Physic-Chemical Characterization of 'NGWO' White Clay for Industrial Use

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Abstract– **Ngwo White clay from Udi in Enugu State of Nigeria was analyzed mineralogically and physic–chemically. The results of the chemical analysis are as follows: SiO_2:53.2%, Al_20_3:5.906%, K_2O:0.121%, Na_2O:1.4175%, Fe_2O_3:0.444%, CaO: 0.0061%, PdO: 0.0049%. The results of the physical analysis are as follows: 8%moisture, 1%Ash, 1.67g/ml bulk density, 5.63P[H] and 4%Loss on Ignition. The ratio of SiO_2:Al_2O_3 greater than one showed that it can be used in the synthesis of zeolite. The FTIR analysis for the mineral contents confirmed the presence of kaolinite, illite and calcite as the major minerals and chlorite, montmorillonite, quartz, and smectite as the minor minerals in the sample. Sodium montmorillonite found in the clay makes it suitable to be used in oil industry as a drilling mud.**

Keywords– **FTIR, Clay, Minerals, LOI and Zeolite**

I. INTRODUCTION

Clay is a naturally occurring material composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and will harden when dried or fired [1]. They have varying chemical composition depending on both the physical and chemical changes in the environment where clay deposits are found [2]. Natural clay minerals are well known and familiar to mankind from the earliest days of civilization. Because of their low cost, abundance in most continents of the world, high sorption potential for ion exchange, clay materials are strong candidates as adsorbents [3].

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.

Clay is composed mainly of silica, alumina and water, frequently with appreciable quantities of iron, alkalies and alkali earths [4]. Two structural units are involved in the atomic lattices of most clay minerals. One unit consists of closely packed oxygen and hydroxyls in which aluminum, iron and magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygen or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a hexagonal network that is repeated indefinitely to form a sheet of composition, $Si_4O_6(OH)_4$ [4].

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 [5].

The aim of this research work is to determine the physical and chemical composition of clay sample from Ngwo in Enugu state of Nigeria and to ascertain its suitability for Industrial purposes.

II. EXPERIMENTAL METHODS

A. Materials

The clay sample used was obtained from Ngwo in Udi L.G.A of Enugu State of Nigeria. The sample was used as received without any modification. The samples after removing the contaminant, were ground to increase the surface area. Standard method according to [5] was used in the physical characterization of the clay samples. Prior to chemical characterization by Atomic absorption spectrophotometer, the sample was digested.

B. Digestion of clay samples

0.1g of the sample was weighed into a Teflon crucible and moistened with Aqua regia (mixture of HCL and $HN0_3$ in the ratio of 3:1 by volume). 15ml of hydrofloric acid was added and the mixture covered and heated in a fumed chamber at 100^0C until the solution became clear. The solution was allowed to cool and then transferred into a 250ml volumetric flask and was made up to 250ml mark with distilled water. Atomic absorption spectrophotometer was used to analyze the minerals.

C. Loss on ignition

0.5g of the clay sample was weighed in a dried platinum crucible and was put into a furnace at 600^0C for 3 hours. It was then cooled in a dessicator and weighed. Weight lost is the loss due to ignition, which is given as follows:

$$LOI = \frac{\text{Weight Loss X } 100}{\text{Weight of sample used}} \quad (1)$$

D. Determination of the oxides using atomic absorption spectrophotometer (AAS)

After the digestion of the samples, the recommended standard method was used for the elemental quantitative analysis. The minerals were analyzed using AAS (Win Lab 32). Air acetylene flame was used for the analysis of each metal except for S_i and Al which required nitrous oxide flame instead at flame temperature of 2250^0C. The machine automatically controlled the ratio of the fuel and oxidant gas. And deionized water was used as the blank. The result obtained in PPM was converted to percentage oxide.

E. Fourier transformed infrared spectroscopy (FTIR)

This was used for the identification of clay minerals and poorly crystalline mineral phases, and also for identification of possible adsorbed elements or functional groups.
The samples were prepared by applying the disc techniques (mixing 1mg clay sample with 200mg kBr) and was put in a mold. These intimate mixtures were then pressed at very high pressure (10 tons per cm^2) to obtain the transparent disc, which were then placed in the sample compartment. The spectra were recorded over the range of 4000–350cm^{-1}.

III. RESULTS AND DISCUSSION

A. Physical Characterization

Six parameters were determined: Moisture content, Ash content, Bulk density, pH, Iodine number and Loss-on-Ignition. The results of the physical characterization are shown in the Table 1.

Table 1: Physical characterization

S/N	PARAMETER	UNIT	Clay
1	Moisture	%	8
2	Ash	%	1
3	Bulk Density	g/ml	1.67
4	pH	-	5.63
5	LOI	%	4

From the result, it had high percentage of moisture content and plasticity and strength of clays depend on the amount of water present. Between 1.2 to 14.26% moisture content, the clay will form an aggregate to develop strength. The high moisture content makes the clay sample suitable for molding and when combined with sodium montmoilonite, it can be used in the determination of the refractory ability of bricks.

The high bulk density observed in clay makes it suitable for bricks production. The loss-in-ignition of the sample was low which was lower than 18% specified for the upper limit for refractory clays.

The non combustible volatile matter content of the clay sample was low as evidenced on the low ash content of 1% obtained.

B. Chemical characterization

The result of the oxide analysis is shown on Table 2.

Table 2: Oxide Analysis

S/No	Parameter	Clay (% oxide)
1	Potassium	0.121
2	Calcium	0.0061
3	Silicate	53.2
4	Lead	0.0049
5	Iron	0.444
6	Sodium	1.4175
7	Aluminum	5.906

From the result above, the low content of alumina makes it not suitable to be used as high melting clay.

Moreover, with the ratio of Silicate to Alumina (S_iO_2/Al_2O_3) in the clay been greater than one, makes it suitable for Zeolite synthesis according to [7]. Zeolite is a catalyst used in the catalytic cracking of petroleum. The large amount of silicate present in the clay is associated with crystalline phase quartz which is combined to Alumina in the aluminosilicate structure as suggested by [8]. When this is present in the clay, it decreases the intensity of the clay. When silica content of clay is high, their source of silica can be used for the production of floor tiles. According to [9], the high content of S_iO_2 and Al_2O_3 and low content of Cao present on the clay suggested that the clay is kaolinite in nature.

Also, the result showed that the ratio of Na_2O to CaO is greater than one which indicated the presence of swelling bentonites.

C. FTIR analysis

The results were obtained from a graph of percentage transmittance (T%) against wavelength (cm^{-1}) within the range of 4000 – 350 cm^{-1} in the fig 1 below. The observed absorption wave numbers from the peaks were tabulated with their corresponding minerals as shown on the table 3 below. The result showed that the clay sample had minerals at the lower wavelength below 1000 cm^{-1}. It also showed that the various stretching and deformation is caused by the presence of S_i and Al, at their different "OH" variance.

In the sample, the bands near 3445.71cm^{-1}, 3434cm^{-1}, and 3400cm^{-1} observed more or less clearly in the spectra of illite. This is because of variable (OH) stretching which it has in its wavelength. However, the (H – O – H) stretching also gives a clear picture of illite mineral. In the deformation site of the wavelength at the region of 475 – 466.83 cm^{-1} indicates the presence of illite.

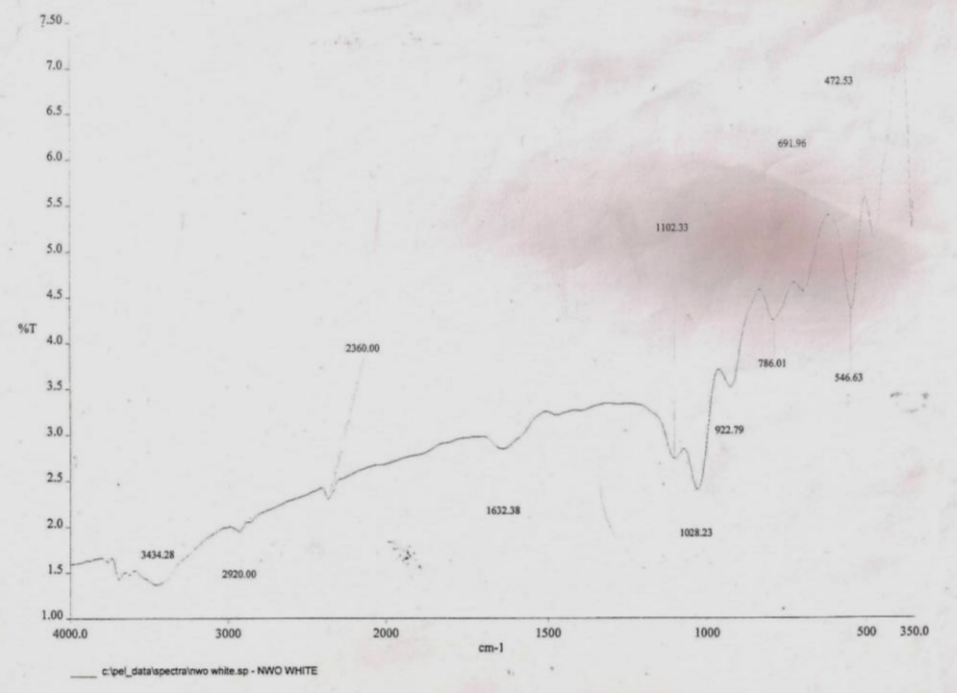

Figure 1: FTIR spectrum of the clay sample

Table 3: The observed adsorption wave numbers and corresponding minerals from FTIR spectra

S/No	Assignment	Wavelength cm^{-1}	Minerals
1	V (OH) – Stretching	3434.28	Illite
2	Si – OH Stretching	2920.00	Calcite
3	Si – OH Stretching	2360.00	Kaolinite
4	H – O – H Stretching	1632.38	Illite
5	V (Si – O) Normal to the Plane Stretching	1102.33	Chlorite
6	V (Si – O) Planar Stretching	1028.23	Montmorillonite
7	(Al – Al – OH) deformation	922.79	Smectite
8	Si – O Stretching	786.01	Quartz
9	Si – O Stretching	691.96	Calcite
10	Si – O – Al deformation	546.63	Kaolinite
11	Si – O – Si deformation	472.53	Illite

The Si – O stretching and bending as well as the OH bending absorption in the range of 1300 – 400cm^{-1} tells us more about the kaolinite mineral present in the clay.

Calcite is the most common carbonate minerals in clay and from the existence of a peak in the range 1410 – 1438cm^{-1}, 2920cm^{-1} and 691.96 having Si – OH stretching indicates the presence of calcite. Calcite was observed in the Si – OH stretching at 2920.00cm^{-1}.

The presence of quartz can also be observed in the region of 790.9cm^{-1}, 693.4cm^{-1}, 538.8cm^{-1} and 468.9cm^{-1}. The appearance of V (Si – O – Si) and δ (Si – O) bands also support the presence of quartzl. The mani Si – O band is present in the IR spectra of both di – and trioctahedral smectites.

Structural OH bending vibration for clay minerals is located at 950 – 600 cm^{-1}. AlAl OH bending band can be assigned to smectite and / or kaolin minerals at 919 – 913cm^{-1} and $AlF_e.^{3+}OH$ bending band for smectite locates at 890 – 870cm^{-1}. The position of this band is decreasing with increasing Fe content. A+ 860 – 840cm^{-1} are AlMgOH bending bands for smectite. The band at approximately 920cm^{-1} is least affected by the type of exchangeable cation, but most affected by water content.

Beidellite and montmorillonite had similar spectra, but beidellite has characteristic Al – O out of plane and Al – O – Si in – plane bands at 818 cm^{-1} and 770cm^{-1}. The same Al – O and Al – O – So vibration are also characteristic for illites, but they locate at 825cm^{-1} and 750cm^{-1}. It showed much content in the sodium montmorillonite as observed in Si – O stretching at 1381.60cm^{-1}. The presence of this mineral in the clay is because the clay contains some rock formation properties of the cations.

IV. CONCLUSION

Clay from Ngwo in Udi L.G.A of Enugu State of Nigeria was characterized physiochemically. The results obtained showed the possibility of using the clay sample for different purposes. The FTIR analysis, confirmed the presence of kaolinite, illite, montmorillonite, calcite, chlorite, smectite, and Quartz.

- The low value of loss-in-ignition showed that it can be regarded as refractory clays.
- The kaolinite which was the major mineral content makes it suitable in the paper production, pharmaceutical, in ceramics production and bricks production.
- It can be good in making of animal dung due to the presence of smeatite and illite minerals present in the clay.
- Sodium montmorillonite found in the clay makes it suitable to be used in oil industry as a drilling mud. It can equally be used in the synthesis of zeolite which is a catalyst in cracking of petroleum.

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