Compositional Characteristics and Industrial Assessment of Some Clay and Shale Deposits in Okigwe and Environs, South Eastern Nigeria

Mamamu, L.O, Okeke, O.C, Fagorite, V.I, Popoola, J.O, Okonkwo, S.I, Adakwa, C.B. and Onwunaonyeshi, G.A.
Department of Geology School of Physical Sciences,
Federal University of Technology, P.M.B 1526, Owerri, Nigeria
E-mail: lima4oscar@gmail.com

D.O.I: 10.56201/ijgem.v9.no6.2023.pg91.107

Abstract
This study assessed the industrial potential of clays and shales in the Okigwe area, Southeastern Nigeria. The soil investigation involved field study and laboratory analyses, collecting samples from various geological formations. A total of 5 samples were collected from clay and shale deposits at various depths, including Imo shale, Nsukka, Mamu, and Ezeaku Formations, in the field. The samples were examined and packed in polythene bags for laboratory analysis, which included tests for geotechnical index properties, chemical and mineralogical compositions. For Atterberg limits, the values of LL for the five samples (Ihube, Leru, Amuro, Lokpaukwu and Umulolo) were 35.01, 53.50, 44.11, 42.10 and 46.00 % respectively indicating Leru shale with the highest value and Ihube shale as the least.PL was 21.60, 29.45, 25.29, 27.40 and 23.40 % at the five locations respectively also indicating Leru shale with the highest value and Ihube shale as the least. While the PI were 13.41, 24.05, 21.22, 23.11 and 22.00 %. The values of linear shrinkage for the five samples were 4.10, 7.20, 9.13, 8.25 and 9.17 %. While the values for net moisture contents were 23.11, 20.12, 23.25, 20.14 and 22.81 %.These indicate that the clay samples meet specifications for industrial usage, providing valuable insights into their geotechnical properties for material selection and the design of safe and stable structures in industries such as construction, geotechnical engineering, mining, and waste management. Chemical analysis identifies silicon and aluminum oxides as the dominant components in both shale and clay samples, with other oxides being negligible. It was observed that the values of five samples were 49.47, 50.67, 53.15, 52.45 and 61.67 % of SiO_2 and Al_2O_3 was 18.48, 15.29, 14.03, 19.52 and 6.94 %. Mineralogical analysis reveals quartz as the predominant mineral in the clay deposits, followed by Montmorillonite and kaolinite. Based on the chemical and mineralogical specifications, it is inferred that the Leru shale and Umulolo clay deposits hold significant potential for various industrial applications, including paper production, paints, ceramics, textiles, and agriculture, particularly as fertilizers. The analysis of the chemical and mineralogical compositions of the clay-shale formation reveals its potential as a valuable source of raw materials for low-grade refractory products. However, the quality of these refractory materials can be enhanced through specific refining procedures, thereby improving their suitability for industrial use and sustainable development.

Keywords: *Clays, Shales, Geotechnical, Mineralogical, Geochemical, and Industrial Potential*

1.0 Introduction

Clay is often (but not always) an ultrafine-grained material with a particle size of less than 2 m according to conventional particle size categorization (Onyekuru et al, 2018). They are distinguished by sheet-like silicate structures with composite layers stacked along the c-axis (Grim, 1968). Thus, the word can refer to both materials with particle sizes smaller than 2 m and a family of minerals with comparable chemical compositions and crystal structure features (Velde, 1995; Onyekuru et al., 2018). Clay minerals are the result of long periods of progressive chemical weathering of pre-existing rocks (typically silicate-bearing) by low concentrations of carbonic acid and other diluted solvents, notably in the world's warm tropical and subtropical climates.

For shale it has been defined as a fine-grained, clastic sedimentary rock composed of mud that is a mixture of flakes of clay minerals and tiny fragments of other minerals especially quartz and calcite (Ayajuru et al, 2019; Oyedele et al, 2018). "Shale" is clay that has been hardened by natural geologic processes, although when ground and moistened it will in many instances become plastic (Hamilton and Babet, 1975). There are some characteristics of clay and shale such as plasticity, chemical makeup, refractoriness, strength and shrinkage that determine its suitability for several industries (Eyankware et al, 2021). These characteristics has been widely used in several studies.

Clays are important raw materials for modern and cottage industries. However, in Nigeria clay deposits have not been adequately utilized considering the quality and quantity that occur all over the country. Underutilization of the clay deposits may be attributed to insufficient geological information on the assessment of the properties of the various clay deposits on one hand and shallow knowledge of what each deposit could be used for on the other hand (Okunlola, 2008).

From the review studies, it is indicative that numerous studies have been carried out on clay minerals, especially its mineralogical and chemical characteristics within the country and the study area. However, for shale few studies have been done to evaluate its mineralogical and chemical properties and usefulness for industrial purposes. Hence, there is a gap in research for the potential industrial usage of shale and the methods to investigate it as well.

It is important therefore that investigations of clay deposits occurring all over Nigeria particularly the study area be carried out to assess their suitability for various industrial purposes so as to conserve the scarce foreign exchange.

The industrial utilization of a clay deposit depends on its geological, mineralogical, chemical and physical properties. The assessment of economic potentialities of a clay body must involve the evaluation of the parameters (Attah and Oden, 2010). In addition to this, assessment of industrial minerals occurring in Nigeria will contribute to geo-database that could assist potential investors. The end result will be creation of employment. The present study is therefore a contribution to the evaluation of clay and shale deposit within the study area.

This study focused on the mineralogical (major minerals) and chemical (major elements and oxides) characteristics using the X-ray Diffraction (XRD) and X-Ray Fluorescence (XRF) as experimental methods. The geotechnical properties (Particle size analysis, Atterberg limits, and linear Shrinkage) were analyzed using standard methods.

2.0 Location and Geology of the Study Area

The selected localities for the study are bounded by Latitudes 5°53' and 6°30' N and Longitudes 6°56' and 7°30' E. Within this area are Uturu, Ikpankwu, Ezinnachi and Ohiya in the Okigwe-Umuahia axis. The area is characterized by undulating topography. As a result of differential weathering and erosion arising from resistant variations of the underlying rocks, the area and its environs aredotted by escarpments, cuesta, and asymmetrical ridges (Ayajuru et al., 2019). The area is largely drained by Imo River, with drainage patterns that are dendritic and radial. The area has a tropical climate composed of two alternating seasons-rainy and dry seasons.

Geologically, Okigwe lies within the Anambra Basin. The basin constitutes a major depocentre of clastic sediments in the southern portion of the Benue Trough (Nwajide, 2005). The outcropping geologic formations in the area are Ajali, Mamu and Nsukka Formations. Soils in the area are derived from the false bedded sandstones or Ajali Formation. Nsukka and Mamu Formations are parallic, that is, consists of sand and shale sequences.

Table 1: Generalized stratigraphic sequence in Okigwe area (modified from Reyment, 1965, Mode, 2004 and Ofoegbu, 1985).

Age	Formation	Lithological Characteristics
Paleocene (55-65 m.y.)	Imo Formation (Imo Shale)	Blue to dark grey shales and subordinate sandstone member (Umuna and Ebenebe sandstone).
Maastrichtian (65-68 m.y.)	Nsukka Formation	Alternating sequence of sandstone and shale with coal seams.
Maastrichtian (65-68 m.y.)	Ajali Formation	Friable sandstone with cross bedding. Alternating sequence of sandstone, siltstone, shale and claystone with coal seams.
	Mamu Formation	
Campanian (68-78 m.y.)	Nkporo Formation (Enugu Shale)	Shale and mudstone with sandstone lenses.

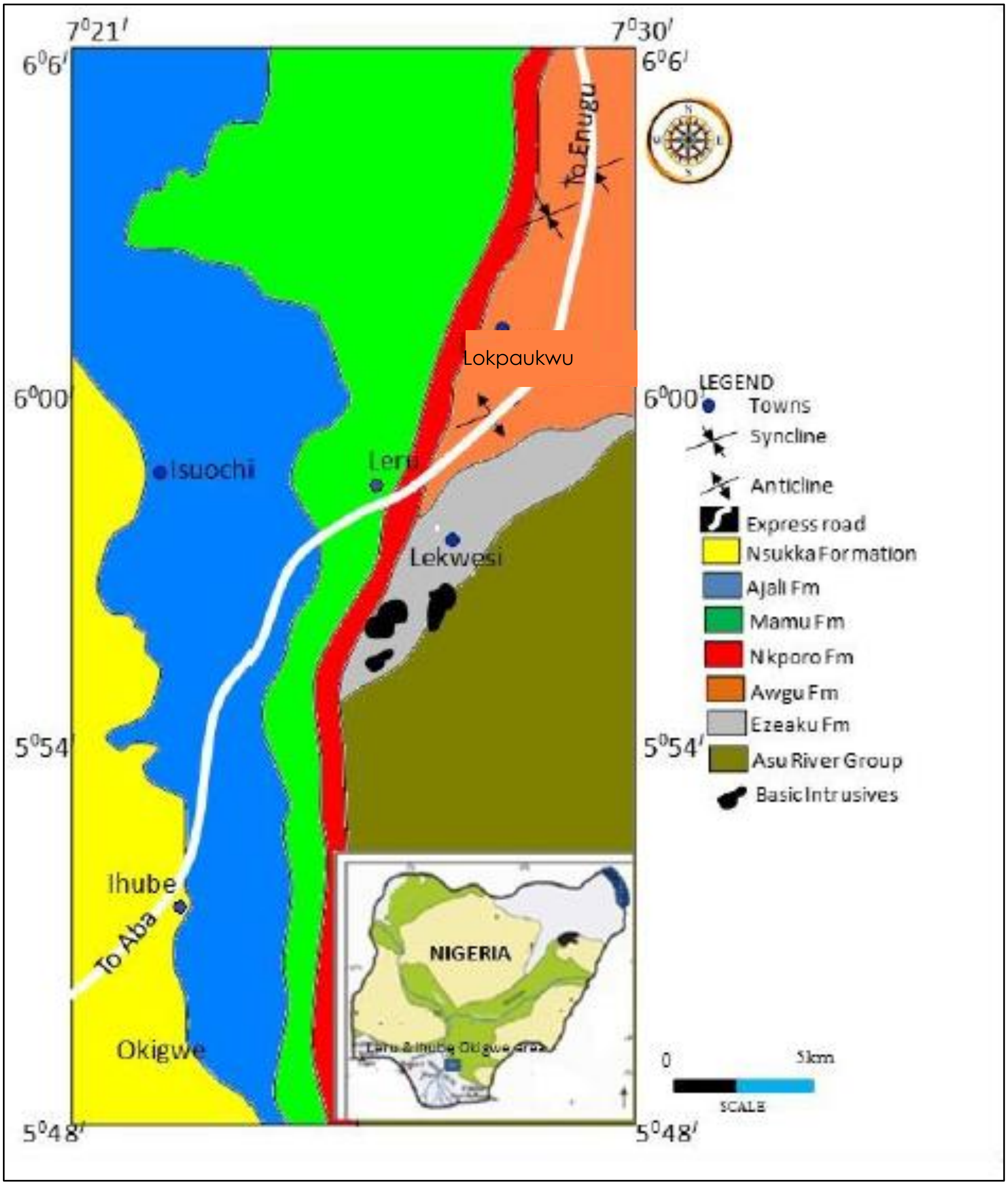

Figure 1: Geologic map of the study area (adapted from Dikeogu et al., 2018; Ayajuru et al., 2019)

Materials and Methods

1.0 Materials

A desk study was carried out as the first stage, which involved the review of previous and related literature, as well as relevant information pertaining to the topic. The second stage involved a reconnaissance survey, followed by a detailed geological mapping. Subsequently, a total of five (5) samples were collected from the field for close examination. The sample collection process utilized a topographic map to identify and demarcate the study area, employing tools such as:

i. *Location map*
ii. *Geologic map*
iii. A *shovel*
iv. A *hand lens*.
v. A *compass clinometer*
vi. *Sample bags, Field note book*
vii. *The Global Positioning System (GPS)*
viii. *Hand Auger*

The third stage in the data acquisition involved the laboratory analysis of the samples that were collected from the field. The soil samples were subjected to a number of laboratory tests, including geotechnical analysis (grain size distribution, compaction test, and Atterberg limits), as well as mineralogical and chemical analyses.

3.2 Methods

3.2.1 Sample Collection and Sampling Procedure

The soil investigation, which comprised the field study and laboratory analyses, was undertaken. The field investigation for clay and shale data collection involved sampling and measurement in the study area. Samples were collected from the clay and shale deposits at different depths, ranging from 2 meters to 20 meters. The techniques for sample collection followed those of Okengwo et al. (2015). Subsequently, a total of five (5) samples were collected from the four geological formations (Imo shale, Nsukka, Mamu, and Ezeaku Formations) in the field. These samples were closely examined and packed in polythene bags for laboratory analysis.

Table 2: Sampling Points

S/No.	Sampling Point	Coordinates	Elevation
1.	Nsukka Fm. (Ihube Shale)	N 05' 51". E 007' 20"	509 ft.
2.	Mamu Fm. (Leru Shale)	N 05' 44". E 007' 14".	397ft.
3.	Imo shale. (Amuro Clay)	N 05' 44". E 007' 19"	32.4ft.
4.	Ezeaku Fm. (Lokpaukwu Clay)	N 05' 32". E 007' 12"	65.5 ft.

5.	Nsukka Fm. (Umulolo Clay)	N 05' 49''. E 007'. 20''	42.6ft.

3.2.2 Laboratory Analysis

Geotechnical, chemical and mineralogical analysis of the Nigeria geological survey agency, Kaduna were carried-out on the five samples collected from the field and examine for their colour, texture and other physical properties.

i. **Geotechnical index properties (atterberg Limits, Liner shrinkage etc):** They are basic measure used as an integral parts of several engineering classification systems to characterize the fine-grained fractions of soils and to specify the fine-grained fraction of constriction materials and are used either individually or together, with other soil properties to correlate with engineering behaviour. As a dry soil takes on increasing amounts of water, it undergoes dramatic and distinct changes in bahaviour and consistency. From the analysis, it implies that the Leru shale and Umulolo clay deposit hold significant potentials for various industrial applications such as paper production, paint, ceramics and agriculture, particular as fertilizers.

Figure 2: Weighing pans used in Atterberg Limit Tests

Figure 3: Apparatus used in the Liquid Limit Test

ii. **Geochemical analysis:** This is to determine the chemical composition (major oxides and trace element) in the clay and shale sample, using the x-ray fluorescence (XRF). From the analysis it was observed that the dominant oxides present in the five (5) samples are SiO_2 and Al_2O_3.

iii. **Mineralogical analysis**: It is used to ascertain the characteristics of major minerals, using x-ray diffraction (XRD), the major mineral found in the five (5) samples was quartz, followed by Montmorillonite and Kaolinite, however other minerals were negligible.

4.0 Results and Discussion

4.1 Results

The results of Geotechnical Index Properties, chemical analysis and mineralogical composition of the samples obtained are presented in tables below

4.1.1 Geotechnical Index Properties of clay and shale

Table 3 below shows the geotechnical index properties for both clay and shale respectively from five geologic formations.

Table 3: Geotechnical Index Properties of clay and shale from Geologic formations in Okigwe area

Geotechnical Index Properties	Nsukka Fm. (Ihube Shale)	Mamu Fm. (Leru Shale)	Imo shale. (Amuro Clay)	Ezeaku Fm. (Lokpaukwu Clay)	Nsukka Fm. (Umulolo Clay)
Atterberg Limits					
Liquid Limit (LL) (%)	35.01	53.50	44.11	42.10	46.00
Plastic Limit (PL) (%)	21.60	29.45	25.29	27.40	23.40
Plasticity Index (PI) (%)	13.41	24.05	21.22	23.11	22.00
Linear Shrinkage (%)	4.10	7.20	9.13	8.25	9.57
Net Moisture Contents (%)	23.11	20.13	23.25	20.14	22.81
Specific Gravity (%)	2.55	2.12	2.29	2.21	2.57
Clay Fraction (%)	23.10	21.12	23.11	22.33	24.56
Activity of clay	8.12	9.15	9.86	8.13	9.92
Dry Density (Mg/m^3)	1.34	1.12	1.19	1.12	1.40

(a) Atterberg Limits

The liquid limit represents the moisture content at which a soil transitions from a liquid-like state to a plastic state. It indicates the soil's ability to flow under loading conditions (Tang et al., 2016). The values of LL for the five samples (Ihube, Leru, Amuro, Lokpaukwu and Umulolo) were 35.01, 53.50, 44.11, 42.10 and 46.00 % respectively indicating Leru shale with the highest value and Ihube shale as the least.

The plastic limit represents the moisture content at which a soil ceases to behave plastically and starts to exhibit brittle behavior (Stirling et al., 2015). It indicates the lower limit of plasticity of the soil. PL was 21.60, 29.45, 25.29, 27.40 and 23.40 % at the five locations respectively also indicating Leru shale with the highest value and Ihube shale as the least.

(b) Linear Shrinkage and Net Moisture Contents

The linear shrinkage represents the moisture content at which a soil undergoes significant volume reduction upon further drying (Romero, 2013). It indicates the soil's tendency to shrink and crack when subjected to drying. The values of linear shrinkage for the five samples (Ihube, Leru, Amuro, Lokpaukwu and Umulolo) were 4.10, 7.20, 9.13, 8.25 and 9.17 % respectively indicating Umulolo clay sample as the highest value and Ihube shale as the least. While the values for net moisture contents were 23.11, 20.12, 23.25, 20.14 and 22.81 % respectively indicating Amuro clay with the highest value and Leru shale the least. The net moisture content refers to the amount of water present in the clay or shale after accounting for any free or absorbed water (Zhang et al., 2020). This moisture content can affect the physical and mechanical properties of clay and shale, thereby influencing their suitability for different industrial purposes.

(c) Specific Gravity and Clay Fraction

Specific gravity values of the five samples were 2.55, 2.12, 2.29, 2.21 and 2.15 % respectively with Umulolo clay as the highest and Leru shale the least. While the clay fraction was 23.10, 21.12, 23.11, 2.33 and 24.56 % respectively indicating Umulolo clay as the highest and Leru shale as the least. The presence and characteristics of the clay fraction influence the behavior, properties, and suitability of clay and shale for different applications (Kumari and Mohan, 2021).

(d) Activity of clay and Dry Density

Clay activity refers to the ability of clay particles to undergo changes in volume and plasticity when in contact with water (Wagner, 2023).

The activity of clay values of the samples were 8.12, 9.15, 9.86, 8.13 and 9.92 respectively at the locations sampled indicating Umulolo clay as the highest value and Ihube shale as the least.

For the dry density, the values at the five locations were 1.34, 1.12, 1.19, 1.12 and 1.40 Mg/m^3 respectively revealing Umulolo as the highest and Leru shale and Lokpaukwu clay as the least.

4.1.2 Chemical Composition of clay and Shale

Table 4 shows the chemical compositions (major oxides and trace elements) in the clay and shale samples (Figure 4.1 and 4.2). The results indicate that the dominant oxides present in the shale and clay samples are the oxides of silicon and aluminum. As can be deduced from the table, the samples from Ihube, Leru, Amuro, Lokpaukwu and Umulolo contains 49.47, 50.67, 53.15, 52.45 and 61.67 % of SiO_2 respectively with Umulolo with the highest concentration and Ihube the least. While Al_2O_3 was 18.48, 15.29, 14.03, 19.52 and 6.94 % respectively with Lokpaukwu as the highest and Umulolo the least concentration. Apart from the dominant oxides, the percentage differences in composition of other oxides found in the samples are negligible.

Table 4: Chemical composition of clay and shale from Geologic formations in Okigwe area

Major Oxides (%)	Nsukka Fm. (Ihube Shale)	Mamu Fm. (Leru Shale)	Imo shale. (Amuro Clay)	Ezeaku Fm. (Lokpaukwu Clay)	Nsukka Fm. (Umulolo Clay)
SiO_2	49.47	50.67	53.15	52.45	61.67
TiO_2	0.98	1.03	0.56	0.38	0.75
Al_2O_3	18.48	15.29	14.03	19.52	6.94
Fe_2O_3	5.89	6.22	10.93	9.84	5.54
CaO	4.12	6.41	1.08	0.39	6.00
MgO	4.68	2.30	1.74	2.36	0.70
MnO	0.07	0.04	0.61	0.34	0.53
K_2O	1.19	1.02	0.75	0.51	2.96
Na_2O	0.08	0.05	1.22	0.83	1.54
P_2O_5	0.26	0.01	0.02	0.01	1.20
SO_3	0.41	0.70	0.01	-	0.02

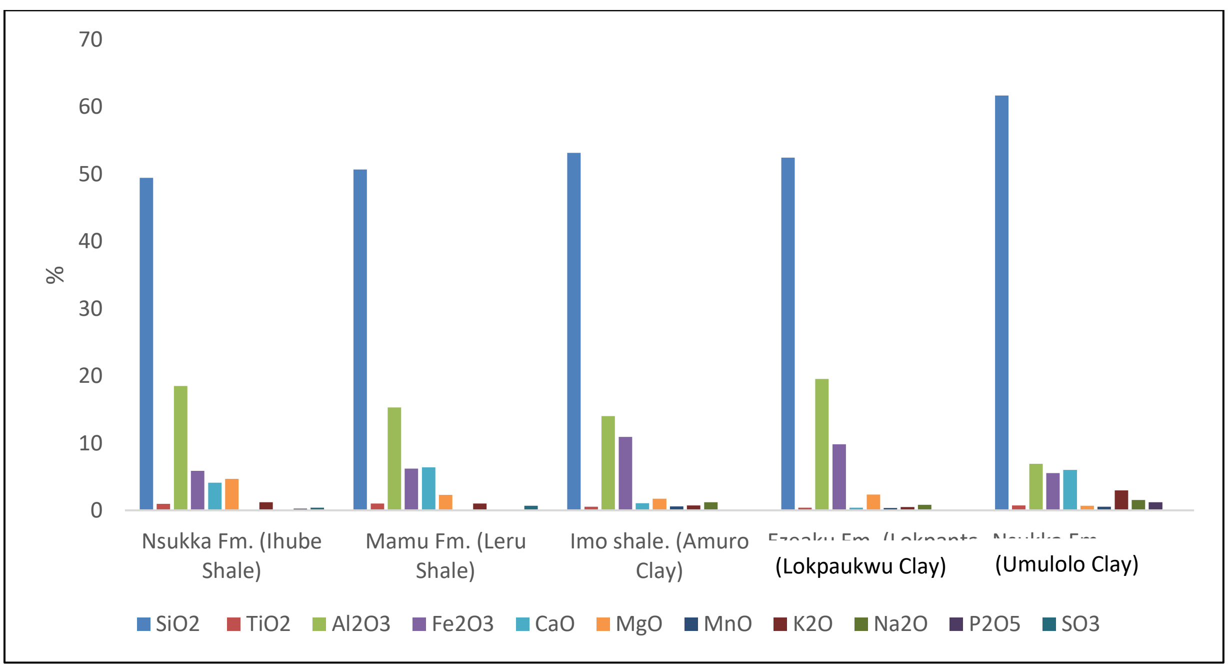

Figure 4: Major oxides of clay and shale from Geologic formations in Okigwe area

Mineralogical Composition of clay and shale

Table 5 shows the mineralogical composition of the shales and clay based on X-ray diffraction (Appendix section). Here the major occurrence was Quartz with composition of 28.98, 31.22, 17.88, 15.87 and 12.92 % respectively at Ihube, Leru, Amuro, Lokpaukwu and Umulolo (Figure 4.3). Quartz was identified in all the samples indicating that it is the dominant mineral in the clay deposits in the study area. Its high dominance clearly explained its grittiness and suggestive that the clays could be of residual origin (Akhirevbulu et al., 2010). Followed by Montmorillonite with composition of 17.20, 17.36, 16.67, 18.11 and 19.02 % respectively and kaolinite with 14.12, 16.8, 19.89, 20.54 and 22.93 % respectively (Table 4.3 and Figure 4.3). While other compositions were negligible.

Table 5: Mineralogical Composition of clays and shales from Geologic formations in Okigwe area

Mineralogical Composition	Nsukka Fm. (Ihube Shale)	Mamu Fm. (Leru Shale)	Imo shale. (Amuro Clay)	Ezeaku Fm. (Lokpaukwu Clay)	Nsukka Fm. (Umulolo Clay)
Quartz (%)	28.98	31.22	17.88	15.87	12.92
Montmorillonite (%)	17.20	17.36	16.67	18.11	19.02
Kaolinite (%)	14.12	16.8	19.89	20.54	22.93
Illite (%)	4.20	5.66	3.59	3.44	4.40
Chlorites (%)	ND	ND	22.20	25.89	26.23
Hallarysite (%)	ND	ND	13.01	12.08	12.11

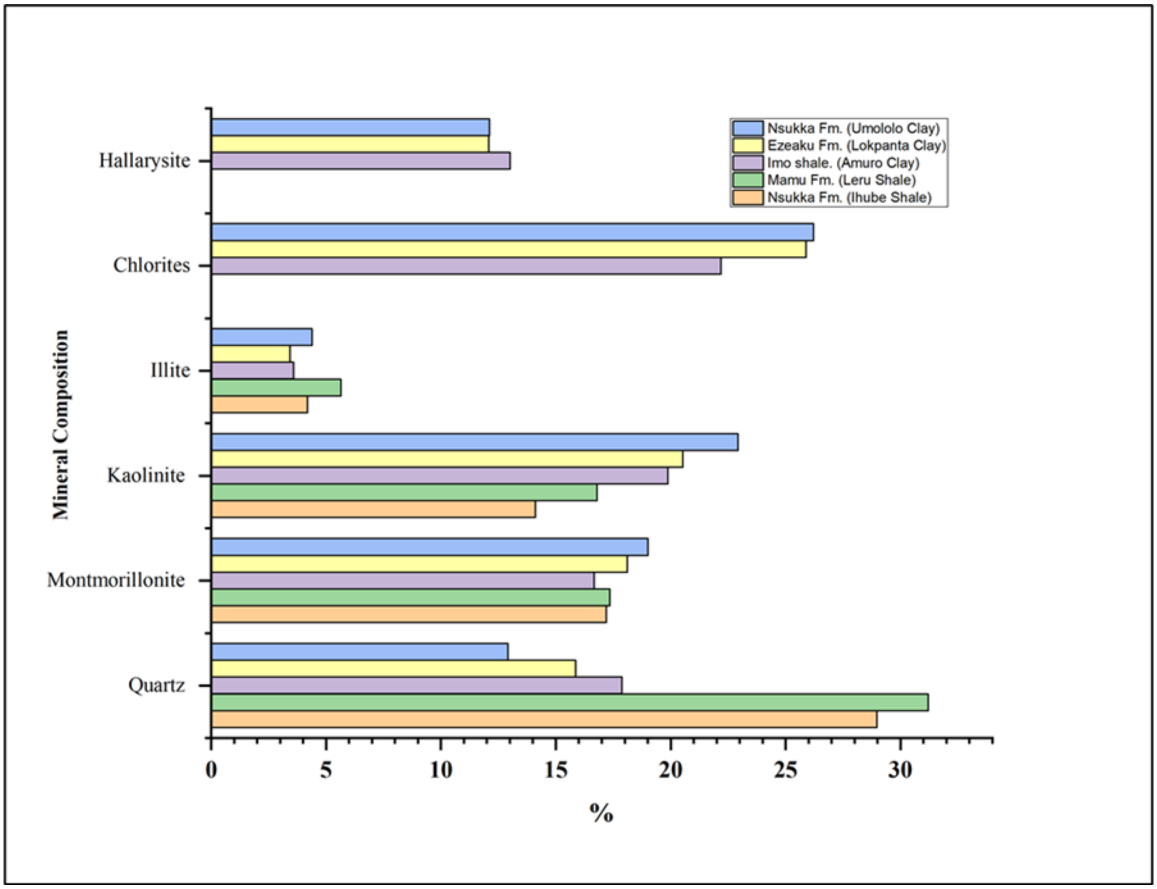

Figure 5: Mineralogical Composition of clays and shales from Geologic formations in Okigwe area

4.2 Discussion

Implication of geotechnical properties and mineralogical composition of clay and shale for industrial purposes

The presence of SiO_2 and Al_2O_3 in the samples distinctly identifies the deposits as hydrated alumino-silicates with contamination from unbound silty quartz. Table 4.4 displays the chemical characteristics of certain industrial clays and shales, which served as the foundation for evaluating the industrial capabilities of the clay and shale deposits.

Table 6: Chemical specification of some industrial clays and shales (Adapted from Okeke et al., 2001; Ayajuru et al., 2019)

Major Oxides (%)	(i)	(ii)	(iii)	(iv)	(v)	(vi)	(vii)
SiO_2	47.0	45.78-47.90	45.80-45.90	45.00	67.50	38.00	46.07
Al_2O_3	40.0	37.90-38.40	33.50-36.10	38.10	26.50	9.45	38.07
Fe_2O_3	-	13.40-13.80	0.30-0.60	0.60	0.50-1.20	2.70	0.33
TiO_2	-	1.35-1.75	0.00-1.70	1.70	1.10-1.0	-	0.50
Na_2O	-	0.20-0.30	0.00-1.60	-	0.20-1.50	2.76	0.27
K_2O	-	0.10-0.40	0.00-1.60	-	1.10-0.10	2.76	0.43
CaO	-	0.25-0.30	0.00-0.50	-	0.18-0.30	15.85	0.38
MgO	-	0.20-0.30	-	0.10-0.19	0.20-0.70	-	-
H_2O^+	13.0	-	12.00-14.70	14.70	12.00	3.04	13.47

Taking into account, the SiO_2 and Al_2O_3 contents of the samples and in reference to the chemical specification of some industrial clays and shales as presented in table 4.4. Except for the Umulolo clays which had a low Al_2O_3 composition, Ihube, Leru, Amuro and Lokpaukwu clay and shale deposits are very good raw materials for brick clay.

Economic Evaluation

Analysis of the chemical compositions of the clay-shale formation has revealed its potential as a valuable reservoir of raw materials for the production of low-grade refractory products. Nevertheless, the quality of these refractory materials can be enhanced by implementing certain refining procedures. Specifically, the raw material can be subjected to a screening process to eliminate the presence of silica in the form of quartz and other non-clay fractions. Moreover, additional improvements can be achieved through techniques such as acid bleaching or magnetic reduction to eliminate iron oxide and titanium impurities (Ianicelli, 1971; Hughes, 1982; Okunlola and Egbulem, 2015).

5.0 Conclusion and Recommendations

5.1 Conclusion

Geotechnical index properties, mineralogical and chemical parameters of clays and shales have been assessed and used in this study to ascertain the industrial potentials of shales and clays of some part of Okigwe and its environs, Southeastern Nigeria. Geotechnical index properties (atterberg limits, linear shrinkage, net moisture contents, specific gravity and clay fraction, activity of clay and dry density) revealed that the clay were within specifications for industrial usage and applications. These has further granted insight in terms of understanding the geotechnical properties of clays and shales which is crucial for selecting suitable materials, designing safe and stable structures, and industrial applications such as construction, geotechnical engineering, mining, and waste management.

The mineralogical analysis identified Quartz in all the samples indicating it as the dominant mineral in the clay deposits of the study area, followed by Montmorillonite and kaolinite, while others were negligible. The chemical analysis showed that the dominant oxides present in the shale and clay samples are the oxides of silicon and aluminum. While the composition of other oxides found in the samples were negligible.

Analysis of the chemical and mineralogical compositions of the clay-shale formation has revealed its potential as a valuable reservoir of raw materials for the production of low-grade refractory products. Nevertheless, the quality of these refractory materials can be enhanced by implementing certain refining procedures.

5.2 Recommendations

In the light of the above findings, the following are some suggestions from this study;

1. The local industries in the region should actively exploit the clay and shale resources to significantly enhance economic production.
2. While the results obtained from the examined samples demonstrate a wide range of industrial applications, they generally do not match the quality of commercially available counterparts from Europe and Asia. Therefore, it is recommended to establish a comprehensive clay mineral dressing and preparation plant, implement remediation processes through appropriate blending, beneficiation with lime, and refining techniques. It is anticipated that these processing methods will address compositional deficiencies.
3. Specifically, the raw material can undergo a screening procedure to eliminate the presence of silica, such as quartz and other non-clay fractions, in order to improve the quality of refractory materials.
4. Additionally, further enhancements can be achieved by employing techniques like acid bleaching or magnetic reduction to eliminate impurities of iron oxide and titanium.
5. Ultimately, it is essential to establish government policies and frameworks to guide the sustainable development and utilization of these resources within the region and the country as a whole.

Acknowledgments

The Authors are grateful to Mr. Gerald Onwu of Geology Department, FUTO, for his assistance in sample collection; and to staff of Nigerian Geological Survey Agency (NGSA) Laboratory, Kaduna, for their assistance in the analysis of the samples.

References

Akhirevbulu, O. E., Amadasun, C. V., Ogunbajo, M. I., and Ujuanbi, O. (2010). The Geology and Mineralogy of Clay Occurrences Around Kutigi Central Bida Basin, Nigeria. *Ethiopian Journal of Environmental Studies and Management*, *3*(3).

Attah, L. E., and Oden, M. I. (2010). Physico-chemical properties and industrial potential of some clay deposits in Calabar area, south eastern Nigeria. *Global Journal of Environmental Sciences*, *9*(1and2), 39-49.

Ayajuru, N.C., Okeke, O.C., Ikoro, D.O., and Dikeogu, T.C. (2019). Geochemical, Mineralogical and Industrial Potentials of Shale at Okigwe Area, Southeastern Nigeria. *International Journal of Advanced Academic Research Sciences*, *Technology and Engineering*, *5*(11), 34-45

Dikeogu, T.C., Okeke, O.C., Amadi, C.C., and Udeogu, C.J. (2019). Textural and Geochemical Characterization of Sandstone Sediments Associated with Nsukka Formation in Okigwe Area, Southeastern Nigeria. *International Journal of Research Publications*, 1.

Eyankware, M. O., Ogwah, C., and Ike, J. C. (2021). A Synoptic Review of Mineralogical and Chemical Characteristics of Clays in the Southern Part of Nigeria. *Research in Ecology*, *3*(2).

Hamilton, W.N., and Babet, P.H. (1975). Alberta Clays and Shales: Summary of Ceramic Properties. Alberta Research, *Economic Geology Report*, 3.

Hughes, J. C. (1982). High gradient magnetic separation of soil clays in Nigeria, Brazil and Columbia: The Interrelationship of Iron and Magnesium extraction by acid Ammonium Oxalate and Carbon. *Journal Soil Sci.*, *33*, 501-519.

Ianicelli, J. (1971). High extraction magnetic filtration of kaolin. *Clays Clay Miner., 24*, 64-68. http://dx.doi.org/10.1346/CCMN.1976.0240203

Kumari, N., and Mohan, C. (2021). Basics of clay minerals and their characteristic properties. *Clay Clay Miner, 24*, 1-29.

Nwajide, C.S. (2005). A guide to geological field trips to Anambra and related basins in Southeastern Nigeria. *Great AP Express Publishers Ltd*.

Okeke, O.C., Ofulume, A.B., and Ezemakam, C.I. (2001). Assessment of the Industrial Potential of Major Porter Clay deposit in Plateau State. Science Forum: *J. Pure and applied science*, *4* (1).66-76.

Okengwo, O.N., Okeke, O.C., Okereke, C.N., and Paschal, C.C. (2015). Geological and geotechnical studies of gully erosion at Ekwulobia, Oko and Nanka towns, southeastern Nigeria. *Electronic Journal of Geotechnical Engineering*, *20*, 113-122.

Okunlola, O.A. (2008). Compositional characteristics and functional Industrial application of Itakpe Clay Occurrence, Central Nigeria. *Eurp. Journal of Sc. Research*, *19*(3), 453-461.

Okunlola, O., and Egbulem, C. (2015). Geological setting, compositional and economic appraisal of clay-shale occurrence in Itu-Mbonuso/Iwere Area, South-Eastern Nigeria. *Journal of Geography and Geology*, *7*(1), 85-96.

Onyekuru, S.O., Iwuoha, P. O., Iwuagwu, C.J., Nwozor, K.K., and Opara, K.D. (2018). Mineralogical and geochemical properties of clay deposits in parts of Southeastern Nigeria. *International Journal of Physical Sciences*, *13*(14), 217-229.

Oyedele, E.O., Okeke, O.C., and Dike, E.C., (2018). Evaluation of Swelling and Slaking Characteristics of Shales from Okigwe Area, Southeastern Nigeria. *Journal of Scientific and Engineering Research*, 5(10), 222-230.

Reyment, R. A. (1965). *Aspects of the geology of Nigeria: the stratigraphy of the Cretaceous and Cenozoic deposits*. Ibadan university press, 133

Romero, E. (2013). A microstructural insight into compacted clayey soils and their hydraulic properties. *Engineering Geology*, *165*, 3-19.

Stirling, R. A., Hughes, P., Davie, C. T., and Glendinning, S. (2015). Tensile behaviour of unsaturated compacted clay soils—A direct assessment method. *Applied clay science*, *112*, 123-133.

Velde, B. (1995). *Origin and Mineralogy of clays*. New York, Springer Verlag pp. 8-44.

Zhang, Q., Fan, X., Chen, P., Ma, T., and Zeng, F. (2020). Geomechanical behaviors of shale after water absorption considering the combined effect of anisotropy and hydration. *Engineering Geology*, *269*, 105547.