh sieves, this was to ensure homogeneity of the samples. The powdered samples were them packaged and labelled, ready for the analysis. It was carried out at the National Geosciences Research Laboratory (NGRL), Kaduna.

**Determination of Chemical Composition**

In determining the chemical constituents of the clay samples in terms of the individual oxides; the analysis of the samples was carried out as follows:

**Determination of Percentage Silica $SiO_2$.**

0.5g of grind samples was weighed into a platinum crucible. This was fused with potassium carbonate and sodium tetra carbonate in the ratio of 4:1. Fusion takes place in the muffle furnace at 1100°C for a period of 1hour. The solution was evaporated on a hot placed on the plate to minimise over heating during baking.

The baked mass was dissolved in hydrochloric ratio 1:1, and washed with ionised hot water. The residue was $SiO_2$, while the fettered solution containing oxides was kept aside. The residue on the filter paper was placed in a platinum crucible and burnt at 800°C. Burning was continued until the carbon in it is burnt leading to no trace of black particle.

The crucible and content was weighed as ($W_1$) after cooling. Three drop of concentrated sulphuric acid and 5ml of hydrofluoric acid were added into the crucible. The platinum crucible was then placed on the plate in oven completely evaporated of its content. This was noticed when the water film of the sulphuric acid disappears at about 800°C for 10minutes and was then allowed to cool in a desiccators ($W_2$). The percentage silica content was determined from the formula.

$$\%SiO_2 = \frac{W_1 - W_2}{W_1} \times 100\% \quad (1)$$

**Determination of Percentage $Fe_2O_3$.**

From the shock solution, 25ml was measured into a beaker. Then 3ml of hydrogen peroxide $H_2O_2$ was added into the beaker and warmed but once boiling was observed, it was taken off the hot plate to cool. The mixture was then precipitated using ammonium hydroxide ($NH_4OH$) using the ratio 1:1. The solution was then filtered using filter paper into a 500ml flask. Ionised water was used to fill the flask to mark. The residue on the filter paper was taken into a beaker and dissolved in hydrochloric acid (HCl) at a proportion of 1:1.

The solution turned yellowish because of the presence of iron. Then drops of Tin II chloride ($SnCl_2$) was used to titrate the mixture until colour disappeared at the end point. 5% of mercury (II) chloride ($HgCl_2$) solution was prepared and 10ml of it was added to the mixture 30% of mixture of sulphuric acid and phosphoric acid, mixed at a ratio of 1:1 was added. 100ml of ionised water was added and 8 drop of di-phenylamine indicator. Potassium dichromate was then used to titrate the solution until end point. The end point was indicated when the

colour changed to pink. The percentage $Fe_2O_3$ was calculated from formula.

% $Fe_2O_3$

$$= \frac{\text{volume of potassium dichromate} \times 0.006 \times 142}{0.2} \times 100\% \quad (2)$$

**Determination of Percentage $Al_2O_3$ (Gravitational Method).**

From the solution, 25ml was measured into a beaker. 3ml of hydrogen peroxide was added and solution was warmed. On boiling, it was brought down from the hot plate and allowed to cool. The solution was precipitated using ammonium hydroxide at a ratio of 1:1. It was then filtered and washed properly with ionised water. The residue on the filter paper was taken out to an already weighed platinum crucible ($W_1$). The crucible contents were burns at 800°C. Burning continued until both filter papers, crucible content is completely burnt and brownish colour was obtained. The colour was brown because of the presence of Fe. The crucible was transferred to a muffle furnace and the temperature rose to 1100°C and weighed ($W_2$). To calculate for percentage of $Al_2O_3$.

$$\%Al_2O_3 = R_2O_3 - Fe_2O_3 \quad (3)$$

$$\%CaO = \frac{\text{Volume of potassium permanganate} \times 0.28}{0.025 \times 1000} \times 100\% \quad (5)$$

**Determination of Percentage MgO**

To determine MgO, the filtrate solution for the determination of $Al_2CO_3$ was used. 25ml was taken from the filtrate of $Al_2O_3$ determination and put into a flask. 2ml of aluminium chloride salt was added with concentrated aluminium hydroxide until a PH of about 11-12 was obtained. Powdered Eric- chrome indicator was added to indicate the presence of Ca and Mg in solution. The presence of Ca and Mg was indicated in the solution when it changed from pink colour once the powdered

$$R_2O_3 = \frac{W_2 - W_1}{0.25} \times 100 \quad (4)$$

Where $W_2 - W_1$ = Weight of residue.

Where $R_2$ is subscript for combined oxides in the precipitated solution.

**Determination of Percentage CaO**

To determine for CaO, the filtered solution for determining $Fe_2O_3$ was used. 25ml was taken from the filtered and put into flask. It was heated to between 60°C-70°C and then 20ml of 5% prepared aluminium oxalate and 5ml of acetic acid ($CH_2COOH$) added in aratio of 1:1. The solution was allowed to cool until room temperature for 2 hours. The solution was then filtered using filter paper and washed with hot ionised water. The filter paper and residue was put into a beaker and 20ml of sulphuric acid and 100ml of ionised water added in a proportion of 4:1. The beaker content was heated to 70°C. The mixture was then titrated with 0.1m potassium permanganate until end point was reached. The end point was obtained when the colour changed completely from purple to pink.

Eric-chrome indicator was added. Any other coloration indicates the absence of Ca and Mg.

When the solution turned pink, confirming the presence of Ca and Mg, then titration was carried out with 0.005M of EDTA. The end point of the titration was light green. Calculation for MgO is:

$$\%MgO = \frac{V_1 - V_2 \times 0.005}{0.025 \times 1000} \times 100 \quad (6)$$

Where $V_1$ = volume of EDTA consumed

$V_2$ = volume of potassium permanganate consumed in CaO titration.
2.04 = Equivalent factor.

**Result and Discussion**

**Mineralogical Composition of Clay**

From table 4.1 of the mineralogical composition of clay, 20.854° of intensity 18.7% and 26.634° of intensity 100.0% was observed to be quartz minerals and from table 4.2 the mineralogical composition of clay, 12.318° of intensity 100.0% and 19.801° of intensity 80.0% was observed to be kaolinite minerals. It was observed that the high abundance of quartz minerals in the clay deposits sample shows that the clay has high silica content, and are bound to be strongly acidic as the silica provides several sites for the hydroxyl groups or water molecules to bind.

From the result of the mineralogical composition of clay shown in appendix, it was observed that, table 4.1, the silicon oxide contains quartz minerals and mixture of quartz and kaolinite minerals. Also table 4.2, Aluminum silicate hydroxide contain kaolinite minerals.

Table 4.1 and 4.2 gives the result of mineralogy composition of clay sample.

Table 4.1: Mineralogical Composition of Clay $SiO_2$

| S/N | °2THETA (deg) | INTENSITY (%) |
|---|---|---|
| 1 | 20.854 | 18.7 |
| 2 | 26.634 | 100.0 |
| 3 | 36.537 | 7.7 |
| 4 | 39.458 | 7.0 |
| 5 | 40.282 | 2.6 |
| 6 | 42.442 | 4.7 |
| 7 | 45.785 | 2.3 |
| 8 | 50.129 | 11.8 |
| 9 | 50.608 | 0.4 |
| 10 | 54.862 | 3.2 |
| 11 | 55.314 | 1.4 |
| 12 | 57.219 | 0.2 |
| 13 | 59.994 | 8.4 |
| 14 | 64.021 | 1.5 |
| 15 | 65.771 | 0.3 |
| 16 | 67.727 | 4.6 |
| 17 | 68.125 | 6.1 |
| 18 | 68.294 | 6.1 |
| 19 | 73.449 | 1.8 |
| 20 | 75.642 | 2.4 |
| 21 | 77.654 | 1.3 |
| 22 | 79.862 | 2.3 |
| 23 | 80.022 | 1.4 |

Table 4.2: Mineralogical Composition of Clay $Al_2Si_2O_5(OH)$

| S/N | °2THETA (deg) | INTENSITY (%) |
|---|---|---|
| 1 | 12.318 | 100.0 |
| 2 | 19.801 | 80.0 |
| 3 | 24.851 | 100.0 |
| 4 | 34.963 | 80.0 |
| 5 | 35.862 | 80.0 |
| 6 | 37.670 | 80.0 |
| 7 | 38.422 | 90.0 |
| 8 | 40.875 | 10.0 |
| 9 | 45.571 | 40.0 |
| 10 | 51.008 | 40.0 |
| 11 | 55.080 | 50.0 |
| 12 | 59.983 | 10.0 |
| 13 | 62.353 | 100.0 |
| 14 | 63.785 | 30.0 |
| 15 | 65.084 | 20.0 |
| 16 | 68.142 | 10.0 |
| 17 | 70.238 | 10.0 |
| 18 | 72.083 | 10.0 |
| 19 | 73.528 | 30.0 |
| 20 | 75.025 | 10.0 |
| 21 | 76.156 | 5.0 |
| 22 | 77.103 | 30.0 |
| 23 | 80.353 | 10.0 |

Figure 4.1 and Figure 4.2, shows the mineralogical composition pattern of the clayey body, predominance of silica and alumina.

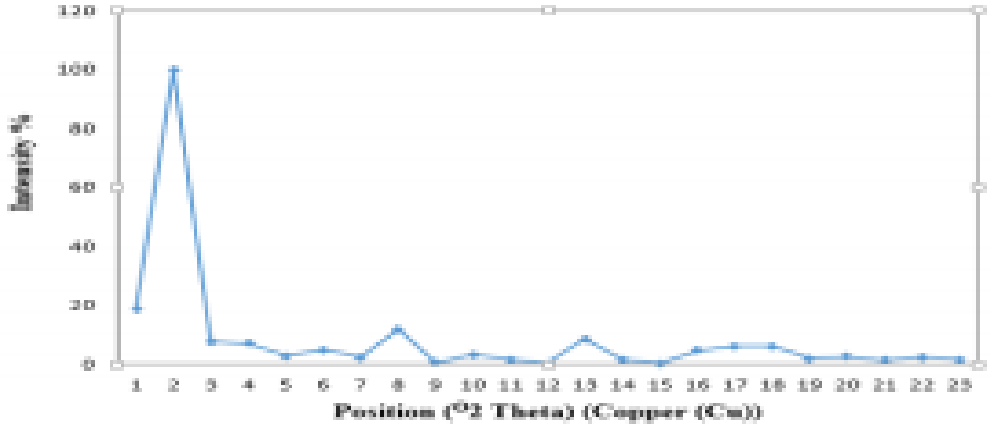


Figure 4.1: Pattern of Silicon Oxide

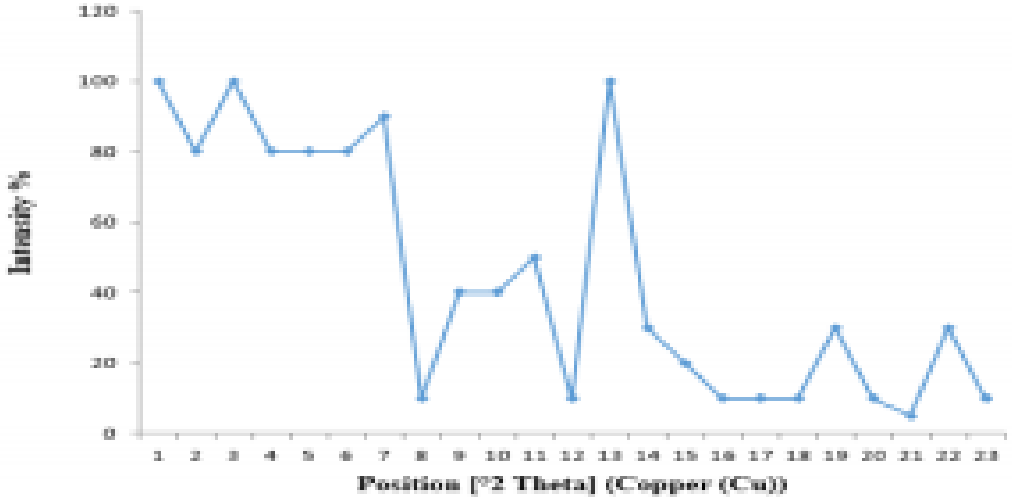


Figure 4.2: Pattern for Aluminium Silicate Hydroxide

The qualitative mineralogical phase identification was performed by X-ray diffraction (XRD) in powder samples using an Analytical Empyrean, diffraction DY674. These results reveal that the dominant clay mineral in the clay deposits at Bida is kaolinite and this coincides with the findings about the clays mined from Bida. It is not surprising that clays from these areas have been used for pottery, ceramic, bricks and tiles.

**Chemical Composition of the Raw Materials (wt)%.**

The results of the chemical composition of the clay and steel chips samples without an additive are presented in Tables 4.3 and 4.4, respectively.

In a study involving clays, it is important to establish the elemental constitution of the solid because surface and bleaching properties of clays and clay material depend on the elements present.This analysis was conducted to express the chemical composition of the clay sample in terms of its oxides. The classical chemical analysis (wet analysis) technique was used in this investigation. Table 4.3, shows the chemical composition and the Table 4.3: Chemical Composition of Clay Sample

| Oxide | % Chemical Composition |
|---|---|
| $SiO_2$ | 56.96 |
| $Al_2O_3$ | 18.19 |
| $Fe_2O_3$ | 6.39 |
| $TiO_2$ | 1.06 |
| CaO | 0.24 |
| MgO | 0.74 |
| $K_2O$ | 1.24 |
| $Na_2O$ | 0.18 |
| $ZnO_2$ | 0.03 |
| Loi | 9.81 |

loss on ignition (LOI) of the raw materials. The chemical composition of the clay is a typical kaolinite-based material with low amounts of alkaline oxides and relatively high amount of $Al_2O_3$. The percentage of 6.39% of $Fe_2O_3$ is responsible for the natural red colour

after firing. The high percentage of LOI indicates an elevated fraction of clay minerals.

**Conclusion and Recommendations**

The mineralogical and chemical characterization, and XRD Analyses of Clay and Clay Minerals of Bida, Niger State was carried out. The mineralogical composition results show that the clay sample from Bida is kaolinite, which does not expand and its water absorption is low, preferable for ceramics. The quartz minerals indicate the present of silicon oxide which provide sites for the water molecule to bind, and the Aluminum silicate hydroxide contain kaolinite minerals.

To complement this research, further research work should be carried out to understudy the physical properties as bulk properties, Loss of ignition, Green compression and shear strength, dry compression and shear strength, Green permeability and moisture content, thermal conductivity, etc., to fully characterize the clay materials from Bida, Niger State Clay deposits; to give a broader picture on the application of this materials other than in pottery and bricks making and further provide firsthand information on whether there is need for additives and the kinds of additive necessary to enhance further utilization of this important raw material.

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