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Evaluation of Physico-Chemical and Mineralogical Properties of Okigwe Clay Mineral Deposit in South-East Nigeria

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ABSTRACT

This study characterized untreated and acid treated Okigwe clay, Nigeria, to assess its physicochemical and mineralogical properties and potential industrial applications. The samples were treated with 1.0 M H_2SO_4, and their functional groups, mineral phases, elemental composition, and oxide composition were examined using Fourier-transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD), energy dispersive X-ray fluorescence (EDXRF), and chemical assay using atomic absorption spectroscopy and flame photometry, as applicable. FTIR analysis indicated O–H, C–H, C=C, and C–O related bands in the samples, with differences between the untreated and treated clay. XRD analysis identified quartz, albite, kaolinite, orthoclase, muscovite, illite, and vermiculite in the treated sample; quartz, illite, muscovite, albite, dickite, vermiculite, and orthoclase were reported in the untreated sample. EDXRF results showed oxygen, aluminium, and silicon as major detected elements, alongside iron and several minor or trace elements. The oxide assay of the treated sample reported 49.73% SiO_2, 23.51% Al_2O_3, 19.22% Fe_2O_3, and 3.06% TiO_2. These findings indicate that the deposit is predominantly silicate/aluminosilicate in character. The quartz-rich composition suggests possible relevance to ceramic, construction, and related mineral based applications.

Key words: Okigwe clay, mineralogical compositions, elemental composition, chemical assay

INTRODUCTION

Clay is a readily available basic material. However, the geographic distribution and spread of some high-grade clay deposits are restricted. Bentonites are clays created by the modification of volcanic ash that are mostly composed of the clay mineral montmorillonite. The petroleum industry employs them as drilling muds and catalysts, foundries use them as bonding clays,

taconite pellets use them as bonding agents, and several industries use them as adsorbents [1]. Generally, clay minerals are widely utilized in various industries, including ceramics, construction, pharmaceuticals, and petroleum refining. Because characterizing clay deposits aids in assessing their suitability for particular uses, their utilization is primarily dependent on their properties. As such, this study focuses on the characterization of Okigwe clay, a naturally occurring clay deposit found in Okigwe, southeastern Nigeria. Okigwe is known for its rich clay deposits, which have potential applications in different industries. However, for effective utilization, it is essential to understand the physicochemical and mineralogical of the clay. Clay minerals share some common characteristics and differences as well. The presence and absence of any property influence the desired effect; thus, proper characterization should be made to know the nature of the clay deposits of interests. These properties involve: Mineral composition, structural properties, chemical properties and physical properties.

With the increasing importance of environmental protection and the development of petroleum exploration research, clay minerals are important additives in industrial processes. The characterization of clay materials is essential for understanding their composition, properties, and industrial applications. Various studies have been conducted on clay deposits across different regions, focusing on their mineralogical, chemical, physical, and thermal properties. Local bentonite clay deposits in Nigeria are estimated by the Nigerian Mining Corporation and the Raw Materials Research Development Council (RMRDC) to be over 700 million metric tons. The majority of these deposits are located in Afuze, Edo State, Mid-Western Nigeria, which contains roughly 70–80 million metric tons of bentonite clay. Significant amounts of bentonite clays can be found in the South-East Nigerian Abakaliki Formation [2]. Additionally, bentonite clay deposits have been documented in the states of Abia, Anambra, and Lagos. According to Omonigho and Olugbenga [3], these bentonite clay deposits are found in nearly every state in the Federation. Additionally, a research parastatal of the Federal Government of Nigeria, has taken the lead in estimating the country's bentonite resource. The location and estimated values of Nigeria's proven bentonite clay reserves were presented by RMRDC [4]. The characteristics and handling of some Nigerian clays were investigated by Akpoturi et al [5].

The study involved the collection and laboratory analysis of clay samples from ten towns in seven local government areas of Rivers and Bayelsa States: Oboburu and Omoku (ALGA), Kono (BOLGA), Bakana (DELGA), Ndele (KELGA), Borokiri (PHALGA), Akaranbiri (SALGA), Agidama, Igbainwari, and Sabagrei (YELGA). These samples'

characteristics fell short of API (American Petroleum Institute) requirements, making them unsuitable for drilling mud preparation in their home states. Ibrahim et al [6] investigated the use of non-fermentable polymers to improve Gambe clay for the formulation of drilling mud. Three carboxy methyl cellulose (CMC) with varying average molecular weights were the unfermentable polymers used in the attempt to improve Gambe clay. The clay came from Gambe town in Adamawa State, Nigeria, and was enhanced with 6 wt% Na_2CO_3 and beneficiated to remove quartz.

X-ray diffraction and X-ray fluorescence tests were employed to establish the mineral and oxide content of the clay, which was then utilized to create the drilling fluid. The drilling fluid properties of suspected bentonite clay were examined by Onyemaleze et al [7]. Their investigation sought to identify compared the sand content, pH, mud weight, and rheological characteristics of the presumed bentonite clay from Ejeme-aniogor in the Aniocha South Local Government Area in Delta State. The Fann-VG meter was used to prepare low, mid, and high viscous mud after the clay was collected from a depth of approximately three feet, dried, crushed, and sieved using a 212-mesh size. The various parameters were then examined. From the review, it is evident that clay deposits vary significantly in composition and properties, which influences their industrial applications.

Studies on Nigerian clays have shown high kaolinite and quartz content, making them suitable for ceramics, construction, and refractory materials. However, further beneficiation and processing may be necessary to enhance their properties for advanced industrial use. This review provides a strong foundation for the characterization of Okigwe clay, enabling comparisons with previous studies and determining its potential applications in various industries. The aim of this study is to use suitable instrumental approaches to identify the functional groups, chemical composition, and elemental composition of Okigwe clay.

MATERIALS AND METHODS

Materials

Samples of native clay were obtained from Okigwe in Imo State. A graduated measuring cylinder, heating mantle, x-ray fluorescence spectroscopy (Supreme 8000, Oxford Instrument), x-ray defractometer, Fourier transform infrared spectrometer, water bath, electronic weighing balance, grinder, sieve, mixer, viscometer, drilling mud balance, pH meter, and stop watch are among the tools and equipment used in this work.

Treatment of the clay

Okigwe clay sample was processed through the following treatment steps: crushing of the raw clay, screening, slurry (with addition of water), dewatering, drying, crushing, milling/pulverizing and packaging. The standard method used by Nlemedim et al [8] 1.0 Molar solution of H_2SO_4 was used for the treatment of the clay through leaching process. The powdered clay was sieved into very fine particles (0.05 mm). 600 grams of the fine particles was obtained through the screening process. A 250 ml flat-bottom flask was then filled with 40 grams of the fine clay sample and 180 ml of 1.0 Molar solution of H_2SO_4. The mixture was heated to 95 degrees Celsius for one hour using a magnetic stirrer. After that, the liquid was tested using a blue litmus paper and repeatedly rinsed with distilled water to entirely remove the acid. The resulting slurry was filtered, dried and ground into powder form. The procedure above was repeated to get a mass of 520 grams of the treated clay samples.

Determination of the functional groups of the samples

The samples' functional groups were identified using an FTIR (Fourier transform infrared) spectrophotometer (Cary 630, Agilent Technologies USA). The spectrophotometer concurrently gathered high spectral resolution data across a broad-spectrum range during the FTIR experimental process. The raw data was transformed into an actual spectrum using a mathematical procedure called the Fourier transform. Appropriate functional groups were identified by analyzing the various FTIR produced peaks [9]

Determination of the mineralogical compositions of the clay samples by X-ray Diffractometer

Each clay sample was produced using the sample preparation block, compressed in the flat sample holder to form a flat, and then placed on the XRD cabinet's sample stage. The Theta-Theta settings of the reflection-transmission spinner stage were used to evaluate the sample. With a two-theta step of 0.026261 at 8.67 seconds per step, the two-theta began at 4 degrees and ended at 75 degrees. The tension was 45VA and the tube current was 40 mA.

The Gonio Scan was utilized along with a 5 mm width mask and a programmable divergent slit. As the sample and detector rotated through their respective angles, the intensity of diffracted X-rays was continually recorded. When the mineral has lattice planes with d-spacings suitable to diffract X-rays at that value of θ, the intensity peaks. Even though each peak is made up of two distinct reflections ($K\alpha_1$ and $K\alpha_2$), the peak positions overlap at small values of 2θ, with $K\alpha_2$ appearing as a hump on the side of $K\alpha 1$. Higher values of θ result in

greater separation. These linked peaks are usually regarded as a single peak. The center of the peak at 80% peak height was used to calculate the diffraction peak's 2λ position. Peak positions at 2θ and X-ray counts (intensity) were used to display the results.

Energy-dispersive X-ray fluorescence (EDXRF) spectroscopy

EDXRF spectroscopy was used to quantitatively analyse the clay sample. The elemental and oxide contents of the corresponding samples were determined by chemical analysis [10]. The sample was placed inside the spectrometer's sample holder and exposed to high-energy X-rays from the controlled X-ray tube. The spectrum graph of X-ray intensity as a function of the peaks displayed XRF peaks with different intensities. Additionally, the samples' chemical compositions were represented as a percentage of each component's overall concentration.

Chemical assay

The approach outlined [11,12] was used to identify the chemical compositions of the mineral deposit on dry samples. This was accomplished by heating a portion of the test sample to between 105 °C and 110 °C for an hour in order to eliminate moisture, cooling it in a desiccator, and then storing it in a sealed container. Aqua regia was used to digest the test sample (1.0 gram), which was weighed into a beaker. Atomic absorption spectrophotometer (Bulk Scientific 210 VGP) and flame photometer (model Sherwood 410) were used to identify the chemical components of the samples, and the conventional method as described was used to calculate the total silica content [13].

RESULTS AND DISCUSSION

Functional groups of the samples

The treated and untreated clay samples' functional groups are shown in Tables 1 and 2. They have polar and heteroatoms of oxygen and nitrogen. There is also presence of single and double bonds. The functional groups of the untreated Okigwe clay sample in Table 1 revealed O-H, C=O, C-H, C=C, N-H, C-H and C-O as the prevailing functional groups. Alkoxy C-O, C=C, C-H and O-H were revealed as the main functional groups of the treated Okigwe clay (Table 2). It shows that the treated clay can compete favourably with the bentonite [14].

Mineralogical compositions of the clay samples as determined by XRD

Mineralogical compositions of Okigwe clay sample as determined by XRD are shown in Figure 1. which showed the result untreated Okigwe clay. The result revealed that the untreated clay contains 55.6% quartz, 28.7% Illite, 5.4% muscovite, 1% albite, 0.1% dickite, 9.1% vermiculite

and 0.01% orthoclase. Figure 2 showed the result of XRD of treated Okigwe clay. It revealed 57% quartz, 26% albite, 14% kaolinite, 0.5% orthoclase, 2% muscovite, 0.6% Illite, 0.2% vermiculite. Comparing the result of the treated and untreated clay, the treatment enhanced the properties of the clay [15].

Table 1: Functional Groups of Untreated Okigwe Clay

Peaks	Bond	Compound Type	Mode	Intensity
3763.4	O-H	Alcohol	Stretch	Strong
3701.2	O-H	Alcohol	Stretch	Strong
3652.8	O-H	Alcohol	Stretch	Strong
2892.4	C=O	carboxylic acid, alcohols & ketone	Stretch	Strong
2072.4	C-H		Stretch	Strong
1982.9	C=C	Alkene	Stretch	Strong
1640.0	N-H	Amide	Bend	Strong
775.3	C-H	meta disubstituted aromatic	Bend	Strong
693.3	C-H	monosubstituted aromatic	Bend	strong to medium
913.2	C-H	monosubstituted alkene	Bend	Strong
1032.5	Alkoxy C-O	Esters	Stretch	Medium
1006.4	Alkoxy C-O	Esters	Stretch	Medium

Table 2: Functional Groups of Treated Okigwe Clay

PEAKS	BOND	COMPOUND TYPE	MODE	INTENSITY
3652.8	O-H	Alcohol	Stretch	Strong
3693.8	O-H	Alcohol	Stretch	Strong
3623.0	O-H	Alcohol	Stretch	Strong
1990.4	C-C	Alkene	Stretch	Strong
1114.5	Alkoxy C-O	Esters	Stretch	Medium
793.9	C=C	trisubstituted alkene	Bend	strong to medium
752.9	C-H	monosubstitued aromatic	Bend	Strong
678.4	C-H	cis disubstituted alkene	Bend	Strong
909.5	C-H	monosubstituted alkene	Bend	Strong
998.9	C-H	monosubstituted alkene	Bend	Strong

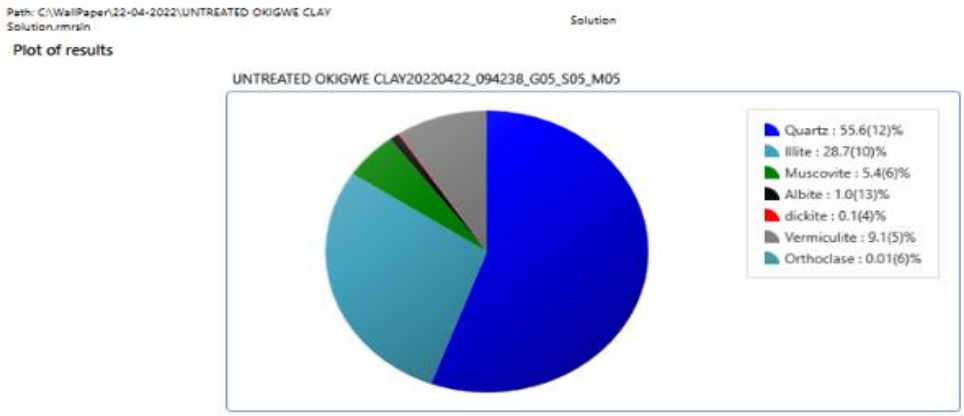

Figure 1: Composition of untreated Okigwe clay

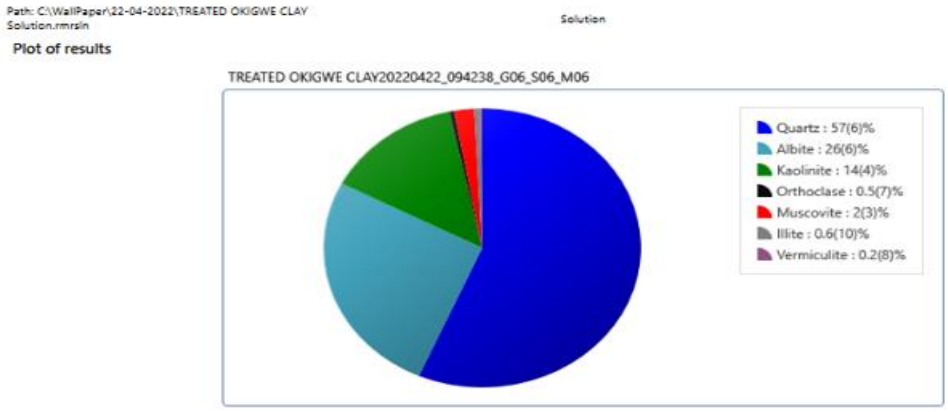

Figure 2: Composition of treated Okigwe clay

Elemental composition of the clay samples as determined by XRF

Table 3 displays the elemental composition of the Okigwe clay samples as measured by XRF. From the XRF investigation, the elements O, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Cu, Zn, Rb, Sr, Zr, Nb, Ag, Ba, and Ta are prevalent in all of the samples. Oxygen, aluminum, silicon, and calcium have high values in the clay samples, whereas other trace elements have low values. The presence of silicate compounds typical of kaolinitic clay is indicated by the high values of silicon and oxygen [16].

Oxide assay

Table 4 displays the chemical analysis results for the treated and untreated clay samples in terms of oxide composition. The results revealed the predominance of silica (49.73%), alumina (23.51%), iron oxide (19.22%), and titanium oxide (3.06%). This indicates that the deposit consists largely of aluminosilicate minerals.

Table 3: XRF results (Elemental compositions)

S/N	Element	Untreated Okigwe	Treated Okigwe
1	O	47.391	45.893
2	Mg	-	1.211
3	Al	13.643	12.444
4	Si	25.924	23.245
5	P	0.058	0.073
6	S	0.063	0.056
7	Cl	0.603	0.532
8	K	0.854	0.588
9	Ca	0.069	0.126
10	Ti	2.245	1.834
11	V	0.097	0.092
12	Cr	0.025	0.019
13	Mn	0.024	0.027
14	Fe	8.435	13.442
15	Co	0.047	0.069
16	Ni	0.002	-
17	Cu	0.037	0.038
18	Zn	0.005	0.005
19	Rb	0.008	0.012
20	Sr	0.036	0.030
21	Zr	0.167	0.125
22	Nb	0.022	0.015
23	Mo	0.005	0.005
24	Ag	0.011	0.020
25	Ba	0.212	0.092
26	Ta	0.018	0.008
27	W	0.001	0.001

Table 4: Result of Oxide Assay

Components	% in untreated Okigwe clay	% in treated Okigwe clay
SiO_2	55.458	49.727
V_2O_5	0.172	0.164
Cr_2O_3	0.037	0.027
Fe_2O_3	12.059	19.218
MgO	1.029	2.008
Al_2O_3	0.022	23.512
TiO_2	0.006	3.060
BaO	25.778	0.102
Cl	0.603	0.532

CONCLUSION

The predominant functional groups of Okigwe clay samples include O-H stretch, C-H stretch, C=C stretch, C-O stretch, and C-H bend. Treated Okigwe clay has new absorption peaks for alkoxy C-O stretch, C=C bend, C-C stretch and C-H bend. The intensity of the treated clay was enhanced which may be attributed to the removal of impurities by the treatment process. The clay samples contain polar and heteroatoms of O and double bonds. The predominant minerals as reviewed by the mineralogical composition (XRD) are quartz, albite, kaolinite, orthoclase, muscovite, Elite and vermiculite. These minerals show that the clay has a good stability, strength, durability, thermal resistance and chemical resistance. From the XRF analysis, O, Al, Si, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Co, Cu, Zn, Rb, Sr, Zr, Nb, Ag, Ba, Ta, and W are prominent in both treated and untreated samples. The clay samples exhibit low values for various trace elements and high values for oxygen, aluminium, silicon, and calcium. The presence of silicate compounds, typical of kaolinitic clay, is indicated by the high values of silicon and oxygen, and can be exploited for a variety of industrial uses,

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