

Geotechnical Properties and Geochemical Composition of Kaolin Deposits in Parts of Ifon, Southwestern Nigeria

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ABSTRACT : This study dealt with the geotechnical and compositional characteristics of the kaolin deposits in Ifon area of Ondo State using a combination of methods including X-ray diffraction analysis. The results show that kaolinite and quartz are the dominant clay minerals while pyrophyllite, macassite, pyrite and gippsite occur in minor amounts. The bulk chemical composition is dominated by SiO_2 and Al_2O_3 which abundance respectively range from 28.1 % - 38.98% and 21.61% - 29.77% with appreciable amounts of FeS_2 (0.18% - 15.37%). The ratio of SiO_2 to Al_2O_3 is 1.3 and is greater than the theoretical value of 1.18 for kaolinites, thus indicating the presence of impurities. Other oxides include Fe_2SO_3 , CaO , NaO , MgO , K_2O and TiO_2 . In the unfired state, the kaolin is whitish in colour but turns to pinkish white after firing which indicates that they could be made into ceramic products of natural good colour. Geotechnical tests show that the samples are dominated by clay-sized particles with a linear shrinkage that ranged from 4%-10%, plasticity index (16.8% - 23.9%) and loss on ignition (9.6% - 24.5%). The results of all the analyses suggest that Ifon kaolin is a suitable industrial raw material for the manufacture of ceramics, refractory bricks and tiles; as well as additives in the production of paper, paint and fertilizer.

Keywords: kaolin, Ifon, Nigeria, ceramics, firing clay, XRD.

I. INTRODUCTION

Kaolin is commonly the product of weathering of naturally occurring hydrated aluminium silicates. In many uses, the term kaolin typically includes the raw clay and refined commercial products (Bloodworth et al., 1993). As a clay-rich rock, it is dominated by kaolinite, a clay mineral that is composed mainly of $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. Ideally, kaolin has specific gravity that ranges from 2.58 to 2.63 with a refractive index of between 1.56 and 1.62 (Gushit et al., 2010) and feels plastic or slippery when touched. Because it is chemically unreactive under normal conditions, kaolin is in high demand as a raw material in the production of paper, ceramics, paint, chalk, cosmetics, pharmaceuticals and agrochemicals. Estimates of industry demand for kaolin is shown in Figure 1 with papermaking concerns dominating all the sectors.

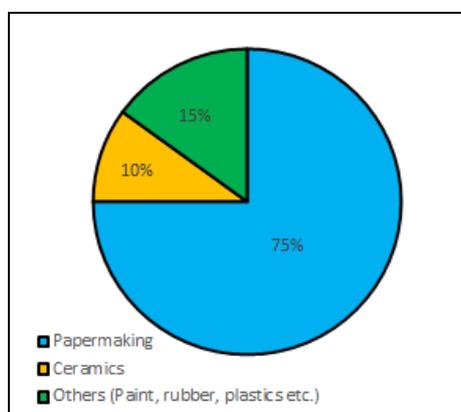


Figure 1: Estimated sectoral demand of kaolin (source: Bloodworth et al., 1993). Papermaking industries have the greatest need for kaolin.

Kaolin is found in large quantities in many parts of Nigeria. The occurrence of clay-rich deposits in the southwestern region has long attracted the attention of many researchers (e.g. Jones and Hockey, 1964;) and further elicited other studies (e.g. Elueze, 1983; Enu and Adegoke, 1986) that looked at their industrial potentials. These earlier studies dealt with the geology of the areas under study and documented results which show that kaolin occurs in commercial quantities. By default, many of the industrial properties ascribed to some of the kaolin deposits appear to be influenced by inferences from similar deposits in other kaolin-rich regions of the world. Thus, the seeming gap in local knowledge may have contributed to the unimpressive appreciation of economic and industrial values of Nigerian kaolin deposits with the implication that local industries depend on imported kaolin while dismissing local sources as not meeting the required industrial specifications. It is reported (RMRDC, 2003) that Nigeria is endowed with different grades of kaolin with a proven reserve that runs into billions of tonnes. However, as shown in Table 1, it is disappointing that Ifon kaolin is not captured in this national estimate and is not listed among the areas to be considered for kaolin-related investments.

Table 1: Major kaolin deposits in Nigeria and estimated reserves (modified from Gushit et al., 2010)

State	Major Locations	Estimated Reserve (million tonnes)
Abia	Isikwuato, Nneochi, Obingwa, Umuahia area, Ikwuano	Very large
Anambra	Anambra-East, Ayamelum, Ekwusigo, Njikoka, Aguata, Ihiala, Nnewi-South	4.2
Bauchi	Alkaleri, Bauch, Damban, Darazo, Ganjuwa, Kirfi, Misau, Ningi	18
Benue	Obi, Ogbadibo, Apa, Oturkpo	10
Borno	Gwoza, Chibok, Damboa	Large
Edo	Esan, Ovia and Etsako areas, IkpobaOkha, Akoko-Edo, Egor, Igueben, Oredo, Orhionmwon, Uhunmwonde, Owan-East	Very large
Enugu	Enugu	50
Kaduna	Maraban-Rido	5.5
Katsina	Batagarawa, Batsari, Dan-Musa, Danja, Dutsin-Ma, Ingawa, Kankara, Kankia, Safana, Malumfashi, Musawa,	Very large
Kebbi	Bagudo, Suru, Wasagu / Danko	Very large
Kwara	Baruten, Edu, Ifelodun, Ilorin, Ilepodun, Isin, Kaiama, Pategi	Large
Niger	Bosso, Edati, Gbako, Katcha, Lapai, Lavun, Mashegu, Mokwa, Paikoro, Shiroro, Wushishi	Very large
Ogun	Abeokuta area, Ado-Odo/Ata, Idarapo, Ifo, Igbado area, Ijebu area, Ipokia, Obafemi-Owode, Odeda, Odogbolu, Remo-North	Large
Ondo	Irele, Odigba	Not available
Oyo	Atisbo, Ifedayo, Iseyin, Saki West and East.	1.5
Plateau	Barkin-Ladi, Bassa, Bokkos, Jos-North, Kanke, Pankshin	8.0
Zamfara	Gumi, Gusau, Kaura-Namoda, Maradun, Maru, Talata-Mafara, Zurmi	Not available

It is against this backdrop that the present study provides fresh insights into the geochemical and geotechnical properties of Ifon kaolin deposits in order to initiate a more robust and balanced evaluation of their industrial potentials and possibly enlist it in the national database of commercially viable kaolin resources. This is because the nature and compositional characteristics of clays are the major determinants of their commercial value and engineering behaviour (Onyeobi et al., 2013). These properties of importance are elemental and mineralogical composition, physical properties (e.g. particle size distribution, swelling and shrinkage potentials), organic matter content and amount of non-clay minerals.

II. LOCATION AND GEOLOGICAL SETTING

Ifon, the study area is located in the present-day Ondo State in the southwestern region of Nigeria. As shown in Figure 2, the spatial coordinates are defined by latitudes 6°47' N and 6°49' N longitude 5°30' E and 5°33' E respectively.

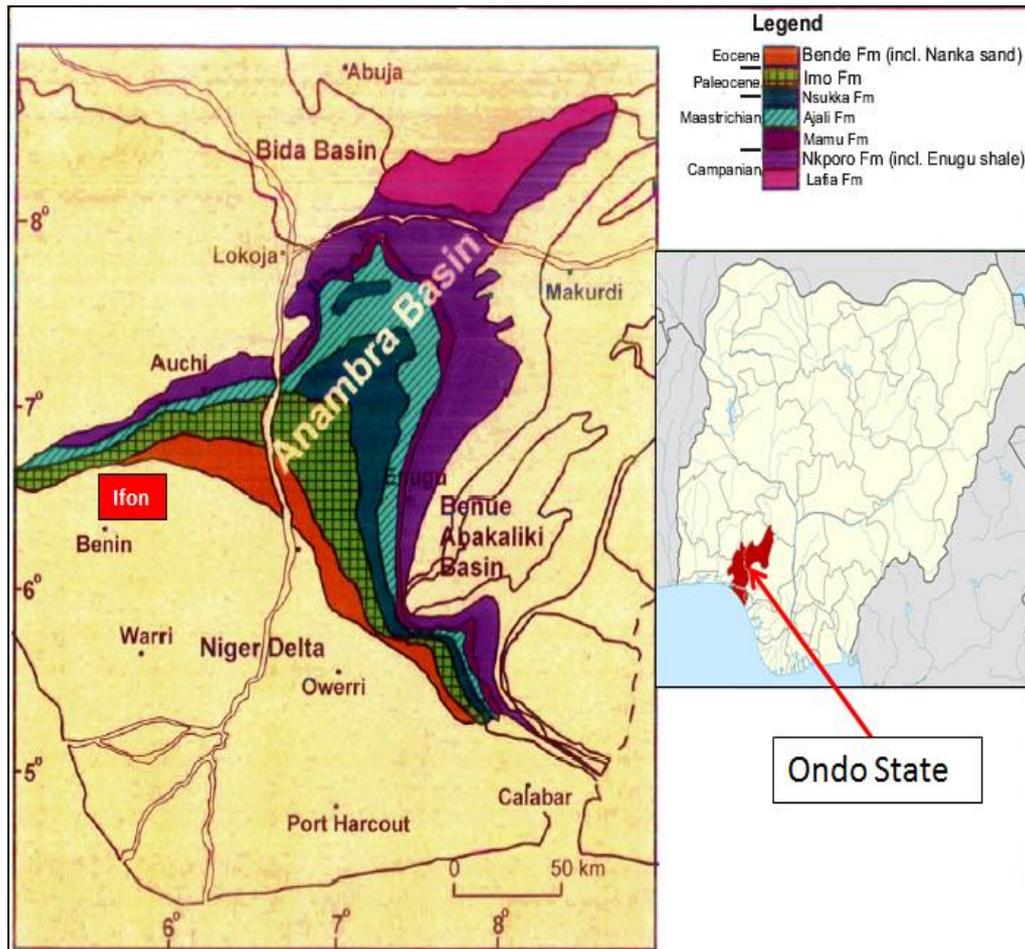


Figure 2: Section map of Nigeria showing the regional geology of Anambra Basin. The study area, Ifon is in red box. Inset is map of Nigeria showing Ondo State (Modified from: Geological Map of Nigeria GSN 1994).

The study area is generally characterised by a gentle to low-lying terrain, though some spot heights measure as much as 230 m above mean sea level. It is drained by numerous tributaries of rivers Ose and Nkporo. These have combined to form well developed valleys and river channels with good exposures of the underlying geological strata in many of the localities. The sedimentary rocks are dominated by Cretaceous and post-Cretaceous formations that belong to the Dahomey and Anambra basins.

Durugbo and Aroyewun (2012) has interpreted the sedimentary rocks along Ifon-Sabongida Road of Ondo State as part of the Abeokuta Group (Araromi and Abeokuta formations) of the Dahomey Basin. However, on the geological map of Nigeria (Figure 2), Ifon and environs sit on the western flank of Anambra Basin that respectively extends north-eastwards and north-westwards to Lafia and Niger Valley. To the north, east, south and west of the Anambra Basin are the Jos Massif, Abakaliki Anticlinorium, Niger Delta Basin and Ibadan Massif respectively. Thus, the Anambra Basin can be described as the northern extension of the Niger Delta. Murat (1972) while describing the evolution of Anambra Basin linked its origin to the late Cretaceous (ca 83.3 ma) tectonics that affected the Abakaliki-Benue Basin. Subsequently, phases of transgression and regression brought about deposits of marine and continental origins (Anyanwu and Arua, 1990) that filled up the accommodation space. Nwajide (2013) presented a detailed discussion of the history and economic potentials of Anambra Basin and other sedimentary basins in Nigeria. Perhaps, Ifon and environs constitute the poorly defined and debatable boundaries of Dahomey and Anambra Basins.

III. MATERIALS AND METHODS

Eight representative samples were collected from the study area and carefully stored in sample bags to avoid contamination. The fresh samples were typically whitish in colour, with a soft, earthy feel when touched. The samples were later subjected to physical and chemical analyses but four of these samples were further subjected to XRD test. Samples were prepared for laboratory analyses by air drying for two weeks after which they were gently crushed (not ground) to increase their surface area. Subsequently, the natural moisture content, grain size distribution, viscosity, liquid limit, plastic limit, plasticity index, shrinkage limit and firing behaviour

were determined. The bulk chemical analysis tested for SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , Na_2O , K_2O and pH. All the physical analyses were carried out in the Geology Laboratory of the Federal University of Technology, Akure while the fired behavior was done in the Presidential Paint Industry Laboratory, Abeokuta. These tests followed analytical guidelines described in the British Standards-BS 1377(1990) and American Standard for Test Materials -ASTM (2004).

IV. RESULTS AND DISCUSSION

i. NATURAL MOISTURE CONTENT

Moisture content is the weight of water in the sample to weight of solids. The addition of water to dry clay has the effect of an increase in cohesion, which tends to reach a maximum when water saturates all the pores between the particles (Andrade et al., 2011). Apart from the more commonly known volumetric changes that occur in kaolin clays with changes in moisture content, their strength is also affected. Because of their high expansive nature when saturated with water, kaolin clays are regarded as poor engineering materials with low shear strength. Processes of stabilizing kaolin clays for engineering purposes need a good knowledge of their moisture content. Experimental results in Banaszak (2013) using kaolin clay, illite clay and electrotechnical porcelain show how the strength of dried materials may vary with moisture content and changes that may occur during the drying process. In all these, there is a specific level of moisture content at which the clay is easily workable; below which the molded body will crack. In this study, dry season samples of kaolin were analysed for their natural moisture content. The result of the moisture content of the kaolin samples ranges from 15.00% to 20.82% as shown in Table 2.

Table 2: Moisture content and Atterberg limit characteristics of Ifon kaolin clay.

SAMPLE	Moisture content (%)	Linear Shrinkage (%)	Liquid Limit (%)	Plastic limit (%)	Plasticity Index (%)
1	15.66	7.3	41.7	23.9	17.8
2	20.37	9.4	47.6	24.2	23.4
3	18.52	6.6	40.7	23.9	16.8
4	20.82	8	44.6	23.9	20.7
5	15	7	44.5	24	20.5
6	17.5	10	44.6	24.1	20.5
7	18.52	6.7	46.9	23	23.9
8	16	4	45.7	22.8	22.9

ii. PARTICLE SIZE DISTRIBUTION

The analysis of grain size distribution in a kaolin deposit is a very important step in the evaluation of kaolin for industrial use. This is because the grain size of kaolin particles affects the quality of its finished products, hence, fine particles (< 0.002 mm) are the most desirable. As demonstrated in Hubadillah et al. (2016), kaolin membrane structure, pore size distribution, porosity, mechanical strength, surface roughness and gas permeation is a function of the size distribution of particles. The result of that study indicated that the morphological characteristics of ceramic support is determined to a certain extent by the different sizes of kaolin particle.

The result of the grain size analysis carried out on the Ifon kaolin samples shows that the samples are dominated by small-sized particles; more of clay size (< 0.002 mm). From the graphs shown in Figure 3, it could be inferred that the proportion of clay-sized particles constitutes more than 50% of the kaolin samples. In line with the findings of Hubadillah (2016), a clay-size dominated distribution of this type would indicate that the kaolin has a higher tendency for agglomeration because smaller particles agglomerate more easily and will also impact on the viscosity of suspensions. This is because the viscosity of smaller-sized kaolin is higher than that of the larger-sized particles; hence it was concluded that smaller particle size would affect the overall properties and performance of kaolin.

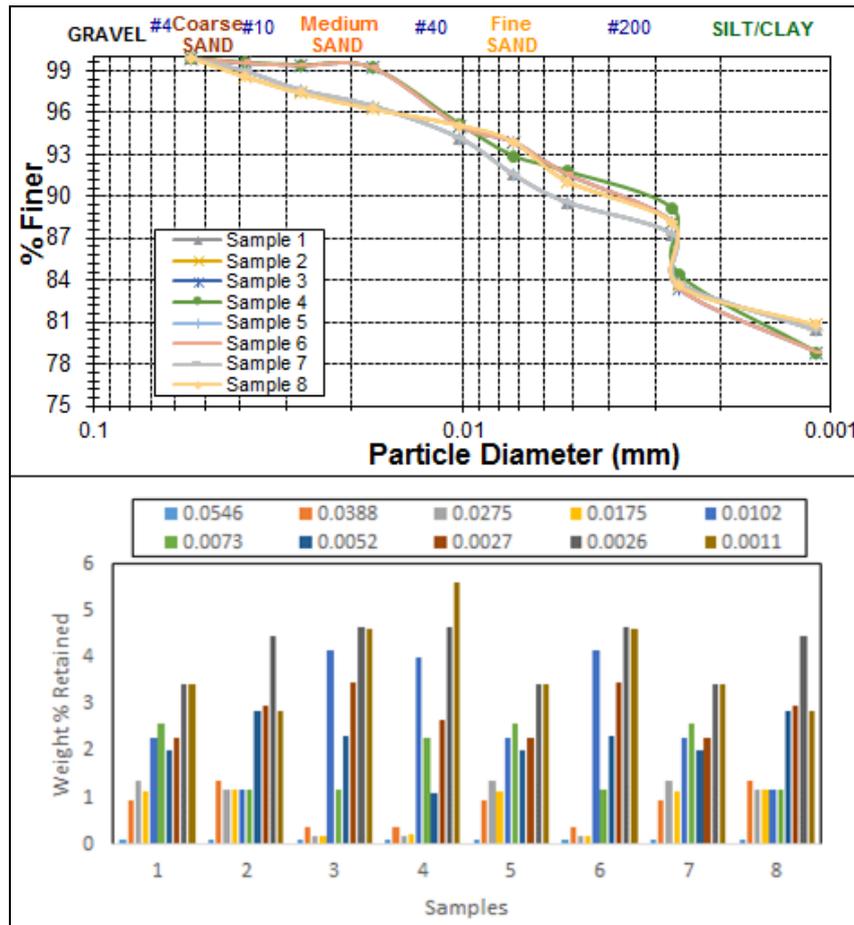


Figure 3: Particle-size distribution of kaolin samples. The weight distribution shows that samples are dominated by <0.0027 mm particle sizes.

iii. **ATTERBERG TEST**

To a large extent, Atterberg limits (liquid limit, plasticity limit, plasticity index) are properties that can reflect the amount and type of clay in the samples. They are also important in understanding of the industrial behaviour of kaolin clays (Spagnoli and Sridharan, 2013). The result of the liquid and plastic limits of the kaolin samples collected from the study area (see Table 2) shows that liquid limit of the samples ranges from 40.7% to 47.6% and the plastic limit ranges from 22.8% to 24.2%. The technological evaluation of kaolin with moderate range of plasticity (Figure 4) shows that the Ifon deposit could be exploited commercially for use in the ceramics industry. The deposits can be adjudged suitable for white ware and sanitary ware when compared with the kaolin industry specifications of NAFCON (1985).

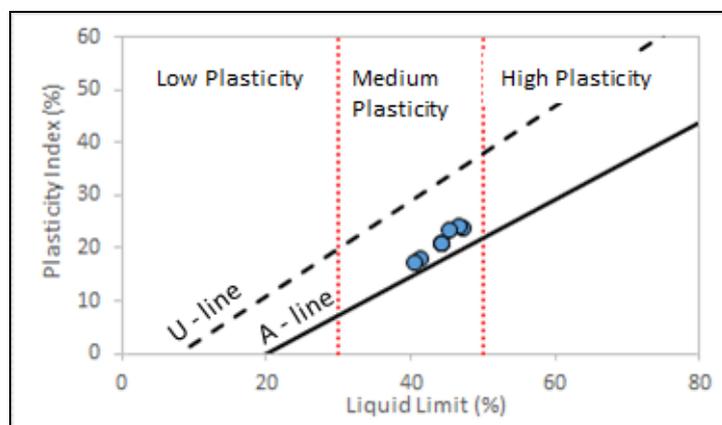


Figure 4: Character of Ifonkaolin samples on a typical Casagrande plot. The plot indicates medium plasticity for the samples considered in the analysis.

iv. DRY LINEAR SHRINKAGE

The linear shrinkage values range from 4.0% to 10.0% (Table 3), with kaolin samples 8 having the lowest linear shrinkage value of 4.0% while clay sample 6 having the highest linear shrinkage value of 10.0%. This implies that kaolin sample 8 with lowest shrinkage limit is likely to contain montmorillonite.

Table 3: Table of results for dry linear shrinkage analysis

SAMPLE	1	2	3	4	5	6	7	8
Original Length, OL (mm)	140	140	140	140	140	140	140	140
Dry Length, DL (mm)	125	122	126	124	123	126	121	120
Linear Shrinkage (%)	7.3	9.4	6.6	8.0	7.0	10.0	6.7	4.0

v. VISCOSITY MEASUREMENT

The resistance offered by a fluid to flow when subjected to speed gradient or shear stress is a function of its viscosity (De Noni Jr et al., 2001). This is one of the major rheological properties of kaolin that determines its suitability in the paper, pulp and paint industry. The paper production process requires adhesive slurries (which is dependent on viscosity) that can flow and give smooth, even coverage to coat papers and improve their surface properties. In this study, kaolin samples were made into slurries and their viscosities determined using the workflow described in Beazley (1974). The approach involves making kaolin slurries of different concentrations by progressive dilution with water and then measuring the viscosities with a viscometer. The measurements were made with a viscometer at a specific temperature of 25°C. A specific temperature datum was chosen because the aggregation of clay particles is known to vary with temperature changes. The result of the analyses shows that the viscosity of Ifon kaolin samples ranges from 3.00 to 5.31 poise as shown in Table 4. Since paint industries prefer kaolin viscosity in the range of 5.0 – 6.0 poise, a large proportion of Ifonkaolin can be considered as good raw materials in the production of paint.

Table 4: Viscosity and firing behaviour of tested samples.

SAMPLE	Viscosity (poise)	Fired Shrinkage (%)	Apparent porosity (%)	Water adsorption (%)	Bulk density (%)	Pre-firing colour	Post-firing colour
1	5.31	20.12	16.01	25.21	0.51	whitish	Pinkish white
2	4.51	24.00	10.11	28.90	0.72	whitish	Pinkish white
3	4.72	24.36	12.89	28.01	0.83	whitish	Pinkish white
4	3.91	19.89	17.10	27.10	0.72	whitish	Pinkish white
5	4.6	24.10	17.30	30.21	0.65	whitish	Pinkish white
6	5.21	21.11	15.90	33.26	0.55	whitish	Pinkish white
7	3.9	20.01	14.00	30.09	0.49	whitish	Pinkish white
8	3.0	19.21	15.01	30.11	0.83	whitish	Pinkish white

vi. FIRING BEHAVIOUR

The firing behaviour was tested using temperatures that ranged from 600°C to 1250°C. This was to cover the range of temperatures at which ceramic products are manufactured. The properties of the fired samples (fired shrinkage, water adsorption, apparent porosity and bulk density) are presented in Table 4. All the measured properties showed an increasing trend when the sintering temperature was 800°C. Above this temperature, samples sintered at 1250°C exhibited higher levels of fired shrinkage with a peak value of 24.36% and bulk density (0.83), with a corresponding reduction of apparent porosity (12.89%) and water adsorption (28.01%). The pink white colour produced by the kaolin samples indicates they will give good colours when used in the making of ceramic products.

vii. CHEMICAL ANALYSIS AND pH

The results of chemical analysis and pH values of the samples are detailed in Table 5 and compared with known industry standards (Table 6). The SiO₂ and Al₂O₃ contents of samples 1 to 8 were consistent with mineralogical observation. The relatively lower K₂O concentration in sample number 1 compared to sample number 2 from the same seam at a difference in depth of 10m indicates its preferential leaching from muscovite (Robertson et al., 1991). The concentration of SiO₂ in the samples is quite low. This might be due to the winnowing action during transportation and sedimentation. Also, the distribution of TiO₂ with depth has no particular trend and could be indicative of its immobile nature during weathering (Heckroodt et al., 1987). The concentration of MgO is attributed to smectite while high amount of Fe₂O₃ in the samples accounts for the presence of FeS₂ minerals, marcasite and pyrite. It is thought that hematite contributes to the iron content of the deposit as depicted with a value of 22.20% found in location 8. The pH of the kaolin ranges from 2.64 to 5.48. The high acidity implies the effect of ongoing weathering. The acidity correlates with the FeS₂ and organic carbon content. The pH is lowest in sample 1 (2.64) while the concentration of FeS₂ (15.37%) is highest in sample number 6. Figure 5 shows that the concentrations of the major oxides are within the limit specified for various industrial purposes.

Table 5: Average concentration (%) of various geochemical parameters in the kaolin samples (1-8).

Major oxides	1	2	3	4	5	6	7	8	pH
SiO ₂	36.82	38.98	34.06	37.59	38.31	29.38	29.76	28.21	2.64
Al ₂ O ₃	21.61	23.91	26.23	26.92	25.45	28.72	27.08	29.77	3.14
Fe ₂ O ₃	10.76	1.97	6.64	9.81	1.35	2.36	2.89	22.2	2.75
CaO	0.13	0.28	0.39	0.41	0.18	0.13	0.14	0.38	2.67
Na ₂ O	0.45	2.04	0.67	0.53	0.67	0.58	0.98	0.36	3.29
MgO	0.13	0.52	0.02	0.15	0.06	0.33	0.40	0.49	3.28
K ₂ O	2.51	1.90	1.05	0.60	2.24	2.24	3.73	1.50	5.48
TiO ₂	1.30	1.00	0.98	1.44	1.59	1.35	1.84	3.35	2.77
FeS ₂	0.18	0.31	9.96	1.15	8.47	15.37	1.34	0.95	NA
Ignition Loss	9.59	9.38	24.49	18.20	17.33	21.62	13.04	17.13	NA

Table 6: Properties of kaolin specified by various industries in Nigeria (source: Gushit et al., 2010)

Major oxides	Pharmaceuticals (%)	Ceramics (%)	Filling agent (%)	Coating Agent (%)	Steel (%)
SiO ₂	48.00	48.00	48.70	47.80	5.00
Al ₂ O ₃	36.00	37.00	36.00	37.00	0.002
Fe ₂ O ₃	0.10	0.60	0.82	0.58	0.002
TiO ₂	0.02	0.30	0.05	0.03	-
CaO	0.01	0.10	0.60	0.40	54.28
MgO	0.20	0.30	0.25	0.16	3.00
K ₂ O	1.10	1.60	2.10	0.10	-
Na ₂ O	0.10	0.10	0.10	0.10	-
Ignition Loss	11.90	12.40	11.90	13.10	-

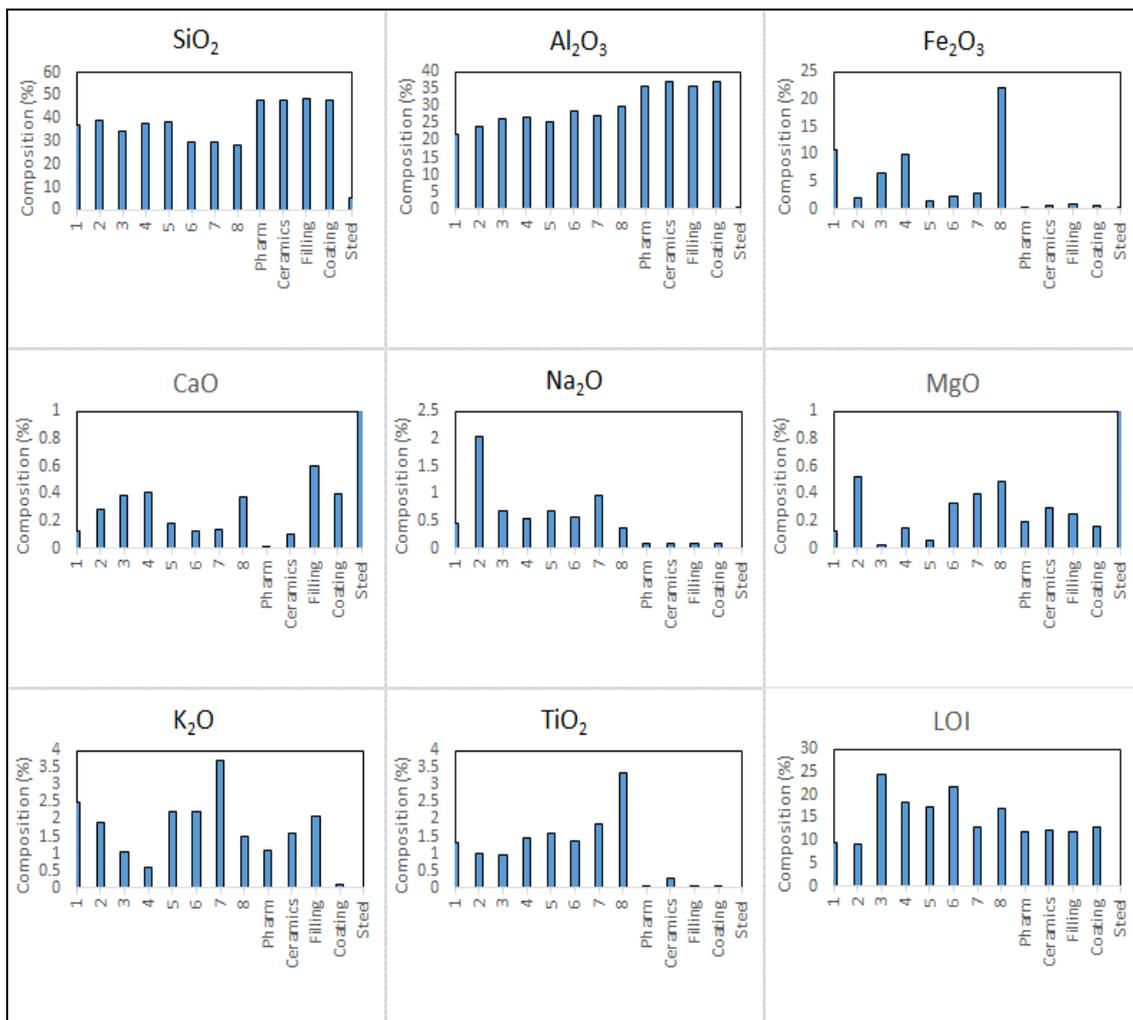


Figure 5: Comparison of chemical properties of Ifon kaolin against specific industrial standards.

viii. XRAY DIFFRACTION STUDIES

The mineralogical analyses of Ifon kaolin samples are shown in Figure 6 and summarized in Table 7. The constituent minerals were expressed as major, minor and trace depending on their concentrations. Three of the samples consist chiefly of kaolinite and quartz with considerable muscovite, gibbsite and pyrophyllite while the remaining samples are devoid of pyrophyllite, suggesting their conversion to kaolinite. The same conclusion was reported by Heckrodt and Buhmann (1987) suggesting the formation of pyrophyllite from muscovite during early stages of weathering and its alteration to kaolinite at a later stage. Microcrystalline quartz and fine-grained mica, *i.e.* illite, constitute minor ingredients.

Most of the samples exhibit an association of pyrite and marcasite along with kaolinite, quartz and gibbsite. Sedimentary pyrite formation occurs during the reaction of a detrital iron mineral with H₂S, which was formed by the reduction of sulphate by bacteria in the presence of organic matter (Bernel, 1984). Trace amounts of smectite were identified only in sample 5. The formation of smectite is favored by a microenvironment with poor drainage and hence minimum leaching conditions with relatively high metal ion concentrations (Keller, 1985).

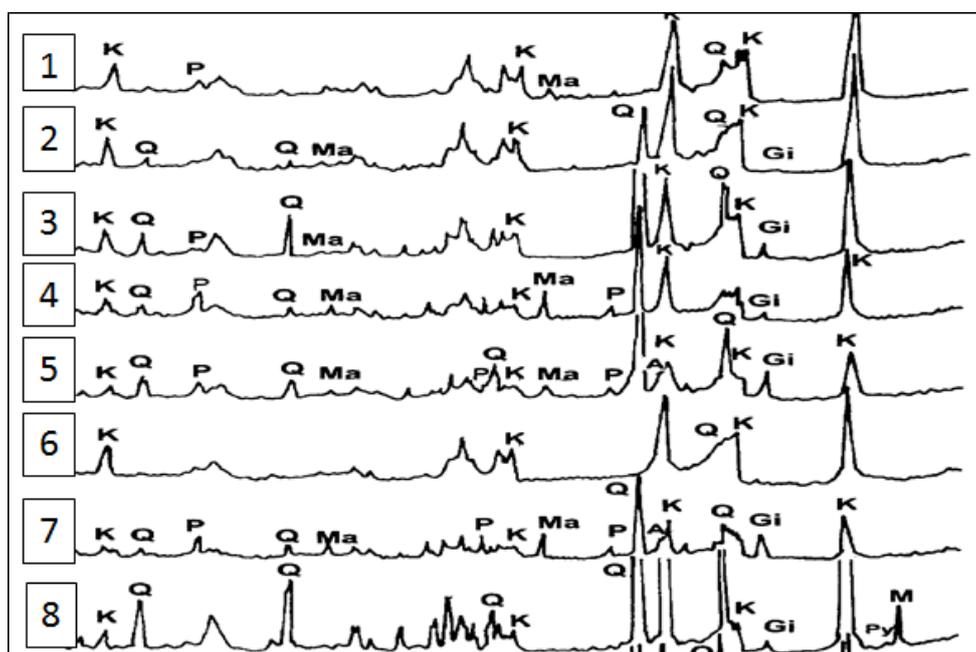


Figure 6: XRD pattern of clay samples. K= kaolinite, Py= pyrophyllite, Gi= gibbsite, P= pyrite, Ma= marcasite, I= illite and H= Hematite.

Table 7: Relative abundance of minerals based on XRD analysis

Minerals	1	2	3	4	5	6	7	8
Kaolinite	M	M	M	M	M	M	M	M
Quartz	M	M	M	M	M	M	M	M
Smectite	A	A	A	A	T	A	A	A
Pyrophyllite	Mn	Mn	A	A	A	A	A	A
Pyrite	Mn	A	Mn	Mn	Mn	A	Mn	A
Macassite	A	Mn	Mn	Mn	T	A	T	A
Hematite	A	A	A	A	A	A	T	Mn
Illite	A	A	A	T	T	A	A	T
Gibbsite	A	Mn	A	Mn	T	A	A	A

KEY: M = major mineral; Mn = minor mineral; T = trace mineral; A = absent.

Gibbsite identified by the 4.84A peak in samples 3, 4, 5 and 7 indicates intense leaching in a strongly deionized environment under tropical climate (Vazquez, 1981) during desilication (*i.e.* dissolution of Si from its aluminosilicate parent material). The association of the above minerals in the same kaolin resulted from the changes in the microenvironment at the time of deposition or mineral formation. Fine quartz grains were noted in the kaolin samples that have large amounts of marcasite/ pyrite. This is explained in Chen et al. (1997) as suggestive of the formation of microcrystalline quartz due to the rearrangement of the feldspar and muscovite structures during kaolinization or kaolinite recrystallization.

The ferruginous sample 8 clay shows an assemblage of kaolinite, quartz and hematite. Hematite is concentrated in the fine clay fraction of the kaolin deposit. The presence of hematite indicates an oxidizing environment and the presence of iron sulfides indicates reducing environment conditions during the formation or deposition of gray carbonaceous and ferruginous clay respectively. The crystallinity index of kaolinite varies from one kaolin layer to another. This variability may be attributed to the intensity of weathering or the extent of transportation of the clay during deposition (Brindley, 1986).

V. CONCLUSION

The compositional analyses of Ifon kaolin deposits have been carried out in this study. Comparisons have been made with the works of other authors and inferences also drawn to confirm the results of the analyses and economic viability of the deposit. The results of the physical analyses carried out on samples collected from the deposits show that Ifon kaolin could be a suitable raw material in the production of ceramic products, paints and cement. An evaluation of the industrial potentials of the kaolin deposit based on the fired behavior and viscosity measurement of the samples shows that the Ifon kaolin is characterized by a recovery clay of more than 50% as shown in the grain size chart. The samples exhibited improvements in colour when exposed to firing. This would indicate that the kaolin contains goethitic iron impurity (Kogel and Hall, 1999) which converts to hematite during calcination with a high tendency for efficient bleaching. The pinkish-white fired colour of the kaolin that remains even after bleaching could be an evidence of the presence of colour-imparting impurities.

The X-ray diffraction analysis also shows that kaolinite and quartz are the major minerals in Ifon kaolin deposits. The constituent minor minerals are marcasite, pyrite and hematite with traces of illite and smectite. Considering the major minerals and valuable physical and chemical properties of the analysed kaolin samples, this study is of the view that the kaolin deposits found in Ifon area are of good quality and have great potentials as industrial raw materials for the production of ceramics, refractory bricks, paper, paint and fertilizers.

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