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CHARACTERIZATION OF CLAY FROM OZA-NOGOGO IN DELTA STATE, NIGERIA FOR POTENTIAL INDUSTRIAL USES

S. E. Uwadiae* and V. Ekeleme

(Department of Chemical Engineering, Faculty of Engineering, University of Benin, PMB 1154, Benin City, Edo State, Nigeria)

***Corresponding Author's E-mail:** sylvester.uwadiae@uniben.edu.ng

Abstract

The aim of this study was to characterize clay from Oza-Nogogo in Delta State for its physical and chemical composition in order to assess its potential industrial applications. The clay samples were collected by means of a plastic shovel and digger, hand-picked to minimize the possibility of contamination and placed in a small polyethene bags. The samples were dried at ambient temperature for 10 days, pulverized and sieved screened to obtain fine particles before analysis. The physiochemical properties were determined using x-ray diffraction (XRD) for the mineralogical composition, X-ray florescence (XRF) for the chemical composition and Scanning Electron Microscope (SEM) for the microstructures of the clay sample. The cation exchange capacity (CEC), specific surface area and pore volume of the sample were also determined. The XRD studies showed that the clay sample is predominantly kaolinitic with some quantities of quartz, illite and feldspar. Kaolinite being the dominant clay mineral alone constitutes between 70-76% and quartz ranged from 22-28% and other minerals ranged from 1-6%. The XRF analysis showed that the sample contained high amount of silica (SiO_2) by mass of 51.3% followed by Alumina (Al_2O_3) 40%, iron oxide (Fe_2O_3) 3.703%. All other minerals present were in negligible proportion. The major elemental contents of clay samples detected in the study were Si, Al, Fe, Ti and K. The SEM micrographs showed the relative sizes in the clay particles and revealed that the clay particles seem to consist of much smaller platelets which indicated that the clay sample was made up of very fine particles. The specific surface area, CEC and pore diameter were 7.846 m^2/g , 6.8 meq/100 g and 28.642 nm respectively. Due to the high kaolinite content, Oza-Nogogo clay showed a potential of a basic raw material for pharmaceutical, drugs, paints, paper and ceramic industries.

Keywords: X-ray diffraction; X-ray florescence; Scanning Electron Microscope; Cation Exchange Capacity, Oza-Nogogo clay

1. Introduction

Apart from petroleum, gas and coal resources, the exploration, mining and exploitation of mineral resources in Nigeria have not received sufficient attention (Abolarin *et al.,* 2006). Delving into geological survey of Nigerian soil, it was reported that clay as one of the major Nigerian mineral deposits cover an estimated proven reserves of billions of tones (Durotoye and Elueze, 1989).

Clays have been severally defined as anhydrous complex compounds of alumina (Al_2O_3) and silica (SiO_3) that exist in various proportions and contain varied amounts of impurities of iron, organic matters and residual minerals (Akinfolarin and Awopetu, 2014); naturally occurring material that composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and susceptible to hardening when fired at high temperature (Dogan *et al.,* 2002) and abundant fine earthly powders produced by the weathering and disintegration of granite and feldspathic rocks (Nweke and Ugwu, 2007). Although clay usually contains phyllosilicates, it may contain other materials that could impart plasticity when wet and also harden when fired. However, associated phases in clay may include organic matter and materials that do not impart plasticity (Bakker, 1993).They have varying chemical composition depending on both the physical and chemical changes in the environment where clay deposits are found (Salawudeen *et al.,* 2010). The nature of clay and its composition determines, not only its quality and commercial value but also, to a large extent, its engineering behaviour

(Grim, 1968 and Onyeobi *et al.*, 2013). Among the characteristics of clays that influence their engineering performance are clay mineral composition, physical properties such as particle size distribution, porosity, structure and geologic history.

Clay minerals share a basic set of structural and chemical characteristic and yet 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 leads to their applications in extremely diverse fields. The specific clay minerals are identified by several techniques including thermal differential analysis, scanning electron microscope, infrared spectrometry and X-ray diffraction. Chemical analysis is an essential step to establish the nature of minerals (Newman, 1987).

Depending on the physical and chemical characteristics, clays may find application in a number of industries such as plastics, paint, ceramics, ink, catalysts, pharmaceutical and fibre glass among others (Murray, 1980; Emufurieta *et al*., 1992).

Despite the abundance clay deposits in Nigeria, there is a dearth of information on their characterization for various applications in agriculture, industry and environment. It is the aim of this study to characterize clay from Ozan-Nogogo using different analytical techniques with the view to assessing their physicochemical properties and relating them to their suitability for use in the relevant industries.

2. Methodology

2.1 Sample Colletion and Preparation

The clay samples used in this study, were collected from the deposits at Oza-Nogogo in Ika South Local Government Area of Delta State, Nigeria, at depths of 40 cm with the aid of shovel and digger and then hand-picked to minimize the possibility of contamination. The samples were dried at ambient temperature for 10 days. Thereafter, the samples were finely ground with a mortar and passed through a 2 mm mesh sieve (Njoka *et al.*, 2015) to obtain very fine particles used in analysis.

2.2 Characterization of Clay Samples

Phenom Scanning Electron Microscope (Model Pro X) with energy dispersive X-ray spectrometer was used to determine the microstructures of the clay samples (Osabor *et al*., 2009). Qualitative chemical analysis of minerals was carried out on the clay samples to produce Backscattered images (BSI) (Osabor *et al.*, 2009). The mineralogical composition of the clay was obtained by XRD (Empyrean XRD, Panalytical BV of Netherland) studies. (Chiari *et al.,* 2003) X-ray Fluorescence Spectrometer (Mini Pal for EDXRF) was used to determine the chemical composition of the clay samples (Dean *et al.*, 2004). Cation exchange capacity (CEC) of the clay was determined using the $BaCl_2$ compulsive exchange method as prescribed by Soil Science Society of America (Sumner and Miller, 1996). The specific surface area of the samples was determined using Micromeritics instrument (Tristar 3000) and by using Brunauer–Emmett–Teller (BET) method. The specific surface area of Oza-Nogogo clay was determined from the Brunauer,Emmett and Teller (BET) multipoint method (Bruanuer *et al.*, 1938; Brame and Griggs 2016).

Also pore volume was determined using the surface area analyzer which utilizes the BET theory for the analysis and plots of each sample data and then presents the results of pore volume (De Lange *et al.*, 2014). Three replicates was done and average value was calculated.

3. Results and Discussion

The X-ray diffractogram result of the Oza-Nogogo clay sample is shown in Figure 1.

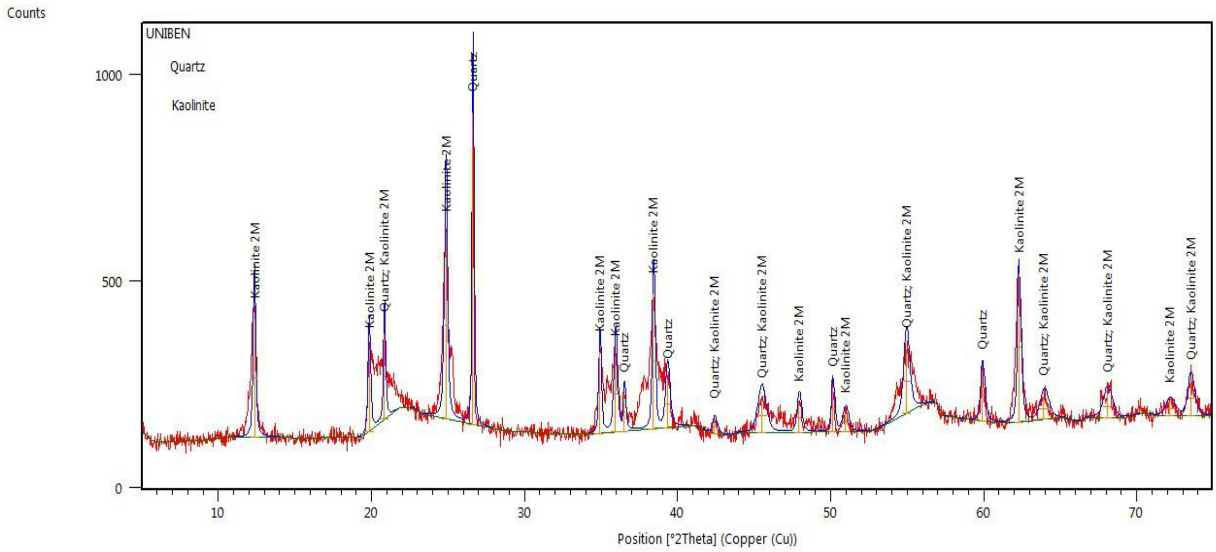

Figure 1: X-ray diffraction pattern of Oza-Nogogo clay

The diffractogram (Figure 1) shows the X-ray diffraction pattern of the clay sample. The results of mineralogical analysis of the clay samples in the present investigation show that the clay samples are predominantly kaolinitic and quartz. Careful investigation of Figure 1 reveals that the samples were composed essentially of $Al_8Si_8O_{36}H_0$ (kaolinite 2M) and Si_3O_6(Quartz). Kaolinite group observed at the following peaks 12, 20, 25, 35,36,38,48, 51,63 and 72Å while quartz were seen att the following peaks: 27, 37, 39, 50 and 60Å. A simple comparison with the mineral composition of some well-known clay deposits indicates that the investigated deposit is similar to Kaduna and China clay deposits which also had kaolinite and quartz with very small variations in the mineral contents (Osabor et al., 2009). The clay samples are all of sedimentary origins and seem to have gone through different level of transformation before depositions, which affects their physical properties like plasticity and shrinkage (Osabor *et al.*, 2009). Due to the high kaolinite content, this clay can serve as basic raw materials for pharmaceutical, drugs, paints, paper and ceramic industries.

The results of the X-ray Fluorescence analysis of the elements in the clay sample are shown in Table 1.

In the XRF analysis, the chemical compounds present in the clay and their concentrations were obtained with major elements present expressed in form of their oxides. The XRF result shows the compounds presents with its related concentration unit. From the result obtained (Table 1)it is observed that the sample contains high amount of silica by mass of 51.3% followed by Alumina 40%. All other minerals present are in negligible proportion. The major elemental contents of clay samples detected in the study were Si, Al, Fe, Ti and K.

The sample is found to be a mixture of kaolinite and illite in various proportLow electrical conductivity and high permeability can be brought about by high levels of quartz and low levels of clay mineral content.

Table 1: Chemical composition of Oza-Nogogo clay sample

Oxide	Percent
Al_2O_3	40
SiO_2	51.3
K_2O	0.274
CaO	0.144
TiO_2	3.06
V_2O_5	0.11
Cr_2O_3	0.02
MnO	0.009
Fe_2O_3	3.703
NiO	0.006
CuO	0.008
ZnO	0.001
Ga_2O_3	0.021
Ag_2O	0.74
Ta_2O_5	0.03
Re_2O_7	0.093
Bi_2O_3	0.094

The low plasticity generally exhibited by kaolinitic clay materials might be due to the levels of mineral oxide impurities such as TiO_2, MnO, and Fe_2O_3 present in the clay and the slightly low shrinkage is associated with the presence of high amount of quartz which tends to decrease the magnitude of shrinkage (Ombaka, 2016).

Clay from Oza-Nogogo can be used for refractory purposes since Ryan (1976) showed that for good refractory characteristics, clay should have a percentage composition of Al_2O_3, between 30 and 50% with a limited amount of Fe_2O_3, TiO_2 and CaO. Also, for kaolinite mineral to be utilized industrially, other accessory minerals such as quartz should first be reduced to acceptable levels through appropriate beneficiation techniques (Njoka *et al*., 2015).

The SEM micrograph of Oza-Nogogo clay sample is shown in Figure 2.

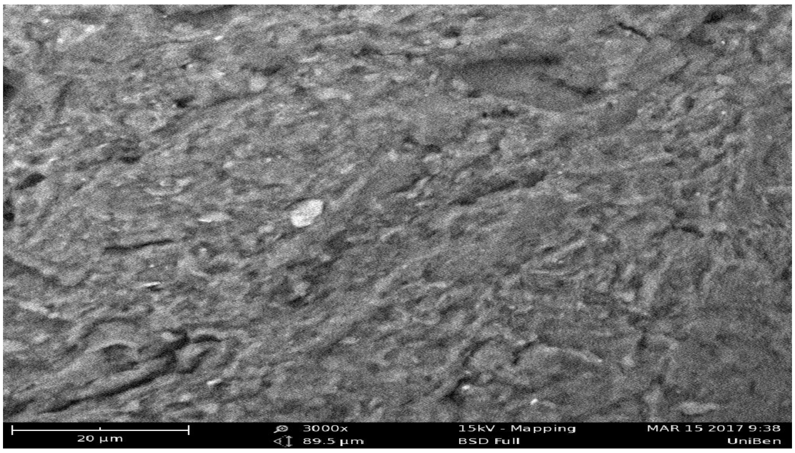

Figure 2: SEM Micrographs of Oza-Nogogo Clay (Magnification x3000)

Figure 2 is the SEM micrograph of the clay sample at magnification of x3000. On examination of SEM micrographs, the clay particles seem to consist of very small platelets which indicate that the clay sample is made up of very fine particles. Well crystalline kaolinite particles of varying sizes that are rough edged are also observed. Some kaolinite particles are below 1µm size. The coarse region is an indication of the presence of some particles of quartz (Henderson *et al.*, 1970). Rounded nature of quartz particles and spherical grains indicate recycled nature and maturity of sediment accumulation. Quartz particle at this high magnification shows spongy surface and microcracks. The spongy quartz might have resulted due to the soil environment at Oza-Nogogo. Here the quartz-kaolinite contacts are more sharply defined and more parallel, indicating a closer control on corrosion of the quartz by the later-forming kaolinite crystallites themselves.
Hot and humid weathering environment also favour weathering of kaolin group minerals. Formation of an intermediate between micas and clay minerals are also possible. Coarse fraction also shows patches, rich in Ti and Fe bearing minerals and containing traces of Mn indicated by XRF, which coats quartz and kaolinite particles as impurities are also observed.
The results of pore size characterization and the CEC of the sample is shown in Table 2.

Table 2: Pore size characterization and CEC of Oza-Nogogo clay sample

Parameter	Value
Specific surface area (m^2/g)	7.846
Average pore diameter (nm)	28.642
CEC (meq/100g)	6.8

As observed, the specific surface area for the clay is 7.846 m^2/g; this is below the range of 10 to 20 m^2/g established for kaolinite (Bohn *et al.,* 1985) and far less than the value for standard adsorbents and catalysts. This may be due to the high quartz content of the clay. The CEC of a clay is defined as the milli-eequivalents of cation that can be exchanged under standard conditions per100 gm of clay and it is expressed as a number. It is a measure of the capacity of soil to hold on to cations (Neal and Worral, 1977). The CEC was also observed to be low (6.8 meq/100 g). This is however within the range of values of 3 and 15 meq/100 g for Kaolinite given elsewhere (Grim, 1968). With a pore diameter of 28.642nm, Oza-Nogogo clay is mesoporous based on the classification of pore size as recommended by International Unit of Pure and Applied Chemistry (IUPAC) (Sing, 1985). Hence this clay is not suitable for use as an adsorbent.

4. Conclusion

The combination of the three characterization techniques adopted in this research work (XRD, XRF & SEM) showed consistency in the revelation of the quantities of different constituents of the clay sample. The mineralogical analyses (XRD) showed that the clay samples are predominantly kaolinitic with some quantities of quartz.
The chemical analysis (XRF) showed the compounds present with its related concentration unit. From the result obtained it is observed that the sample contains high amount of silica by mass of 51.3% followed by Alumina 40%. All other minerals present are in negligible proportion. The major elemental contents of clay samples detected in the study were Si, Al, Fe, Ti and K.
Due to the high kaolinite content, Oza-Nogogo clay can serve as basic raw materials for pharmaceutical, drugs, paints, paper and ceramic industries only after reducing the level of quartz in it to an acceptable level. However, this in its present form will be suitable for use as a refractory material.

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