

Industrial Suitability of Imiegba Clay Deposits in the Southern Anambra Basin, Nigeria: A Geochemical, Mineralogical, and Geotechnical Approach

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Abstract- Clay soil samples from the area “Imiegba” In Etsako East local Government area of Edo State, Benin were analyzed for their geochemical composition, mineralogical and geotechnical characteristics, x-ray diffraction analysis and index property tests were carried out to determine elemental composition, mineralogical nature and geotechnical attributes of the clay. The XRD reveals that Quartz (SiO₂) is the most abundant with the average mean of 48.10% and ranged between 42.83 and 55.78%. Mineralogical analyses reveal kaolinite as the dominant clay mineral with quartz as the dominant non-clay mineral. The geotechnical index test shows that particle size distribution of the studied clay is a fine-grained with percentage fine fraction of 99%. The clay is generally light grey to brownish grey in color in hand specimen and turns brownish on firing. Other physical properties exhibited are liquid limit (55.8%), plasticity index (36.9%), and linear shrinkage (11.7%). They are essentially fine grained (> 70%). The study reveals that they have unique property which can be used by various production industries for the production of quality glaze, pottery, ceramic wears, paints and fillers and can also be beneficated to suit other industrial needs.

I. INTRODUCTION

There is a demand to speed up the exploration and exploitation activities of the different solid mineral deposits in Nigeria for sustainable diversified economy of the country. This will improve industrial techniques and technological advancement needed to take care of the increasing population of our people. The present monolithic petroleum driven economy is not the best giving to the unstable and occasionally decreasing oil revenue coupled by international politics of trade. This study will hopefully lead to the

unveiling of new mineral deposits which will form an economic basis for future industrial diversification of southern Nigeria. The nature of clay and its composition determine not only its quality and commercial value but also, to a large extent, its engineering behaviour. Among the characteristics of clays that influence their engineering performance are clay mineral composition, physical properties such as particle size distribution, plasticity, shrinkage, non-clay mineral composition, amorphous components (e.g organic matter, content) and geologic history (Grim, 1968). Deposits of clay raw material are widely distributed in Nigeria. In order to determine the financial gain of clay materials from a particular deposit, it is of paramount to examine the morphology, micro structure, determine the mineralogical composition in such clay deposit.

This study addresses the geochemical, mineralogical and geotechnical character of two clay deposits in the southern Niger Delta in view of their industrial application and utilization. Similar studies have been out by many researchers. This work attempts to audit the compositional and basic geotechnical attributes of two clay deposits at Imiegba In Etsako East local Government area of Edo State,, Nigeria, with a view to establishing their industrial relevance, as a contribution to the nation’s economic development.

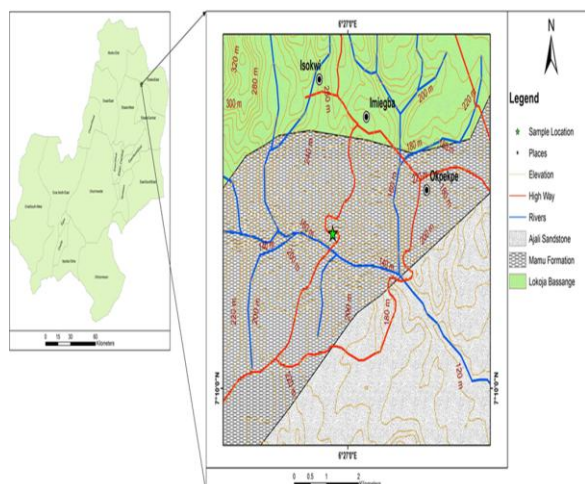


Fig 1.0 Geological Map of the study area.

II. MATERIALS AND METHODS

Systematic sampling methods were used in collecting the sample deposit which were collected from freshly exposed surfaces in sealed polythene bags to prevent contamination and loss of moisture; they were coded as IM 1, IM 2, IM 3, IM 4, IM 5 and IM 6 respectively. Six samples were collected vertically of each beds from Top to bottom in an upward coarsening sequence, the bottom are made up of dark shale of fine grain texture, the dark shale is as a result of high organic content while the middle are brownish sandy shale, the brownish shale is as a result of oxidation, high content of iron while the top are coarse sandstone of medium grained and are white to yellow colored. The samples were air-dried, ground to powder with mortar and pestle and sieved with a laboratory sieve.

Samples are crushed by agate crusher. Clay mineral associations have been studied using X-ray diffraction. X-ray diagrams were obtained using a Phillips PW 1730 diffractometer with CuK α radiations and Ni filter. A tube voltage of 40 kV and a tube current of 25 mA were utilized. Three X-ray diagrams were performed, after air-drying, ethylene-glycol saturation and heating at 490 °C during 2 h. The identification of clay minerals was made according to the position of the (001) series of basal reflections on the three X-ray diagrams (Moore and Reynolds, 1989) and by comparison with the Powder Diffraction File (JCPDS, 1995). Quantitative mineralogical composition was obtained using the method of Fiori. C. et al. (1986). Chemical analyses were carried out by a Philips PW

1400 XRF spectrometer, operating with Rh tube at 30 kV and 60 mA.

III. MATERIAL AND METHODS

Soil sampling Four representative clay samples from Imiegba deposits were collected from good exposures and kept in a sealed polythene bags to prevent moisture loss and contamination. These samples were taken to laboratory for analysis.

Analytical methods

In clay characterization studies, several methodologies are applied to determine mineralogy, geochemical composition and geotechnical behaviour of the clay deposit. Some of these methodologies include: x-ray fluorescence analysis for the determination of the geochemical composition of the clay.

Mineralogical Analysis

A quantitative determination of the mineralogical properties of the clay samples using X-ray diffraction were carried out at Rolab Research and Diagnostic Laboratory Challenge, Ibadan. Powdered samples were pelletized and sieved to 0.074mm. These were later taken in an aluminium alloy grid (35mm x 50mm) on a flat glass plate and covered with a paper. Wearing hand gloves, the samples were compacted by gently pressing them with the hand. Each sample was run through the Rigaku D/Max-III C X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan quipped with an x-ray tube capable of producing a beam of monochromatic x-ray, a sample holder, inbuilt standards, Peak/width,goniometer and x-ray detector and set to produce diffractions at scanning rate of 20/min in the 2 to 500 at room temperature with a CuK α radiation set at 40kV and 20mA. The angles and intensities of diffractions for each mineral are recorded electronically using a detector. After the scan of the sample the x-ray intensity can be plotted against angle 2θ to produce a chart. The angle 2θ for each diffracted peak can be converted to d- spacing using bragg's equation. ($n\lambda=2d\sin\theta$). The diffraction data (d value and relative intensity) obtained was compared to that of the standard data of minerals established by Brown (1951), Carrol (1971) and the JCPDS (Joint Committee on Powder Diffraction Standard) the mineral powder diffraction file (1980), which

contained and includes the standard data of more than 3000 minerals.

Chemical Analysis

XRF is a rapid, relatively non-destructive process that produces chemical analysis of rocks, its purpose is to identify the elemental abundances of the samples i.e. Identification of both major and trace elements. The samples were analyzed for major element oxides (SiO₂, Al₂O₃, Fe₂O₃, MgO, CaO, Na₂O, K₂O and MnO) in percentage while Mn, Ni, Co, Cr, and Zn were analysed in part per million using a phillips PW1606 X-ray Fluorescence Spectrometer.

Geotechnical Tests

A quantitative determination of the geotechnical properties of clay samples was carried out in civil engineering soil laboratory, University of Benin, Benin City Edo State Nigeria. The various analyses carried out include the determination of moisture content, Atterberg limit (liquid limit, plastic limit and plasticity index) and specific gravity, natural moisture content and linear shrinkage. These laboratory analyses were carried out according to British Standard Methods of test for soils for civil engineering purposes. (BS 1377: part 2: 1990). The liquid limit, plastic limit and plasticity index of clays are also used extensively, to correlate with engineering behavior such as compressibility, permeability, compatibility, shrink swell and shear strength and to specify the end product materials.

Atterberg Limits

The following moisture conditions – liquid limit, plastic limit, along with shrinkage limit are referred as the “Atterberg Limits. It is also called consistency test. Depending on the water content of the soil, it may appear in four states: solid, semi-solid, plastic and liquid. In each state, the consistency and behavior of a soil is different and consequently so are its engineering properties. Thus, the boundary between each state can be based on a change in the soil’s behavior.

Liquid Limit Test Procedure

The liquid limit is define as the moisture content at which soil begins to behave as a liquid material and begins to flow. The liquid limit is determined in the lab as the moisture content at which the two sides of a groove formed in a sample come together and touch

for a distance ½ inch after 25 blows. Since it is very difficult to get this to occur exactly, the test is repeated until the groove closes ½ inch with over 25blows and under 25 blows. The results are plotted as blow count versus moisture content.

Determination Of Plasticity Index

The moisture contents determined for the liquid limit (LL) and plastic limit (PL) tests were used to compute the plasticity index, PL. the difference between the LL and PL gave the PL for each clay, to the nearest whole number. The plasticity index may be considered as a measure of the cohesion possessed by a soil and is a dimensionless number.

Plasticity Index = Liquid Limit – Plastic Limit

$$PI = LL - PL$$

Soil with medium to high plasticity index tends to be clay, those with lower plasticity index to be silt, and those with a plasticity of zero (0) i.e. non plastic tends to have little or no silt or clay.

Natural Moisture Content Determination

Moisture content is determined by loss on drying techniques and this method is used to determine the percentage of water in a sample by drying the sample to a constant weight. The natural water content also called the natural moisture content is the ratio of the weight of water to the weight of the solids in a given mass of soil. This ratio is usually expressed as percentage and this testing conforms to ASTM D2216-90. The natural moisture content will give an idea of the state of soil in the field.

Calculation

The moisture content of the sample is calculated using the following equation:

$$W\% = (w_2 - w_3) / (w_3 - w_1) \times 100$$

Where W% = Percentage of moisture in sample.

W1 = weight of can

W2 =weight of can + wet sample.

W3 = weight of can + dry sample.

Determination Of Linear Shrinkage

Linear shrinkage is the decrease in length of a soil sample when oven-dried, starting with a moisture content of the sample at the liquid limit.

IV. CALCULATION

Calculate the percentage linear shrinkage (LS) of the specimen.

$$LS \% = (L_s)/L \times 100$$

Where

L = Length of the mould (mm) or initial length of specimen

L_s = Longitudinal shrinkage of the samples (mm) or length of oven dried specimen.

V. RESULTS

CHEMICAL ANALYSIS OF THE CLAY SAMPLE (XRF)

The tables below presents the results of the chemical analysis showing the different oxides forms of the major elements contained in the clay samples, and are characterized by ten (10) elements. The results showing the major element oxide compositions and calculated Weathering Indices of the clay samples in the study area are as shown in Tables 4.1 and 4.2. Quartz (SiO₂) is the most abundant with the average mean of 48.10% and ranged between 42.83 and

55.78%, in samples. The tables also showed that the major oxides apart from SiO₂, include Al₂O₃ with average mean of 34.22% while TiO₂, MnO, MgO, CaO, Na₂O, K₂O, and N₂O₅ were present in the clays only in small amounts. Iron as Fe₂O₃ in the Imiegba clay sample is less than 2% while that of Laterite is greater than 2% the values of K₂O varied from 0.62 to 1.95% of the sample with an average mean of 1.39% and shows the present of illite. The low amount of MgO and CaO shows the absence of associated carbonate or dolomitization process in the study area. Table 4.2 present the result of chemical analysis showing the different trace elements contained in the clay samples and are expressed in part per million (ppm). This includes Cu, in the range of (27.26 – 28.36), Cr (100.30 – 115.2), Ni (30.55 – 32.55), Zn (209.4 – 211.46). The concentration amount of Cu and Ni are very low which will not imply any coloration.

Table 1.0: GEOCHEMICAL ANALYSIS RESULT

ELEMENTAL OXIDES	IM A %	IM B %	IM C %	MEAN	RANGE	LAS %
SiO ₂	42.83	45.70	55.78	48.10	42.83-55.78	52.97
Al ₂ O ₃	32.21	35.20	35.24	34.22	32.21-35.24	28.42
Fe ₂ O ₃	1.04	0.08	0.77	0.63	0.08-1.04	3.65
TiO ₂	0.06	0.05	0.05	0.05	0.05-0.06	1.14
CaO	0.14	0.11	0.04	0.09	0.04-0.11	1.85
P ₂ O ₅	0.06	0.01	-	0.04	0.01-0.06	-
K ₂ O	1.95	1.60	0.62	1.39	0.62-1.95	0.94
MgO	0.04	0.06	0.05	0.05	0.04-0.06	0.08
Na ₂ O	0.36	0.25	0.29	0.30	0.25-0.36	0.05
LOI	12.44	12.40	12.42	12.42	12.40-12.44	10.20
CIA	92.9	94.7	97.4	95	92.9-97.4	90.9
CIW	98.5	99.2	99.1	98.9	98.5-99.2	93.7
Al ₂ O ₃ /TiO ₂	53.7	70.4	70.4	64.83	53.7-70.4	24.9

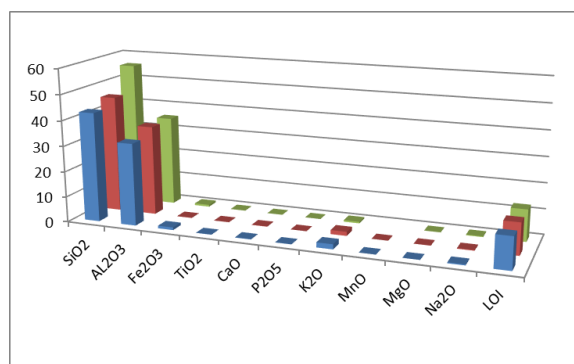


FIG 2.0 GEOCHEMICAL ANALYSIS OF THE CLAY

Table 2.0: CHEMICAL ANALYSIS OF THE CLAY SAMPLES (TRACE ELEMENT CONCENTRATION IN PPM)

TRACE ELEMENTS	IM A	IM B	IM C	LAS
Ba	614.86	662.63	670.62	662.67
Cu	27.26	28.26	28.33	28.36
Cr	115.2	114.20	112.40	100.30
Ni	30.55	31.45	32.55	31.65
Zn	211.46	210.44	209.4	210.39

Table 3: Chemical Composition of Ikere Kaolinite Compared with some Industrial Specifications

ELEMENTAL OXIDES	I	II	III	IV	V	VI	VI I
SiO ₂	48.10	49.88	47.00	45.00	48.67	67.50	51.70
Al ₂ O ₃	34.22	37.65	40.00	38.10	19.45	26.50	25-44
Fe ₂ O ₃	0.63	0.88	-	-	2.70	0.5-	0.5-
						1.2	2.4

TiO ₂	0.05	0.09	-	1.70	-	-	-
CaO	0.09	0.03	-	-	15.85	0.18-0.3	0.1-
P ₂ O ₅	0.04	-	-	-	-	-	-
K ₂ O	1.39	1.60	-	-	2.76	1.1-	-
MgO	0.05	0.13	-	-	8.50	0.1-	0.2-
Na ₂ O	0.30	0.21	-	-	2.76	1.2-	0.8-
						1.5	3.5

- I. Average Value for 5 sample Imiegba clay (This study)
- II. Agriculture (Huber, 1985)
- III. Pharmaceutical (Todd, 1975)
- IV. Textile (Keller, 1964)
- V. Brick clay (Murray, 1963)
- VI. Ceramic (Singer and Sonja, 1971)
- VII. Refractory Brick (Parker, 1967)

Mineralogical composition of clay deposits

The results obtained from the x-ray diffraction analysis are presented in Fig 2.0 – 5.0 and a summary of the x-ray results of mineralogy are shown in Table 3.0

Table 3.0: MINERALOGICAL COMPOSITION OF CLAY DEPOSIT

IM A	IM B	IM C	LAS
Kaolinite	Illite/Smectite	Kaolinite	Kaolinite
Quartz	Illite/Muscovite	Quartz	Quartz
Anatase	Quartz	Microcline	Microcline
	Albite	Anatase	Anatase
	Anatase		Hematite

IM – Imiegba clay

LAS – Laterite

The results of the mineralogical composition of the clay revealed that kaolinite, illite, smectite, albite,

anatase, were found to be the predominant clay mineral. Other minerals include quartz, microcline, muscovite, and accessory minerals such hematite.

Although quantitative analysis was not carried out, the diffractogram indicate that kaolinite were present in all the Imiegba clay A and C and also present in the Laterite deposits but only Imiegba clay B was found to contain variable amount of, illite/smectite and illite/muscovite which are absent in Imiegba clay deposit A and C also absent in laterite, this could be an indicative that the imiegba clay deposits were deposited at different time during their time of deposition.

Fig 3.0: X-RAY DIFFRACTOGRAM OF IM B

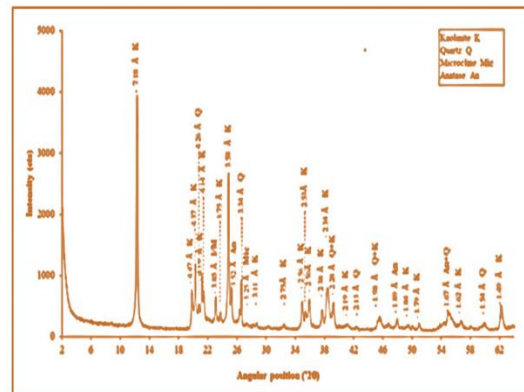


Fig 4.0: X-RAY DIFFRACTOGRAM OF IM C

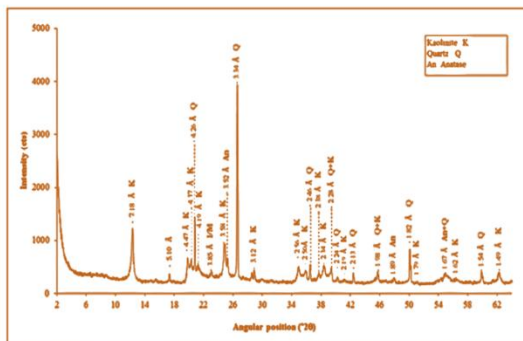


Fig 2.0: X-RAY DIFFRACTOGRAM OF IM A

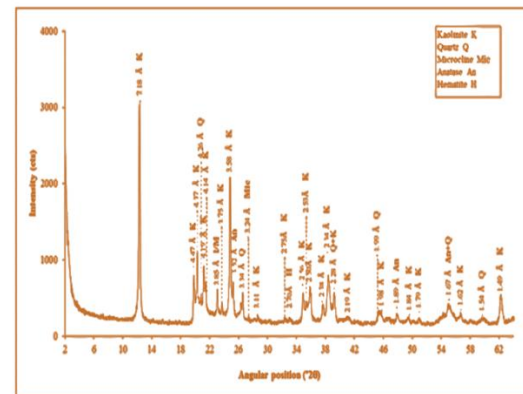
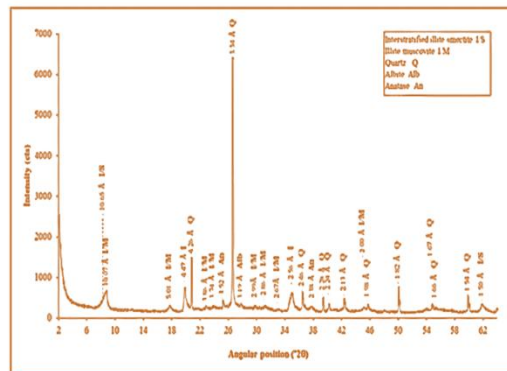


Fig 5.0: X-RAY DIFFRACTOGRAM OF LAS



GEOTECHNICAL CHARACTERISTICS

Table 4.0: Geotechnical characteristics of the studied clay samples

Clay sample	Particle size distribution		Fine %	Atterberg limit			Natural moisture content %	Specific Gravity	Swell test
	Clay	Silt %	Sand %	Liquid		Plasticity			

	%				limit %	Plastic limit%	index %	Linear shrinkage %			
IM A	63	36	1	99	56.0	20.19	35.81	11.40	0.10	2.26	100
IM B	65	34	1	99	60.0	20.62	39.38	11.86	0.11	2.06	30
IM C	58	41	1	99	51.50	15.70	35.80	11.84	0.42	2.12	37

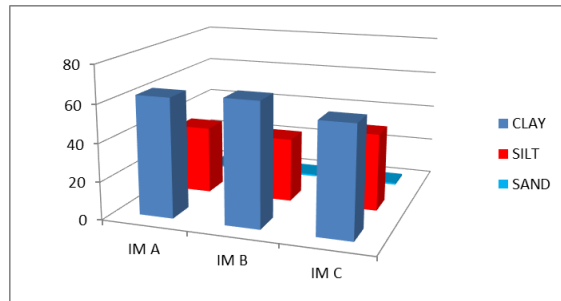


FIG 6.0 GRAIN SIZE

Table 5.0: Plasticity test results of studied samples compared with other clays and shale

%	*Average for study area	I	II	III	IV	V
Liquid limit	55.8 (51.50-60.0)	27 (26.2-29.5)	27 (25.2-29.0)	68 (67.0-68.0)	13.63 (11.10-14.9)	29 (28.0-30.0)
Plastic limit	18.84 (15.70-20.62)	54 (51.5-59.0)	66 (64.4-67.0)	146 (142-152)	29.37 (29.0-29.6)	53.5 (53.0-54.0)
Plasticity index	36.9 (35.80-39.38)	26 (23.0-29.5)	36 (33.8-38.0)	78 (74.0-85.0)	15.83 (14.1-18.4)	24.5 (24.0-25.0)
Linear shrinkage	11.7 (11.40-11.86)	2.49 (0.49-5.50)	3.49 (2.72-4.25)	8.50 (8.3-8.7)	-	6.10 (6.0-6.20)

*Average of 3 samples

- (I) Auchi shale (Emofurieta, 1994)
 (II) Gombe shale (Emofurieta, 1994)

- (III) Okada shale (Obrike et al, 2007)
 (IV) Udobu clay (Omada et al, 2007)
 (V) Ewekoro clay (Nton and Elueze 2005)

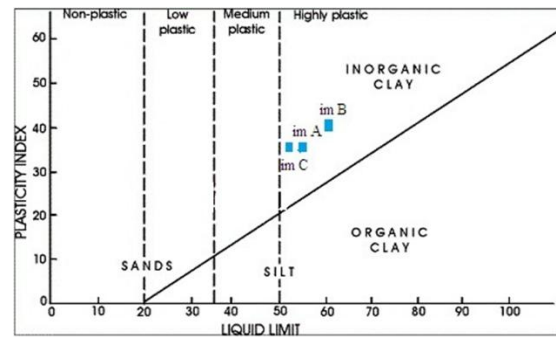


Figure 6.0: Character of Clay samples on a typical Casagrande plot. The plot indicates high plasticity for the samples considered in the analysis.

The result of the basic geotechnical characteristics of the studied clay samples are presented in table 4.0. The particle size distribution of the studied clay indicated that the clay is a fine-grained with percentage fine fraction of 99%. The clay is generally light grey to brownish grey in color in hand specimen and turns brownish on firing. Other physical properties exhibited are liquid limit (55.8%), plasticity index (36.9%), and linear shrinkage (11.7%). They are essentially fine grained (> 70%) and they plot in the region of inorganic clays of high compressibility on the Casagrande plasticity chart (Fig.6.0).

Table 5.0 compares the plasticity test results of the clay with other reference clay and shale samples. The studied and reference samples with the exception of the Udubu clays of the Kerri-Kerri Formation in the southern part of Udubu, are all highly plastic

(LL>50%). The average shrinkage value of the Imiegba clay (11.7%) is higher than average values for Auchu (2.49%), Gombe (3.49%), Ewekoro (6.10%) and Okada shale (8.50%). The high shrinkage could be attributed to the presence of smectite (Obrike et al, 2007).

V. RESULTS

Industrial assessment;

The geochemical, mineralogical, and physical characteristics constitute crucial parameters in the assessment of the suitability of clays as industrial raw materials. Evaluation of the industrial utilization of the clays is based on their geotechnical and geochemical characteristics and comparison with chemical specification of some industrial clay.

Geochemical analysis;

Table 1 shows that Imiegba clay is not fit for the manufacturing of refractory bricks and ceramics because of the insufficient amount of silica, and magnesia present in the clay. In this regard, their alumina content of the clays corresponds to the refractory industrial specification but not the Silica and Magnesia. (Parker, 1967) and ceramics (Singer and Sonja, 1971). The high alumina silica ratio value in the Imiegba clay renders it less suitable for the production of good quality cement. Dunuweera et al (2018), Abatan et al (1993).

The presences of iron oxides, titanium dioxide and chromium in all the clay samples studied make them suitable for paint production. This is because they act as pigment and creates different colour e.g iron oxides can be used for yellow, red, brown or orange paints, titanium oxide is used for white paint while chromium for green paint. Emmanuel et al (2015).

X-ray Diffraction: The X-ray diffractogram of Imiegba clay deposit (Table 3) revealed the presence of kaolinite, illite, smectite, albite, anatase, quartz, microcline, muscovite, as the predominant clay minerals with kaolinite dominating the mineralogy. The semi quantitative mineralogical composition was determined based on relative peak intensities and reflects to a reasonable extent the mineralogical characteristics of kaolinite.

Particle Size Analysis: From (Table 4) The clay deposits studied also contains moderate amounts of silt-size particles (34%-41%) which makes them suitable in their raw state for use as fillers and coating materials in the paint industries. (Aliu et al, 2021). The loss on ignition (LOI) of 12.40 to 12.44% is an indicative of low porosity and that the finished products would show no cracks or damages on firing, thus, rendering them suitable for ceramic wares. Liquid limit and plasticity index are used to classify fine-grained soils. All liquid limit values in this study are <100%, classifying the clay as inorganic clay. Plasticity index ranging from 35.80-39.38 indicates the clay is of high plasticity. This observation was further supported using Casagrande, (1948) plasticity chart. (Figure) further support this assertion. Thus Imiegba clay, an inorganic clay can easily be moulded and satisfy requirements for pottery making. The clay samples studied is suitable for glazed products on account of its low amount of Fe₂O₃ and MgO and the absence of accessory minerals such as pyrite, apatite, siderite and magnesite. These impurities would cause undesirable colourations to the intended use, but can be removed through different beneficiation processes such as air flotation/magnetic separation/dry process, wet beneficiation and micronizing. Aliu et al (2021)

Furthermore, the use of the clay samples studied in the ceramic industries for the manufacturing of ceramic ware, ceramic glaze, would depend on the degree of beneficiation achievable to turn them into good quality fire clays.

CONCLUSION

Compositional features and industrial application of Imiegba clay was evaluated based on mineralogy, chemical composition and physical characteristics of the deposit with a view to determining its suitability as an industrial raw material. From the study, it is obtained that Imiegba clay deposit is predominantly kaolinite as the dominant clay mineral but with high SiO₂ and AL₂O₃ contents and quartz as the dominant non-clay mineral. It is rare for any single clay deposit to completely satisfy all the manufacturing requirement of its derivative product. Obviously, different sets of criteria are important to the production of any specific blend for a specific industry. Abel O.

(2012). Therefore, Imiegba clay can find application in pottery, paper and paint industry with beneficiation of the deposit where necessary for other industrial application.

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