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The Geochemistry and Mineralogical Composition of Ogiso and Okhoro Clay Deposits of the Benin Formation, Nigeria: Insights into Its Provenance and Industrial Significance

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INFORMATION ABSTRACT

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This research work was carried out to determine the chemical composition of clay deposits in the Benin Formation, Southern Nigeria. In this study, ten (10) fresh clay samples were randomly collected from two (two) clay outcrops. X-ray diffraction (XRD), X-ray fluorescence (XRF), grain size analysis, and Atterberg tests analyses were employed to determine the mineralogy, major oxides, trace element composition, and the physical properties of the clay samples. The XRD analysis revealed that the basic mineralogy of the samples studied consists of kaolinite, quartz and iron hematite with traces of zircon, illite and anatase occurring in minor amounts in some of the samples. The XRF analysis revealed the major elemental oxides to be $SiO₂$ with a range of 55.82 to 61.41 wt%, AlO with a range of 21.12 to 24.42 wt% and FeO with a range of 6.05 to 9.06 wt% while the major elemental trace elements include zircon with a range of 0.89 to 1.78 wt%, zinc with a range of 0 to 1.51 wt%, copper with a range of 0 to 1.39 wt% and chromium with a range of 0.01 to 0.11 wt%. The high chemical index of alteration (CIA) values, high chemical index of weathering (CIW) values and moderate ratio of TiO /Zr indicated an intense weathering source area. Conclusively, the mineralogical composition, the elemental trace element, main element discrimination diagram and the elemental ratios of the samples such as TiO /AlO indicated a provenance of intermediate source. The grain size analysis and Atterberg tests revealed sandy-clay deposits that have low to medium plasticity, moderate to high liquid limit, moderate moisture content, relative high swelling capacity, high linear shrinkage and low specific gravity values are less desirable for construction purposes due to potential deformation, cracking and settlement in buildings. The results further indicate the clay samples from the study areas are mainly kaolinite and categorized as commercial ball clays.

1. Introduction

Clays are earthly and naturally fine-grained inorganic polymineralic materials of \leq 2 μ m in size. The layered structures of these hydrated aluminosilicates clay minerals determine their characteristic chemical and physical properties of the clays which are formed as a result of chemical weathering of pre-existing crystalline rocks and feldspar minerals under warm tropical and subtropical climatic conditions or as a result of the hydrothermal alteration (Mpuchane et al., 2008). The basic structure of layer silicates and all silicates ion $(SiO₄⁴)$, where the silicon occupies the tetrahedral site. The aluminium ion $(A³⁺)$ can

substitute for $Si⁴⁺$, but it is generally located in the octahedral sheet. Their characteristic auto construction form the unit cell of layering to yield; 1:1 type of clay minerals that consist both tetrahedral and octahedral sheets to form; kaolinite– serpentinite, kaolinite and halloysite clay minerals (Meunier and Velde, 2004) and 2:1 types of clay minerals that have two tetrahedrally coordinated sheets of cations both positioned in between the octahedral sheet to form illite, montmorillonite, chlorite (Shichi and Tagaki, 2000; Nayak and Singh, 2007; Burhan and Ciftci, 2010).

The major geological process that is responsible for the

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compositional trend of clay rich sediments is chemical weathering although the parent rock in some instances weathers into soils and clays by combined action of biological, chemical and physical weathering (Nesbitt and Young, 1989; Singh, 2009). It has been proven that imprints of the original composition of the precursor are readily preserved in the sediments (Frallick and Kroneberg, 1997, Nesbitt et al., 1996; Cox et al., 1995). Furthermore, these clay minerals (illite, kaolinite, montmorillonite) have been discovered to be useful within the industries as the major raw material in the production of ceramic, paint, paper, refractory, plaster of Paris and pharmaceutical products used in the cure of ulcers, dysentery and cholera, detoxification, adsorption and skin emulsifiers (Mpuchane et al., 2008).

Fig. 1. Geological map of the study area

Table 1. Mineralogical composition (% vol.) of clay samples from Ogiso and Okhoro locations within the Benin Formation

They are also used as filling materials in construction works and as a major constituent in fertilizer production. Sediments rich in clay minerals occur as weathered regolith above basement rocks, especially where the rocks are rich in silicate minerals; or as materials carried into depositional basins during erosional phase. The Benin Formation (Fig. 1) is one

of the sedimentary basins in Nigeria characterized by the deposition of sediments. Although there have been a number of studies in the Benin Formation, the clay deposits of the Benin Formation have not been fully explored by researchers.

Fig. 2. X-ray diffractograms of: Ogiso clay (A) – (G); Okhoro clay (H) – (J) deposits of the Benin Formation depicting kaolinite, hematite, quartz, illite, anatase, and zircon

Most of the past studies focused on the sandstone aquifer potentials. Therefore, this study is aimed at providing the geochemical and mineralogical compositions of the clays deposits in the Benin Formation using x-ray diffraction (XRD) and x-ray flourescence (XRF), since the mineralogical

composition of a rock is critical to its behavior and application. Considering the classification of clay mineral groups (kaolin, smectite, illite, and chlorite), the mineralogy examination of the clay deposits is useful in determining their suitability for different applications.

The demand for clay minerals as raw materials in paper, ceramics, paint and coatings, fibre glass, plastic, rubber, cosmetics, pharmaceutical and medical applications are probably going to arise as a compelling variable for the business development over the conjecture time frame (Grand view research, 2020). Nigeria has several industries that can make use of these readily available and cheap clay minerals as raw materials. However, in order to improve the industrial utilization of clay minerals of Nigeria by local and foreign industries, there is need to assess the clay deposits mineralogy for proper characterization of the clay deposits, hence this study.

Table 2. Major oxide composition of clay samples from Ogiso and Okhoro locations within the Benin Formation

Table 3. Trace elements concentration (ppm) of clay samples from Ogiso and Okhoro locations within the Benin Formation

2. Location and Geology of the Study Area

The study was carried out at Ogiso and Okhoro districts in Benin City, Southern Nigeria as indicated in Fig. 1. The study area lies within latitude $6^{\circ}20'20''$ and longitude 5°36'10" and latitude 6°18'10" and longitude 5°41'50". Ogiso clay deposit shows intercalations of clays and reddish sand (lateritic) while, Okhoro shows a light grey with brownish patches clay overlying with reddish sand respectively. The brownish and reddish coloration in Ogiso and Okhoro can be attributable to oxidation. The climate in both locations is characterized by wet and dry season. Both locations are densely populated with tall trees and shrubs suggestive to be of the tropical rain forest zone. The topsoil ranges from brownish to reddish sand.The study area was accessible by (major and minor roads) and footpaths. The areas have good road network. Most places are built up areas, and there are footpaths to the various outcrops. The Benin Formation is underlain by sedimentary formation of the south sedimentary basin. The geology is generally marked by top reddish earth, composed of ferruginized or lateralized clay sand.

The term Benin sand was first used to describe the reddish earth underlain by sands, sandy clays and ferruginized sandstone that mark the paleo-coastal environment of Paleocene-Pleistocene Age. These sediments spread across the southern fringes of the Anambra Basin and marking the upper fancies off-flaps of the Niger Delta Basin.

The name Coastal plain sands are used to describe the formation of red earth underlain by sands and clays that mark an ancient coastal plain environment now exposed in Calabar, Owerri, Onitsha and the Benin Region with the age Oligocene-Pleistocene. However, the name Benin Formation was reinstated to identify the reddish-brownyellow generally white sand soften with clayey and pebbly horizons with type-locality around Benin. This is also referenced at Calabar and other parts of Southeastern Nigeria.

The formation was further established by well logging of Etete 1-Well, drilled on-shore east of River Niger by Shell Nigeria Petroleum Development Company (SPDC) and described the formation as about 1830 m thick at the seashore but thins landwards. The sedimentary suits of the Benin Formation dip 2˚- 8˚ south. Geologically, the Benin Region comprises the Benin Formation, Alluvium; Drift/topsoil and Azagba-Ogwashi (Asuba-Ogwashi) Formation.

3. Materials and Methods

The method involves sampling of clays and bulk elemental composition and mineralogical analysis of ten (10) clay samples from Ogiso and Okhoro clay deposit outcrops in the Benin Formation. Accessibility and collection of the samples were made possible by river channel exposure (Fig. 1). In the field, the profiles were observed and described based on their color, texture and thickness. Fresh clay samples were collected at different sites labeled and arranged in sample bags and processed in the laboratory. A quantitative determination of the elemental and mineralogical composition of the clay samples using X-ray fluorescence and X-ray diffraction were carried out at the Natural Steel and Raw Material Exploration Agency, Kaduna, Nigeria.

Fig. 3. (A): Ternary diagram of SiO₂-Al₂O₃-Fe₂O₃ (After Schellmann, 1986); (B): Discriminant plot of sedimentary provenance (After Roser and Korsch, 1988); (C): K₂O/Na₂O vs. SiO₂ plot (Roser and Korsch, 1986); and (D): Position of clay deposit samples from the study area on TiO₂/Al₂O₃ binary plot (Ekosse, 2001) of Ogiso and Okhoro clay deposit outcrops

Fig. 4. CIW vs. CIA weathering plot of Ogiso and Okhoro clay deposit outcrops

Prior to XRD processing, 100 g of each dried sample were gently crushed and sieved and then the clay fractions were separated according to Moore and Reynolds (1997). These were later taken in an aluminium alloy grid (35 mm×50 mm) on a flat glass plate and covered with a paper. Each sample was run through the Rigaku D/Max-IIIC X-ray diffractometer developed by the Rigaku Int. Corp. Tokyo, Japan equipped 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 2°/min in the 2 to 50° at room temperature with a cuka radiation set at 40kv and 2mA. 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 was plotted against angle 2ϴ to produce a chart. The angle 2ϴ for each diffracted peak can be converted to d-relative intensity obtained and compared to that of the standard data of minerals established by Brown (1951), Carrol (1971) and the joint Committee on powder Diffraction Standard (JCPDS)

the mineral powder diffraction file (1980), which contained and includes the standard data of more than 3000 minerals. Percentage mineralogical composition was also estimated based on the relative peak intensities of the respective minerals in the XRD charts. Grain size analysis and Atterberg limits (liquid limit, plasticity index, and plastic limit), linear shrinkage, specific gravity, swelling percentage, and moisture content were determined using the methods described by Adeola et al. (2020).

4. Results and discussion

4.1. Geochemical and Mineralogical Composition

XRD data of clay samples from Ogiso and Okhoro outcrops in the Benin Formation, indicate two clay minerals (kaolinite and illite), and two non-clay minerals (quartz and hematite), (Fig. 2). The XRD diffractograms depicts the mineralogical assemblage of quartz, kaolinite, illite and hematite. Quartz is the dominant mineral closely followed by kaolinite as indicated by the high intensity of its numerous peaks in the X-ray diffractograms (Fig. 2), which was confirmed by the mineralogical composition indicated in Table 1. The investigated clay samples are characterized by the presence of kaolinite which constitutes between 1.48–72.41% of the clay samples; and high quartz content (26.89–98.04 %; Table 1) which is responsible for the grittiness of the clay deposits observed in the field.

4.2. Major and Trace Element Geochemistry

The XRF analysis results (Table 2), of the clay samples indicate high silica content ranging from 55.82%–61.41% relatively low alumina content between 21.13%–24.42%; and low iron oxide content ranging from 5.03%–9.06%. While flux materials such as $Na₂O$, MgO , $K₂O$, $TiO₂$ and CaO were also found in low concentrations in the clay samples (Table 2). The high concentration of silica in the clay samples suggests the depletion of other major oxides such as CaO, MnO, MgO, Na₂O, etc. The relative high ratio of quartz ($SiO₂$) and alumina oxide ($Al₂O₃$) concentration in the clay samples suggests the weathering of a siliceous protolithic rock.

Fig. 5. (A): Ternary diagram SiO₂-Al₂O₃-Other oxides of clay samples compared with the chemical composition of commercial kaolins (Ligas et al., 1997) and ball clays (after Fabbri and Fiori, 1985); and (B): Ternary diagram SiO₂-Al₂O₃-Fe₂O₃ (After Aleva, 1994) of clay samples in the study area

Table 4. Major element oxides of the studied clays compared with chemical industrial specification

Elemental oxides	Ogiso	Okhoro	Refractory bricks (Parker, 1967)	Rubber (Keller, 1964)	Ceramics (Singer) and Sonjai, 1964)	Brick clay (Murray 1960)	(additives)	Payne, 1961 Paint
SiO ₂	59.33	59.25	$51 - 70$	44.9	67.5	38.67	48.7	48.68
AL_2O_3	22.67	21.86	$25 - 44$	32.35	26.5	9.45	36	9.45
K ₂ O	0.10	0.14	\sim	0.28	$1.10 - 3.10$	2.76	2.12	2.76
Fe ₂ O ₃	7.54	7.00	$0.5 - 2.40$	0.43	$0.5 - 1.20$	2.7	0.82	2.7
CaO	0.64	0.11	$0.1 - 0.2$	Tr	$0.18 - 0.30$	15.84	0.06	15.84
Ti ₂ O	1.30	1.26	$1.0 - 2.80$	1.8	$0.10 - 1.0$	\sim	0.05	$\overline{}$
MgO	0.16	0.19	$0.2 - 0.7$	Tr	$0.1 - 0.19$	8.5	0.25	8.5
Na ₂ O	0.49	0.6	$0.8 - 3.50$	0.18	$0.20 - 1.5$	2.76	0.1	2.76
MnO	0.08	0.08	\sim	0.01	$\overline{}$	$\overline{}$		$\overline{}$
P_2O_5	0.03	0.03	$\overline{}$	$\overline{}$	$\overline{}$	$\overline{}$		

The relatively low percentage of alumina $(A1₂O₃)$ is probably related to the clay minerals and feldspars. Low CaO, MgO, $K₂O$ and $Na₂O$ suggest the clays are most probably nonswelling clays. According to (Millot, 1970), the composition suggests that the clay samples are hydrated siliceous aluminosilicates.

While the XRF result (Table. 3), indicates relative abundance of the analyzed trace elements; Sr, Ba, Cu, Ni, Pb, Co, Cd, Zn, Cr and Zn in the clay samples. The clay samples all have very low concentrations. The probable reason for this depletion is due to the low abundance in the parent rock.

4.3. Provenance and Tectonic Settings

Numerous scholars have shown that paleo-environment to a large extent, determine both chemical and mineralogical characteristics of clays (Keller, 1970; Singer and Stoffel, 1980). According to the $Al_2O_3-Fe_2O_3-SiO_2$ ternary diagram of Schellmann (1986); the clay samples generally define a trend with gradual change from Al_2O_3 -rich to SiO_2 -rich compositions indicating that the Clay deposits are products of weak laterization (Fig. 3A).

While the discriminant plot (Fig. 3B) displays that the samples plot mainly in the intermediate igneous province field. These characteristics indicate that the original source area was both felsic and mafic, and the negative anomaly is regarded as evidence for a differentiated source, similar to granite (McLennan, 1989; Taylor and McLennan, 1985; 1995). Plate tectonic processes impact distinctive mineralogical and geochemical signatures to sediments and as such clastic sedimentary rocks and sedimentary basins can be classified according to plate tectonic settings (Roser and Korsch, 1986).

The $SiO₂$ versus $K₂O/Na₂O$ diagram suggested that the clay deposit in the study area were deposited between passive and continental Island Arc margins (Fig. 3C). Ekosse (2001) suggested that the ratio of $TiO₂/Al₂O₃$ as a province indicator. From figure 3D, the plot indicates provenance of materials from predominantly granite-rhyolite region.

4.4. Weathering Effect

As demonstrated by Nesbit et al. (1982), a measure of the degree of chemical weathering alteration of sediments was constrained by calculating the chemical index of alteration (CIA): CIA = Molars $[A_2O_3/(Al_2O_3 + CaO^* + Na_2O + K_2O)]$ *100; while the chemical index of weathering (CIW) expression $[A_2O_3/(Al_2O_3+CaO^*+Na_2O)]$ *100 proposed by Harnois (1988) was used in estimating CIW. The CIA is based on the assumption that the dominant process during chemical weathering is the degradation of feldspar and the formation of clay minerals.

Estimated values of CIA (85.81–98.95) and CIW (86.06– 99.22), Table 2 of the clay samples suggest extreme silicate weathering (Fig. 4). The values are also indicative of low to depleted oxides particularly those of K, Ca, and Na in the clay samples.

4.5. Industrial Significance

The clay samples from the study area fall within section 1 (Fig. 5A), which belongs to the commercial ball clays group. While Fig. 5B, depicts the clay samples from the study area belong to the kaolinite group. The industrial potential of the clay deposits from the study area as potential raw materials was achieved by a comparative analysis of the chemical composition of the clay samples in the study area with those of some reference industrial specification clays (Table 4) and clays in other areas (Table 5).

Table 5. Comparison of the average chemical composition of the studied clays with average chemical composition with other clay facies

I: Okija Clay (Anambra state) Onyeobi et al., (2013), II: China Clay GTY (Huber, 1985), III: Kutigi Clay (Niger state) Akhirevbulu et al., (2011), IV: Average Clay-shale (Pettijohn, 1957) AVCS, V: Iyuku Clay (Edo state) Onyeobi et al., (2013), VI: Plastic fire Clay St Louis Huber, (1985) PFC

 It was observed that the average chemical compositions of the studied clay deposits were above the industrial specification of clays (Table 4). Also, the clay deposits when

compared with other clay deposits (Table 5) as reported by Onyeobi et al., (2013); Huber (1985); Akhirevbulu et al. (2011); (Pettijohn 1957); Onyeobi et al. (2013); and Huber (1985); was observed to be above the chemical composition of the referenced clay deposits (Table 5). The analyses thus indicated that the clay deposits in the study area are industrially insignificant.

4.6. Physical Properties

The particle size distribution results (Table 6), indicate that the samples contain high amounts of sand-sized particles ranging from 36.19 % to 50.24 %. Generally, grain size

distribution affects the plasticity and stability of clay and its industrial applications.

However, higher clay content increases the stability and plasticity. In Table 6, the average percentage of silt and claysized materials ranges from 27.13 % to 42.8 % and 18.01 % to 24.3 % respectively. Based on the percentage of clay, silt and sand, the textural classification of clays in the study areas are classified as sandy clay.

Table 7. Geotechnical characteristics of Ogiso and Okhoro clay deposit outcrops

Clay	Moisture	Specific Gravity	Linear Shrinkage	Swelling	Atterberg Limit		
samples	Content $(\%)$		$\binom{0}{0}$	$\binom{0}{0}$	LL	PL	PI
$OG-1$	21.56	2.45	12.56	41.3	-	-	-
$OG-2$	21.96	2.45	12.01	39.48	56.99	20.62	36.37
$OG-3$	21.38	2.43	11.69	38.43	47.21	15.76	31.45
$OG-4$	17.95	2.04	11.96	39.32	53.99	20.19	33.8
$OG-5$	21.29	2.42	12.23	40.21	55.99	14.98	41.01
$OG-6$	21.38	2.43	11.55	37.97	52	14.11	37.89
$OG-7$	21.47	2.44	12.87	42.31	56	20.85	35.15
OKH-1	19.71	2.24	11.01	36.2	52.99	21.5	31.49
OKH-2	19.62	2.23	12.76	41.95	56.97	15.48	41.49
OKH-3	19.92	2.24	12.54	41.23	62	20.62	41.38

The result of Atterberg tests of the clay samples from the study areas are shown in Table 7, which shows the liquid limit ranging from 47.21–62 %; plastic limit 14.11–21.5 %; and plasticity index 31.45–41.49 %. The clay samples have moderate-high liquid limit values (47.21–62 %) and low plastic limit $(14.11-21.5 \%)$. This is due to the high sand content $(36.19-50.24\%)$ in the clay samples.

Fig. 6. Plasticity chart for classification of cohesive soils (after Casagrande, 1948)

Table 7 shows the liquid limit is greater than the plastic limit. When the value of plastic limit is not close to that of liquid limit, the soil is said to be non-plastic. From the plasticity chart (Fig. 6), the plasticity of the clay samples falls in the low-plastic to medium-plastic region, an indication of high sand-sized fraction and silica content indicated in Table 6 and 2, respectively. The clay samples moisture content and swelling capacity indicated in Table 7, ranges from 17.95– 21.96 %; and 36.2–42.31 % respectively. This suggests that

the clay samples have moderate moisture content (moderate pliability and mouldability) and a relatively high swelling capacity (high expansion and water absorption).

While the linear shrinkage and specific gravity of the clays to possess values of ranging from 11.01–12.87 %; and 2.04–2.45 $g/cm³$ respectively (Table 7). The relatively high linear shrinkage and low specific gravity values are less desirable for structural applications due to potential deformation, while their low density can affect their load-bearing capacity, which may result in cracking and settlement in buildings.

5. Conclusion

The clay deposit in the study areas was found to comprise mainly of alumina, silica and iron oxides with trace amount of other oxides. They are also classified as commercial ball clays and as such are useful in the production process of ceramics. They are product of weak laterization. The clay deposits were deposited between passive and continental Island Arc margins and indicated low to medium plasticity. From the analysis and interpretations of the clay samples, it indicates that white clays are high quality kaolinite clays that have wide range of industrial applications, ceramics, pharmaceuticals, etc. The grain size analysis revealed that the clay deposits in the study areas are sandy clays possessing moderate to high liquid limit and low plastic limit, low to medium plasticity, moderate pliability and mouldability, and relatively high expansion and water absorption capacity.

6. Recommendations

The clay deposit in the study area should undergo beneficiation to make them useful for other industrial purposes. The ball clay can also be used for cosmetic purposes, but it is advised that the clays be properly blended, beneficiated with lime and refining processes since the aforementioned processes will overcome compositional deficiencies.

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The views expressed in this paper reflect the collective experiences of the authors gained through the research. The authors may have undoubtedly failed to cite some vital concepts. We take sole responsibility for any omission or bias.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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