

**THE GEOCHEMICAL AND MINERALOGICAL ANALYSIS OF CLAY SAMPLES
FROM UZEBBA AND ENVIRONS, OWAN WEST AREA OF SOUTHERN NIGERIA.**

BY

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PSC1809038

**DEPARTMENT OF GEOLOGY
FACULTY OF PHYSICAL SCIENCES
UNIVERSITY OF BENIN**

SEPTEMBER, 2023

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**A PROJECT WORK SUBMITTED TO THE DEPARTMENT OF GEOLOGY,
FACULTY OF PHYSICAL SCIENCES, UNIVERSITY OF BENIN, IN PARTIAL
FULFILMENT OF THE REQUIREMENT FOR THE AWARD OF A BACHELOR
OF SCIENCE DEGREE (B.Sc) IN GEOLOGY**

SEPTEMBER, 2023

CERTIFICATION

This is to certify that this project work was carried out by Etinosa Israel OKUNBOR with matriculation number PSC1809038 of the Department of Geology, University of Benin, Benin City.

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Date

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(Head of Department)

Date

DEDICATION

This work is dedicated to God Almighty for his love, care and guidance all through the course of this program.

ACKNOWLEDGEMENT

I am incredibly grateful to God Almighty for guiding me through my project's entire duration as well as the challenging time I spent pursuing my academic goals at the University of Benin.

My sincere thanks go to my supervisor Dr. (Mrs.) O. Odokuma-Alonge, and I appreciate the support and knowledge that she shared with me.

My sincere gratitude goes out to the Head of Department, Dr. S.A. Salami, whose fatherly advice was crucial to my achievement as well as his professional and moral contribution.

Special thanks go to my parents Mr. Amadin Monday Okunbor and Mrs. Patience Okunbor, my siblings for their love, care and guidance.

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ABSTRACT

Five (05) clay samples were obtained from various locations in Uzebba and its environs with the aim of ascertaining its mineralogical and geochemical compositions, qualifying the clay samples and determining its economic potentials. The results obtained showed the predominance of SiO₂ (62.23 to 77.48 wt.%), Al₂O₃ (13.50 to 18.88wt.%), Fe₂O₃ (0.83 to 13.83 wt.%), TiO₂ (2.15 to 3.05 wt.%), CaO (0.20 to 1.05wt.%) with traces of MnO (0.02 to 0.03 wt.%), P₂O₅ (0.03 to 0.23 wt.%) and K₂O(0.87-0.39wt.%), respectively. The mineralogical results showed that quartz was predominant in all the samples (51.00 to 64.00%) followed by Orthoclase (4.20 to 20.00%) and Kaolinite (11.00 to 16.80%). From the results obtained it shows that the Uzebba clay is kaolinitic in nature. The clay minerals found in the study area is suitable for ceramic, glass, paint, paper and refractory bricks production.

CHAPTER ONE

INTRODUCTION

1.1 OVERVIEW OF CLAY

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Clay is a type of fine-grained natural soil material containing clay minerals hydrous aluminium phyllosilicates, e.g. kaolinite, $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. Layer-typed aluminium silicates known as clay minerals are found in a wide variety of geologic deposits, terrestrial weathering settings, and marine sediments all over the world (Velde, 2020). Similar effects on the geochemical cycles of metals can be seen in depositional situations where clay minerals precipitate from seawater. Clay mineral swelling is a key factor in petroleum extraction in engineering contexts, acting as lubricants and as an industrial catalyst for the synthesis of numerous chemical compounds. There are three types of clay: surface clay, shale, and fireclay. The majority of bricks are formed from striped-mined surface-minable clays that are found close to the earth's surface. Clays that have been subjected to high pressures and subsequently become hardened are called shale. Fireclays are found at deeper formations and have more uniform physical and chemical properties. They can withstand higher temperatures and are used to produce firebricks for high temperature applications such as refractories (Semiz, 2017).

Several minerals can be found in clay that has been extracted. The colour might burn to a buff, crimson, or salmon depending on how much iron oxide is present. Despite having access to a wealth of natural resources, including coal, oil, tin, clay, and others, Nigeria's economy currently depends significantly on the importation of completed goods. The economy is significantly hampered by this reliance on the importation of goods from abroad whose raw resources are abundant in the nation.

Due to its wide range of applications, clay deposits significantly contribute to global economic growth (Ezomo, 2010). Clay and shale are the basic raw materials required for the manufacture of a wide variety of products, and they are abundantly found in the sedimentary basin of South-South Nigeria, including sections of Edo (Uzebba) and Delta State (Ezomo, 2010). Clay is widely utilized in household life to create earthenware, chinaware, cooking ware, decorations, porcelain, ornamental features, and plumbing elements. Because of its enormous number of uses, clay has endless potential. Clay is valuable in a variety of sectors due to the reliance on its chemical and physical properties (Okpiafoh, 2016).

Most of the clay minerals have been formed from a mixture of oxides or hydroxides and water at moderately low temperatures and pressures, with the probable exception of halloysite. In alumina-silica systems devoid of alkali or alkaline earths, kaolinite is more likely to develop. When potassium is introduced to such systems, Illite is produced. Depending on the concentration of magnesium added, either smectite or chlorite develops. If the reactants are combined extremely slowly and in a considerably diluted state, the clay minerals can be created at normal temperatures and pressures. Certain forms of clay minerals have also been created synthetically by applying chemical processes to clay minerals that cause partial structural alterations. Vermiculite can be created through a protracted process in which any hydrated alkali or alkaline earth cation exchanges places with the potassium of mica. Vermiculite or montmorillonite layers can be intercalated with hydroxide sheets to produce chloritic minerals. Ezomo (2012) stated that these alterations also have opposite reactions, which are known. It is possible to distinguish between neoformation, which suggests a method for the formation of minerals from solution, and transformation, which refers to a mechanism of mineral production including a change from one mineral to another.

1.2 AIM AND OBJECTIVES

1.2.1 AIM

The aim of this study is to carry out the geochemical and mineralogical analysis of clay from Uzebba and environs to determine their suitability for ceramic and refractory purposes.

1.2.2 OBJECTIVES

The objectives of this work are to;

1. Examine the geological features of the clay from the study area.
2. Determine the mineralogical properties of clay from the study area.
3. Present useful geochemical information of the study area
4. Qualify the clays and determine its economic potentials

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1.3 LOCATION OF STUDY AREA

Uzebba is a town in Owan Local Government of Edo State, Nigeria. Uzebba, along with its neighboring towns and villages, Avboisi, Okpuje, Ukhuse-Osi, Ukhuse-Oke in Owan, Etsakor, and Akoko Edo, are known as Afemai. They collectively make up edo-north senatorial district. Uzebba is located at latitudes 6°59' 40" N, longitudes 5° 54' 17" E and Lat/Long (dec) 6.99472,5.90496. Figure 1.1 shows the map of study area.

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1.4 ACCESSIBILITY

Uzebba is a region that is greatly accessible with good road networks that links major area in the region. The inhabitants of Uzebba are predominantly farmers. There are different modes

of transportation and road transport plays a significant role in agricultural productivity. This is as a result of it being the major means of transportation of agricultural produce from the

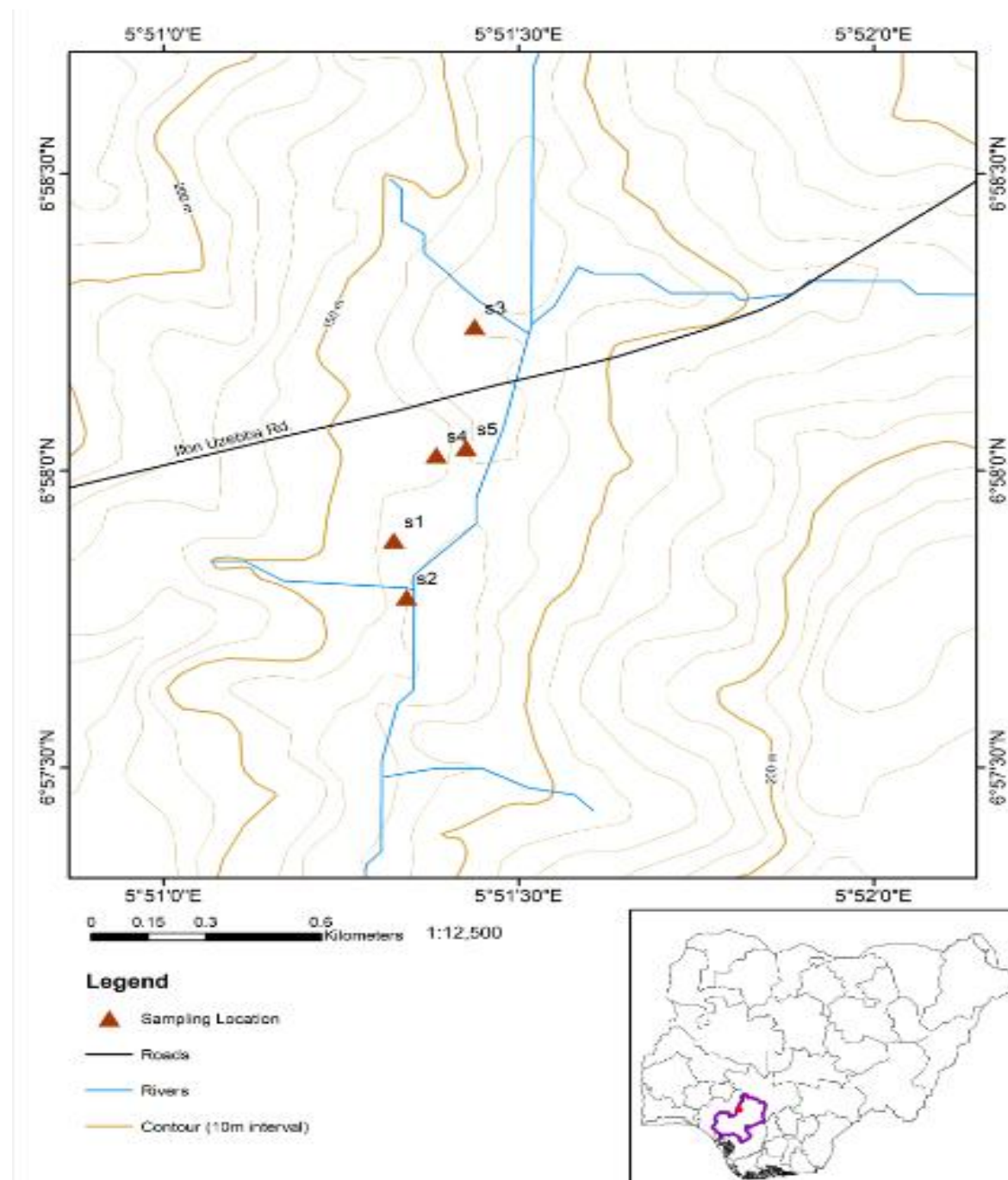


Figure 1.1: Map of the Study Area Showing the Sampled Locations

rural farmlands to the rural market as well as most urban areas in the State like Esan land and Benin City (Ikhile, 2016). Figure 1.1 shows the map of study area

1.5 GEOGRAPHY AND GEOMORPHOLOGY OF UZEBBA

Deep chemical weathering, slope processes, and river activity are some of the geomorphological processes that take place in the Uzebba community. On Benin Region, Uzebba is mainly a region located on the Coastal Plains. The area is located in the most southern portion of a dissected margin, a topographical unit that is located south of the Western plains and ranges and west of the lower Niger Valley and north of the Niger Delta (Ikhile, 2016). The research region includes a comparatively flat to undulating landscape, and the elevation, as measured by a hand-held Global Positioning System (GPS), ranges from 95 to 102 meters above mean sea level. The study area occupies part of the northern part of Edo State characterized by sedimentary rocks which are anchored on relatively stable, crystalline basement rock formations. The boulder rocks are broken down using accurately placed dynamites into fairly portable rock fragments. These fragments are processed into smaller rocks and stones using the quarrying machines installed in small-scale industries.

1.6 TOPOGRAPHY AND HYDROLOGY

Uzebba is located at an elevation of none meters (0 feet) above sea level, Uzebba has a tropical wet and dry or savanna climate. The district's yearly temperature is 30.02°C (86.04°F) and it is 0.56% higher than Nigeria's averages. Uzebba typically receives about 191.42

millimeters (7.54 inches) of precipitation and has 277.41 rainy days (76.0% of the time) annually. Mineral resources that are predominantly found there include limestone and lignite.

1.7 CLIMATE

Uzebba community in Nigeria's tropical rain forest has two distinct seasons (wet from April to October and dry from November to March). Although man's effect on development projects like roads and buildings has had a significant impact on the study area's vegetation, some areas still have pockets of light woodland. Tropical rain forest covers most of the area. The region is inhabited largely by the Owan (Iluhah) people, who are linked to the historic kingdom of Benin.

1.8 HUMAN ACTIVITIES

Agriculture is the mainstay of the economy (Ikhile, 2016). Yams, cassava (manioc), oil palm produce and corn (maize) are the major subsistence crops, while rubber, timber, and palm oil and kernels are cash crops that are produced in large quantity in Uzebba. Extensive farm activities which produce yams, cassava, sweet potatoes, rice, maize, vegetables, fish, snails, poultry, goats, and cattle are carried out in this location. In the centre, the region descends from the Aduge Hills towards the Akuku undulating plains which encompasses Egbira, Enwan, Ikpeshi, Ivbie north- Okpela- Arhe, Okpemheri, Okpe, Oloma, Ososo, Sasaro, Ukaan and Uneme, where robust agricultural activities are practised as climate dictates seasonally.

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CHAPTER TWO

LITERATURE REVIEW

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2.1 GENERAL GEOLOGY

In Nigeria, sedimentary basins from the Late Cretaceous to the Tertiary are found. The largest is the Southern Nigerian Basin, which is made up of the Niger Delta, the Anambra Basin, the Eastern Dahomey Embayment, and the Afikpo Syncline. The Sokoto Embayment, which is located in the northwest of Nigeria, is a portion of the Iullemeden Basin, a much larger sedimentary and structural basin that is focused in the Republic of Niger (Greigert, 1961). The Bornu Embayment, a portion of the broader Chad Basin focused on the Republic of Chad, is located in Northeastern Nigeria. Tectonically created depressions called sedimentary basins collect sediment. The pressure on the sediments rises as they are buried, starting the compaction and lithification processes that turn the sediments into sedimentary rocks. The Sokoto Basin, Anambra Basin, Benue Trough, Chad Basin, Dahomey Basin, Bida Basin (Mid-Niger), and Niger Delta Basin are all parts of the Sedimentary Basin, which contains deposits of Cretaceous and Tertiary age.

2.2 REGIONAL GEOLOGIC SETTING

The south sedimentary basins sedimentary formation underlies the Benin Region. The top reddish soil, which is made of ferruginized or literally sandified clay, is typically what distinguishes the geology. Benin sand was first used by Parkinson (1907) to refer to the reddish soil that makes up the paleo-coastal environment of the Paleocene-Pleistocene Age and is underlain by sands, sandy clays, and ferruginized sandstone. The upper fins off-flaps of the Niger Delta are marked by these sediments, which are dispersed across the southern edges of the Anambra Basin. Tattam (1943) gave the formation of red soil covered in sands

and clays, which marks an old coastal plain environment currently exposed in Calabar, Owerri, Onitsha, and the Benin Region with the date Oligocene-Pleistocene, the term "Coastal Plain Sands."

To distinguish the reddish-brown-yellow, often white sands with clayey and pebbly horizons with a type-locality near Benin, Reyment (1965) restored the name Benin Formation. The Benin Formation's sedimentary suites dip 2° to 8° south. In terms of its geology, the Benin region consists of;

- i. Benin Formation
- ii. Alluvium
- iii. Drift/top soil
- iv. Azagba-Ogwashi (Asaba-Ogwashi) Formation

2.2.1 BENIN FORMATION

The Benin Formation is classified as the most aquiferous formation in Southern Nigeria. Various structural units (point bars, channel fills, natural levees, back swamp deposit, and ox-bow fills) are discernible within the formation, indicating the hydrogeological potentials. It possesses a transmissivity of 880–30,000m²/day, a storage coefficient of 1.53–3.16, and an average porosity of 30%. Akujieze, (2004) investigated the Benin Formation and reported unconfined to semi confined aquifers in certain places. The Benin region is underlain by sedimentary formation. The geology is generally marked by top reddish earth (Figure 2.1), composed of ferruginized or literalized clay sand. Parkinson (1970), first used the term Benin sand 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

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spread across the southern fringes of the Anambra Basin and marking the upper facies off-flaps of the Niger Delta. Murray (2007), used the name Coastal Plain Sands 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.

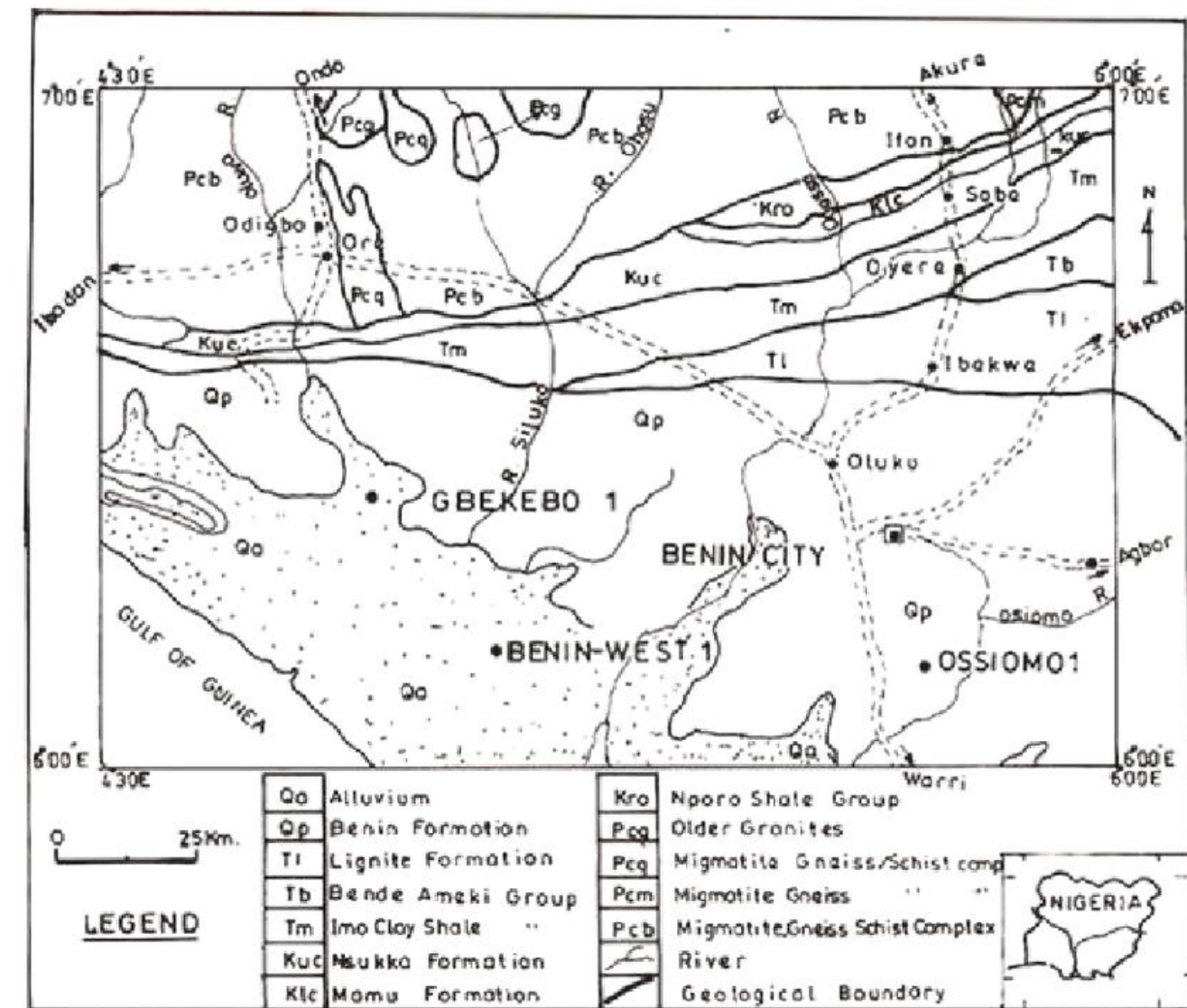


Figure 2.1: Benin Regional Formations (Google Map, 2023)

2.2.2 ALLUVIUM

These can be found near the floodplains of Ikpoba and Ovia. They are composed of gravel, silt, clayey sands, yellowish-white, yellowish-gray, and even wood-plant components. These have been left at the river's bank after being washed down the river valley.

2.2.3 DRIFT/TOP SOIL

Sediments that are still being moved or transported are called drifts. They are composed of mudflows, sands, and light brown-yellow-wish silt that resulted from the weathering of the parent Benin Formation. Fluvial agents, in particular the storms and hoods that dominate the region's wet season, wash down drifts. Roadside drifts fill in spaces and cover areas, hiding the underlying geology. Where drifts are stabilized, a soil profile is formed.

2.2.4 AZAGBA-OGWASHI (OGWASHI-ASABA) FORMATION

Gritty clays alternate with seams of lignite in this mixture of clays, sands, and grits. The Benin Formation is reached by an uphill gradient. According to Akujieze (2004), the Ogwashi-Asaba Formation is exposed in stream channels in the northern sections of the Benin Region, west of Ekiadolor-Iwu, 4 kilometers North of Azalla, and East of Ulekon.

The Niger Delta Basin surrounds the study area (Uzebba), which is made up of stratigraphic sequence and outcropping units. The three main lithostratigraphic units found in the Niger Delta's subsurface—Akata, Agbada, and Benin Formation—decrease in age basinward, reflecting the overall regression of the clastic wedge's depositional environments—marine, deltaic, and fluvial environments—within the Niger Delta (Short and Stauble, 1967). The type section of the Akata Formation was defined in Akata 1 well, 80km east of Port Harcourt (Short and Stauble, 1967).

The Agbada 2 well, which was dug about 1 km to the north-northwest of Port Harcourt, defines the Agbada Formation (Short and Stauble, 1967). The formations have a maximum thickness of roughly 13,000 feet and can be found across the Niger Delta clastic wedge. The lithologies are composed of alternating sands, silts, and shales that are grouped within successions of ten to one hundred feet that are marked by an upward progression in grain size and bed thickness.

From the Benin-Onitsha region in the north to beyond the current coastline, the Benin Formation makes up the upper portion of the Niger Delta clastic wedge (Short and Stauble, 1967). The recent subaerially exposed delta top surface, which has an extent of 4600 feet, is the top of the formation. In Figure 2.2, the base is defined by youngest marine shale. Shallow parts of the formation are composed of entirely non-marine sand deposited in alluvial or upper coastal plain environments during progradation of the Delta (Doust and Omatshola, 1989). The subsurface lithostratigraphic sequence of Niger Delta comprises:

- i. Akata Formation
- ii. Agbada Formation
- iii. Benin Formation

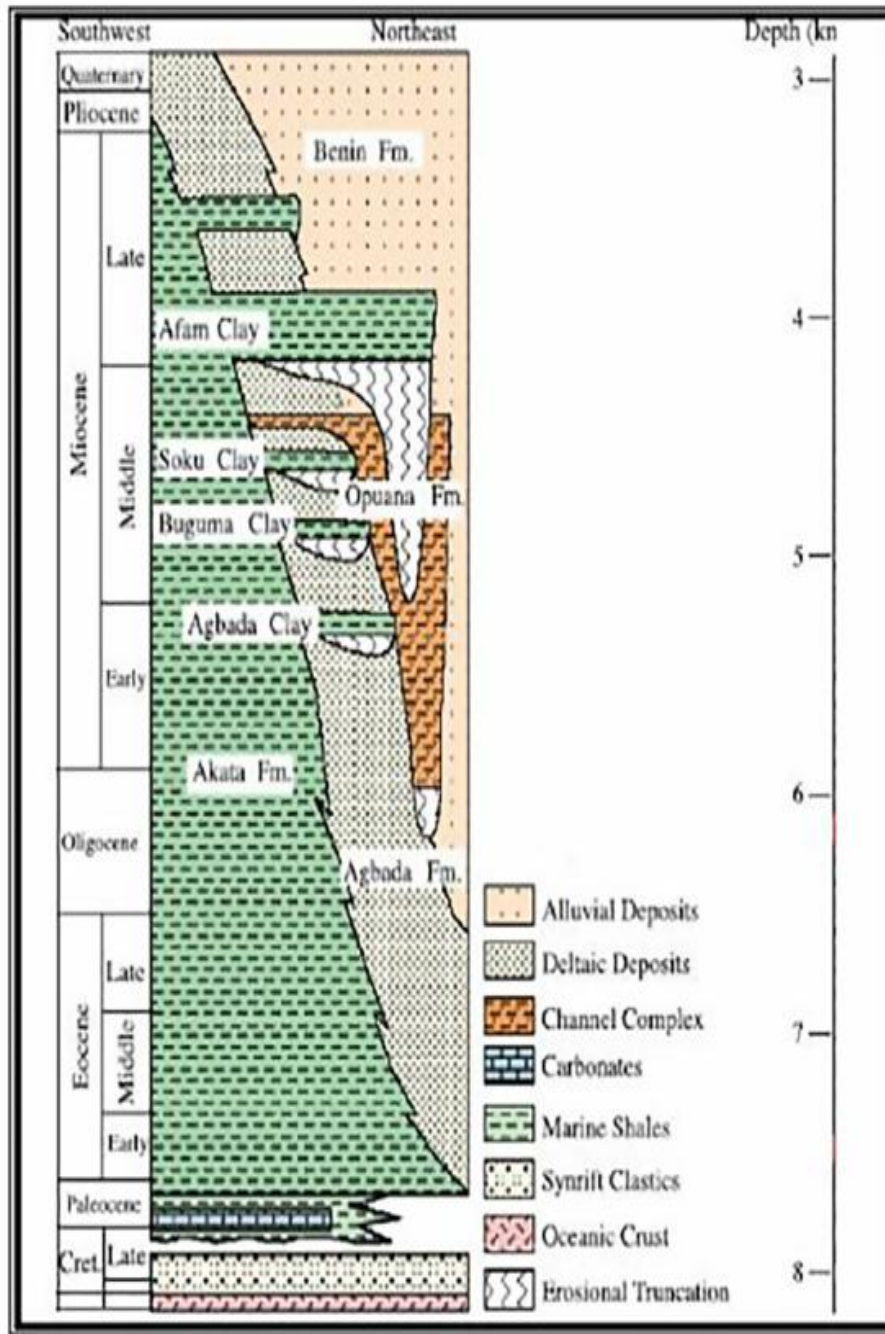


Figure 2.2: Niger Delta stratigraphic column showing the three major sedimentary formations

2.3 LOCAL GEOLOGY

The study area is part of the Benin Formation, which stretches beyond the current coastline to the south and west throughout the whole Niger Delta region. Shale intercalations cover about 90% of the sandstone's surface. It is sub-angular to well-rounded, poorly sorted, coarse grained, gravelly, locally fine grained, and contains wood fragments and lignite streaks. It is a continental deposit that likely formed in an upper deltaic environment. Uzebba which falls within the Benin Formation is of Oligocene to Miocene age which is the youngest in the sequence of formations in the Niger Delta Basin and the other two formations are Akata and Agbada. The lithologies of the Benin Formation comprises reddish brown sands, silt, sandstone and clay, which are generally referred to as “Coastal Plain Sands”. In some localized areas, the clay thickness of the supplementary material, is significantly above 30 m, while in some other areas, they appear as thin beds of less than one (1) meter. Their colours also vary from light brown, reddish brown and pale white colours as observed in the sample collected. The mined clay samples are cut to sizes of supplementary material, and loaded in sack bags for firing before sale. Uzebba is known for its mineral resources. Some of the minerals found in the area include limestone, clay, silica, and granite, which have economic importance for construction, ceramics, and other industries. The geological landscape of Uzebba may exhibit various structural features such as faults, folds, and fractures due to tectonic forces that have acted on the region over millions of years. These structures can influence groundwater flow and the formation of mineral deposits. The local geology has a direct influence on the topography of the area. The presence of the softer rocks may erode to create valleys and lowlands. Understanding the local geology is crucial for assessing groundwater resources in Uzebba. Figure 2.3 shows the geological map of the study area. The geological formations and structural features impact on the availability and quality of

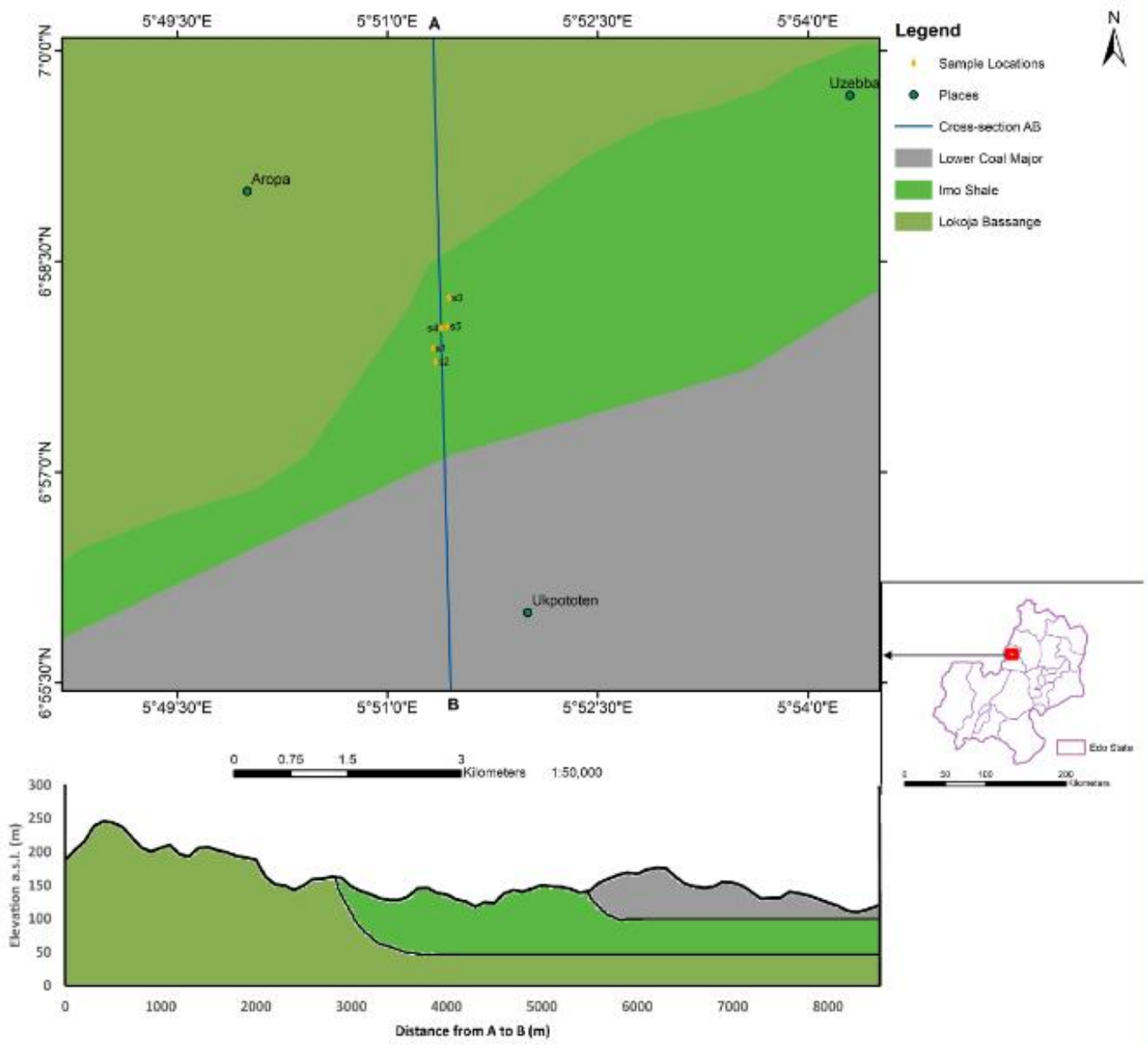


Figure 2.3: Geological Map of the Study Area



Plate 2.4: Profile of Clay Deposits in the Study Area



Plate 2.5: The Dark Coloration of the Clay Body from the Study Area



Plate 2.6: Dark in Colour Clayey Rock in the Sample Location



Plate 2.7: Laminated Clay samples Collected by the Researcher from the Study Area

groundwater, which is vital for both domestic and agricultural use. Plates 2.1- 2.4 show profile of clays in the study area.

2.4 PREVIOUS WORKS

Kankao *et al.*, (2022) studied “Mineralogical, Geochemical Characterization and Physicochemical Properties of Kaolinitic Clays of The Eastern Part of The Douala Sub-Basin, Cameroon, Central Africa”, stated that the studied clay materials are good candidates for the production of ceramics and terracotta building. This study is therefore important for any application of this type of clay in various industrial fields.

Kumari and Mohan (2021) studied “Basics of Clay Minerals and Their Characteristics Properties”. The small size and their distinctive crystal structure make clay minerals very special with their unique properties including high cation exchange capacity, swelling behaviour, specific surface area, adsorption capacity, etc. Due to all these unique properties, clay minerals are gaining interest in different fields.

Aliu *et al.*, (2021) carried out a study on “Geochemical Composition, Mineralogy, Geotechnical Characteristics of Some Clay Deposits in Parts of The Southern Niger Delta, Nigeria”. The XRF reveals that SiO_2 and Al_2O_3 are the predominant oxides. The XRD analysis shows that kaolinite is the predominant clay mineral with varying amount of quartz also traces of illite, smectite. The geotechnical index test shows that the clay soil samples studied also contains considerable amounts of silt-size particles (18%-70%) which makes them unsuitable in their raw state for use as fillers, raw materials in the paint industries.

Adewole *et al.*, (2020) examined “The Geochemical and Mineralogical Characteristics of Clay Deposits at Ijesha–Ijebu and Its Environs, Southwestern Nigeria” indicated that the clay within the weathered profiles above banded gneiss and pegmatite at Ijesha-Ijebu is brownish

with red spots, while the clays derived from sedimentary terrain is chocolate in colour. The X-ray diffraction results showed that kaolinite is the dominant mineral, while quartz, albite and muscovite are the major non clay minerals.

Olaonipekun (2020) evaluated The Mineralogical, Physicochemical and Chemical Characteristics of Clay In Parts Of Eastern Dahomey And Niger Delta Basins, Nigeria using X-ray, SEM and DTA methods. From their findings it was observed that the clays have low Ec and pH values. It was also noted that the major oxides in the clay are SiO₂, Fe₂O₃, Al₂O₃ and TiO₂. Based on comparison with specifications and standards, the kaolins cannot be used for industrial purpose. However, they recommended that the Cretaceous Lakiri kaolins can be used in the paper and ceramic industry (except for kaolins having high TiO₂ and K₂O content).

Onyekuru et., al (2018) in “Mineralogical and Geochemical Properties of Clay Deposits In Parts of Southeastern Nigeria” concluded that the mineralogical analysis showed the presence of kaolinite, bentonite, dickite, quartz, and iron minerals in the clay deposits. Kaolinite and quartz were the most dominant minerals as they occurred in all the samples. Bentonite occurred only in samples from Awomamma and Orlu indicating a possible source from the adjoining Cameroun volcanics.

Obrike (2012). Evaluation Of Imo Clay-Shale Deposit (Paleocene) From Okada, Edo State. The geochemistry, mineralogy and physical characteristics of these clays indicate that it is suitable for various industrial uses including pottery products, although may not be considered good economical raw material for ceramics.

Akhirevbulu and Ogunbajo (2011) examined “The Geotechnical Properties of Clay Occurrences Around Kutigi Central Bida Basin, Nigeria”. Due to the high amount of sand

present in the clay, suggestion where made that it can be utilized in the manufacturing of ceramics, refractory bricks, paper, fertilizer and plant.

Ojo *et al.*, (2011) studied “Sedimentological and Geochemical Studies of Maastrichtian Clays In Bida Basin, Nigeria: Implication for resource potential” and observed that they show low heavy metal concentrations and thus may be beneficiated and suitable as raw materials for ceramic, pharmaceuticals and paints.

Ilgin and Gupta (2010) in their work “Environmental Conscious Manufacturing and Product Recovery (ECMPRO): A Review of the State of the Art.” stated that the tiny size and plate form of clay particles gives clay minerals a high surface area. Most clay deposits are impure. Many naturally occurring deposits include both silts and clay.

Odubiyi (2010) carried out a study on “The Properties Of Clay In Delta State, Nigeria”. This paper provides some insights on the properties of clay to accelerate pottery production and continued improvement.

Jubril and Amajor (1991) studied “The Mineralogical a Geochemical Aspects of The Afam Clay (Miocene), Eastern Nigeria Delta” and observed that they possess characteristics satisfaction for economic and continued improvement.

CHAPTER THREE

MATERIALS AND METHODOLOGY

3.1 MATERIALS

The materials for the study include;

- i. Global Positioning System (GPS)
- ii. Cutlass
- iii. Field note
- iv. Sample Bag
- v. Pick hammer

3.2 SAMPLE COLLECTION

Five (05) samples were collected from different locations in Uzebba marked UZ1, UZ2, UZ3, UZ4 and UZ5, respectively. The samples were collected using a shovel and placed in airtight polythene bags and sent for analysis at National Steel Raw Materials Agency (NSRMEA) in Kaduna State. The equipment used for the geochemical analysis is the Xenometrix (Genius IF) machine, while the Regaku Miniflex machine was used for the mineralogical analysis of the clay samples.

3.3. METHODOLOGY

3.3.1 X-RAY DIFFRACTION METHOD

X-rays are used to determine the crystal structure of materials. It is an experimental method in which a beam of X-ray is made to pass through a sample of the material being tested. Since the atoms are arranged in some order in crystals, they tend to diffract the beam at certain angles and at certain intensity. The angles of the diffracted beam are measured X-ray

crystallography and the crystal structure of the material is calculated thereof. It is also possible to determine if the material is not crystalline.

3.3.1.1 X-RAY DIFFRACTION OF CLAYS

Typically, powder X-ray diffraction (XRD) is an average of randomly oriented microcrystals that should equally represent all crystal orientation if a large enough sample is present. X-rays are directed at the sample while slowly rotated which produce a diffraction pattern which show intensity of x-rays collected at different angles. Randomly oriented XRD samples are not as useful for clay minerals because clays typically have similar X and Y dimensions. The Z dimension differs from clay to clay and is most diagnostic because the Z dimension represents the height of the tetrahedral-octahedral (T-O) or tetrahedral-octahedral-tetrahedral (T-O-T) layer. The Z dimension can increase or decrease because of substitution of the central cation in both the tetrahedral and octahedral layers. The presence and size of a charge balancing cation in the interlayer of T-O-T clays will also affect the Z dimension. Because of this, clay minerals are typically identified by preparing samples so that they are oriented to increase basal (00l) reflection. D positions are calculated using Bragg's law but because clay mineral analysis is one dimensional, l can substitute n, making the equation $l \lambda = 2d \sin \Theta$. When measuring the x-ray diffraction of clays, d is constant and λ is the known wavelength from the x-ray source, so the distance from one 00l peak to another is equal.

3.3.1.2 PREPARATION FOR CLAY MINERAL X-RAY DIFFRACTION

Clays should be separated from the non-clay minerals to reduce interference of 00l peaks. Non-clay minerals can usually be separated by sieving samples at a small enough mesh. Samples should be lightly crushed but not pulverized because non-clay minerals will be reduced to clay sizes and become impossible to separate from the sample. Lightly crushing

breaks apart the soft clays while keeping harder non-clays intact for easier removal. Samples should be as homogeneous as possible, both in grain size and composition before mounting them for X-ray diffraction and long, flat, and thick samples are ideal. Four methods are commonly used for sample preparation and vary in difficulty and appropriateness of use.

3.3.1.3 CENTRIFUGED POROUS PLATE

Produces the best diffraction patterns out of the most common methods but requires the most skill and is most time-consuming. Upon completion, samples have thick aggregates and preferred orientation. A special apparatus designed to hold a porous ceramic plate is placed into a centrifuge container and filled with suspended sample. Centrifuging forces the liquid through the porous plate leaving the sample behind to be dried below 100 °C. An advantage of this method is that exchangeable cations can be removed by passing a chloride solution through the plate once the sample has been dried. Exchanging cations can be useful when establishing peaks for standards with variable interlayer cations. For example, nontronite has an interlayer which can contain both calcium and sodium. If an unknown sample was suspected to only contain one of these cations, a more accurate standard could be prepared by exchanging the undesired cation.

3.3.1.4 XRD LIMITATIONS

XRD also has size limitations. It is much more accurate for measuring large crystalline structures rather than small ones. Small structures that are present only in trace amounts will often go undetected by XRD readings, which can result in skewed results. It cannot identify the amorphous materials and does not give information on profile depth.

3.4 X-RAY FLUORESCENCE

This method operates on the principle of atomic physics and quantum chemistry. The specimens were exposed to the entire spectrum of photons consisting of primary radiations emitted from a standard X-ray tube. These irradiated specimen causing the elements in it to emit secondary fluorescence with their characteristics X-ray line spectra. The energies and intensities of the emitted lines were determined by the detection system. This is made up of two units; the primary channel simultaneous wavelength dispersive spectrometer and the personal computer for control and data processing. The rapid detection system employs pre-positioned (analyzing) crystal around the specimen. These cause the dispersion of the wavelength of the secondary radiation.

The intensities of the individual wavelength are measured in a mass gas flow detector. This system allows simultaneous measurements of up to ten elements at peak and background positions. The output signals from the detector were fed into the analyser, where the photon counts were stored in the computer memories. The count rate was calibrated for each element by comparing it to the count rate from a standard of accurately pre-determined composition. The spectral line energies of wavelengths of the emitted lines were used in the quantitative analysis of the element in the specimen. The intensities of the emitted line were related to their concentration for quantitative analysis.

3.4.1 PROCEDURE FOR XRF TECHNIQUE

1. Crushing of each sample with an electric crusher and then pulverized for 60 seconds using Herzog Gyro-mill (Simatic C7-621).

2. Pellets were prepared from the pulverized sample, first by grinding 20g of each sample with 0.4g of stearic acid for 60 seconds. After each grinding, the Gyro-mill was cleansed to avoid contamination.
3. 1g of stearic acid was weighed into an aluminum cup to act as binding agent and the cup was subsequently filled with the sample to the level point.
4. The cup then taken to Herzong pelletizing equipment when it was passed at a pressure of 200KN for 60 seconds.
5. The 2mm pellets were added into a sample holder of the x-ray equipment (Phillips PW-1800) for analysis.

$$\text{CIA} = (\text{Al}_2\text{O}_3 / (\text{Al}_2\text{O}_3 + \text{CaO} + \text{Na}_2\text{O} + \text{K}_2\text{O})) \times 100 \text{ ----- (1)}$$

3.4.2 SAFETY CONSIDERATIONS

The use of XRF and XRD instruments is user-friendly, however, extremely hazardous implications of X-ray exposure needs special attention. It is, therefore, important for both occasional users and trained personnel to consider essential safety measures for avoiding any direct and secondary radiation exposure. Harmful effects of X-ray exposure are cumulative, and can result in permanent and serious injury such as burns. For making sure that radiation exposure is negligible and under safe limits, a dosimetry badge should be worn and checked by regular users.

3.4.3 XRF LIMITATIONS

Since XRF measurements rely on quantity, there are limits on the measurements. The normal quantitative limit is 10 to 20 ppm (parts per million), usually the minimum particles required for an accurate reading. XRF also can't be used to determine Beryllium content, which is a distinct disadvantage when measuring alloys or other materials that might contain Beryllium.

It can also face limitations in measuring lighter elements. Plates 3.1 -3.2 shows XRD and XRF machines.



Plate 3.1: X-ray diffraction (XRD) machine



Plate 3.2: X-ray Florescence (XRF) Machine

CHAPTER FOUR

PRESENTATION AND DISCUSSION OF RESULTS

4.1 PRESENTATION OF RESULTS

The results of the geochemical and mineralogical analysis are presented in Table 4.1 and 4.5.

TABLE 4.1: CHEMICAL COMPOSITION OF THE MAJOR OXIDES OF THE CLAYS IN THE STUDY AREA

Major Elements (wt.%)	UZ1	UZ2	UZ3	UZ4	UZ5	Average Value (wt. %)
SiO₂	76.51	71.41	77.48	62.23	72.43	72.01
Al₂O₃	13.50	18.93	14.01	15.46	18.88	16.16
Fe₂O₃	1.00	4.06	0.85	13.83	1.01	4.15
TiO₂	2.50	2.78	3.05	2.15	3.01	2.70
MgO	ND	ND	ND	ND	ND	ND
CaO	0.38	0.26	1.05	0.23	0.75	0.53
Na₂O	ND	ND	ND	ND	ND	ND
K₂O	0.87	0.64	0.68	0.39	0.70	0.66
MnO	0.02	0.02	0.02	0.03	0.03	0.02
P₂O₅	0.03	0.07	0.07	0.23	0.13	0.11

From the XRF analysis it was observed that the clay samples have high percentage of SiO₂ with UZ3 having the highest amount of 77.48wt.% and UZ4 having lowest value of 62.23wt.%. Al₂O₃ has the second largest values ranging from 13.50wt.% UZ1 to 18.93wt.% in UZ2 having the highest amount of Al₂O₃ concentration.

The Table showed that the major oxides are expressed in molar proportions and CaO is the content of CaO incorporated in silicate fraction. Fedo *et al.*, (1995) proposed an indirect method for quantifying CaO content of silicate fraction by assuming reasonable values of Ca/Na ratios of silicate material. Procedure for quantification of CaO content (CaO*) of silicate fraction involves subtraction of molar proportion of P₂O₅ from the molar proportion of total CaO. After subtraction, if the “remaining number of moles” is found to be less than the molar proportion of Na₂O, then the “remaining number of moles” is considered as the molar proportion of CaO of silicate fraction. If the “remaining number of moles” is greater than the molar proportion of Na₂O, then the molar proportion of Na₂O is considered as the molar proportion of CaO of silicate fraction (CaO*).

It was discovered that the major oxides composition of the clay samples were dominated by SiO₂, which ranges from 77.48wt.% - 67.48wt.% (Average = 72.13wt.%). The limited range of SiO₂ content may be due to poor sorting and rapid deposition. Al₂O₃ ranges from 13.05wt.%– 18.88wt.% (Average = 16.16wt.%), this can be attributed to composition of lithic fragments with low concentrations of Fe₂O₃ (Average = 4.15wt.%), K₂O (Average = 0.66wt.%), TiO₂ (Average = 2.70wt.%), P₂O₅ (Average = 0.11wt.%), and CaO (Average = 0.53wt.%).

Iron as Fe₂O₃ is one of the major oxides in the clay deposits in the study area. Its high values (>2%) in the samples from Uzebba town is probably responsible for the extreme changes in

colour of the clays to black in Uzebba town. Other factors could contribute to color variations in clays, including the presence of some other constituents such as CaO, MgO, MnO and TiO₂, relative amounts of Al₂O₃, the temperature of firing and the furnace conditions all play important roles. Table 4.2 shows origin, quality and major minerals present in the sample clay.

4.2 PROVENANCE

TABLE 4.2: RATIO OF SiO_2 TO Al_2O_3 (INDICATING MAJOR MINERAL PRESENT), Al_2O_3 TO Fe_2O_3 (INDICATING CLAY FOR GOOD QUALITY CEMENT) AND Al_2O_3 TO TiO_2 INDICATING PROVENANCE (WHETHER THE ROCK IS CLASTIC OR IN-SITU)

Uzebba Study	$\text{SiO}_2/\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3/\text{Fe}_2\text{O}_3$	$\text{Al}_2\text{O}_3/\text{TiO}_2$
UZ1	5.86	13.5	0.5
UZ2	3.77	4.66	4.46
UZ3	5.53	16.48	0.03
UZ4	4.03	1.12	6.43
UZ5	3.84	18.69	0.34

The clay deposits in the study area are mainly clastics and not the product of in situ weathering, hence, the geochemical signatures used to identify provenance were those used for clastic rocks (Madharaju and Ramasamy, 2002; Armstrong-Altrin *et al.*, 2004). The ratios of most clastic rocks have also been used to infer the source rock composition: $\text{Al}_2\text{O}_3/\text{TiO}_2$ ratio increases from 3 to 8 for mafic igneous rocks, 8 to 21 for intermediate rocks and 21 to 70 for felsic igneous rocks (Hayashi *et al.*, 1997). The application of these hypotheses in the analyzed clay samples deposited in the study area showed that the calculated $\text{Al}_2\text{O}_3/\text{TiO}_2$ ratios ranged from 0.03 to 6.43, which is indicative that the clays is mafic igneous rocks. Ekosse, (2001) also used of TiO_2 versus Al_2O_3 binary plots to distinguish between granitic and basaltic source rocks. Tables 4.3 and 4.4 show chemical composition of reference clay samples and standard specification of oxides in clay respectively. Figures 4.1a and 4.1b to 4.5a and 4.5b show the X-ray diffractogram and Pie charts.

TABLE4.3: CHEMICAL COMPOSITION OF SOME KNOWN REFERENCE CLAY

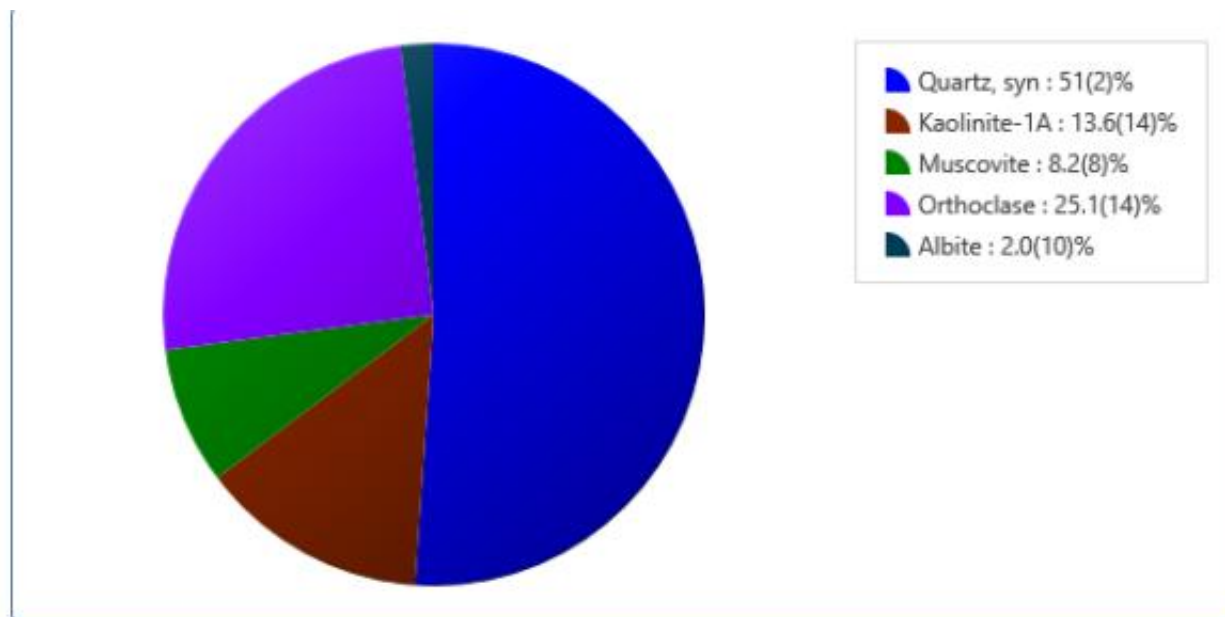
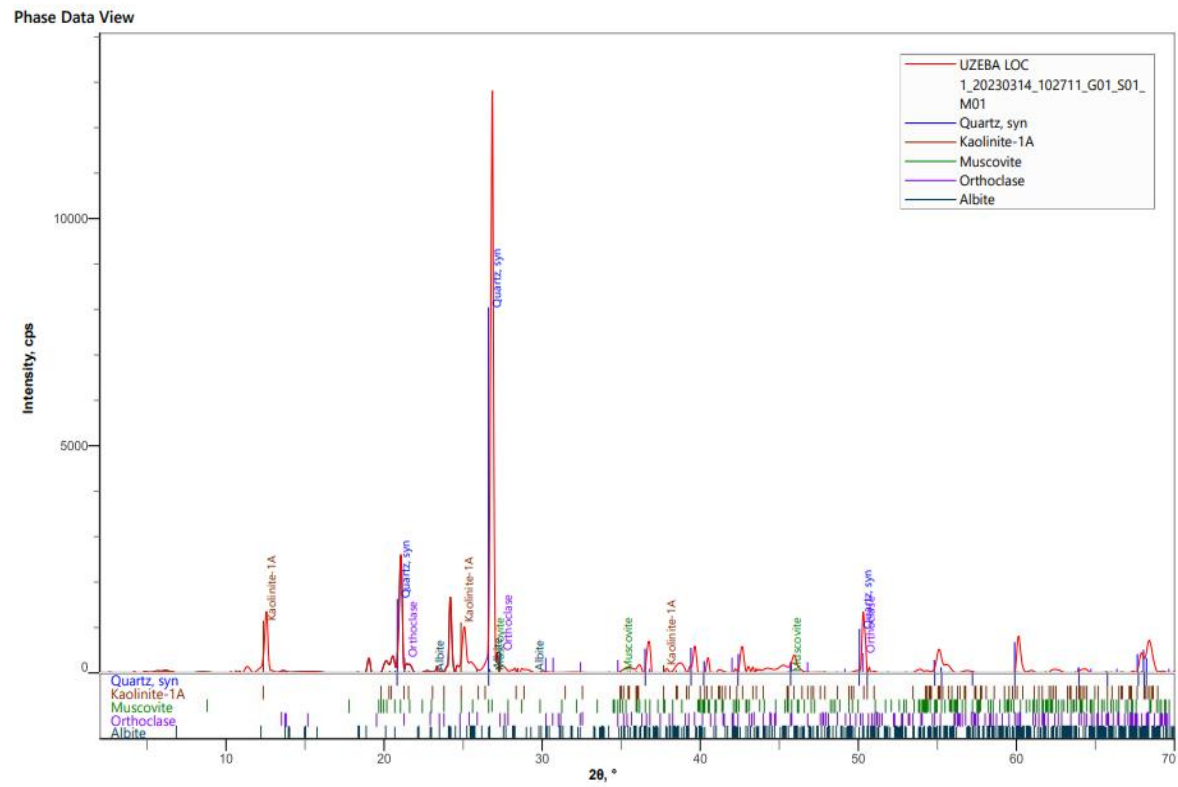
SAMPLES

Oxides (%)	Average value of this study (Uzebba Clay)	Afikpo Shale (Arua & Onyeok (1978)	Afam Clay (Jubril & Amajor, 1991)	Plastic fire Clay of St. Louis (Huber, 1985)	Maastrichtian Clay of Bida Basin (Olushola <i>et al.</i>, 2011)	Dukku Clay Kebbi State (Salihu & Suleiman, 2018)
SiO₂	76.51	59.81	42.20	57.67	63.3	55.59
Al₂O₃	13.50	21.76	26.20	24.00	24.6	35.5
Fe₂O₃	1.00	3.02	5.10	3.23	1.60	4.50
TiO₂	2.50	0.92	ND	ND	1.75	0.02
MgO	ND	1.45	0.70	0.30	0.06	1.90
CaO	0.38	1.32	1.60	0.70	0.04	2.10
Na₂O	ND	0.47	2.90	0.20	0.07	ND
K₂O	0.87	0.87	8.30	0.50	0.32	ND
MnO	0.02	ND	0.03	ND	0.01	ND
P₂O₅	0.03	ND	ND	ND	0.07	ND

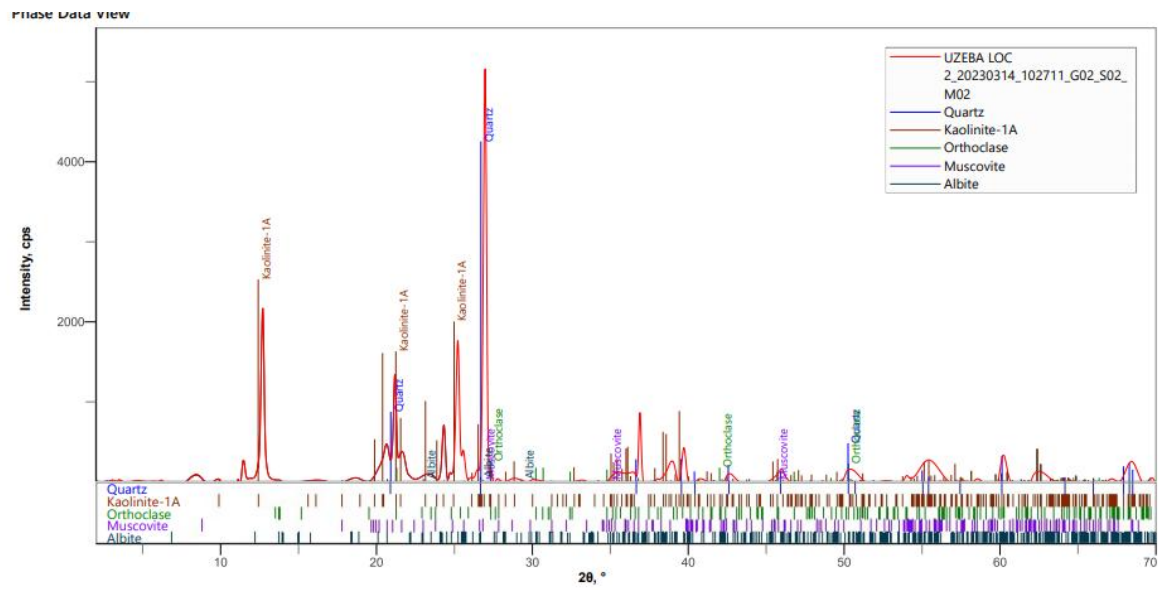
TABLE 4.4. STANDARD SPECIFICATIONS OF THE CONCENTRATIONS (IN WT.%) OF OXIDES IN CLAYS FOR VARIOUS INDUSTRIAL USES.

Oxides (%)	Study Area Average Value	A	B	C	D	E	F
SiO ₂	76.51	51.70	67.50	44.90	45.67- 48.5	38.67	68.47
Al ₂ O ₃	13.50	25.44	26.5	32.35	33.5-36.1	9.45	14.94
Fe ₂ O ₃	1.00	0.5-1.2	0.5-1.2	0.43	0.3-0.6	2.70	8.96
TiO ₂	2.50	1.0-2.8	0.1-1.0	1.80	0.0-1.7	ND	ND
MgO	ND	0.2-0.7	0.1-0.19	Tr	ND	8.5	1.143
CaO	0.38	0.1-0.2	0.18-0.3	Tr	0.0-1.5	15.84	1.65
Na ₂ O	ND	0.8-3.5	0.2-1.5	0.14	0.0-1.6	2.76	2.045
K ₂ O	0.87	ND	1.1-3.1	0.28	0.0-1.6	2.76	2.05
MnO	0.02	ND	ND	ND	ND	ND	ND
P ₂ O ₅	0.03	ND	ND	ND	ND	ND	ND

A) Refractory bricks (Parker, 1967); B) Ceramics (Singer and Sunja, 1971); (C) Rubby (Keller, 1964); (D) Paper (Keller, 1964); (E) Brick Clay (Murray, 1960); (F) Refractory bricks and Ceramics (Malu *et al.*, 2013)



Figures 4.1a and 4.1b: X-ray diffractogram and Pie chat for UZ1



UZEBALOC 220230314_102711_G02_S02_M02

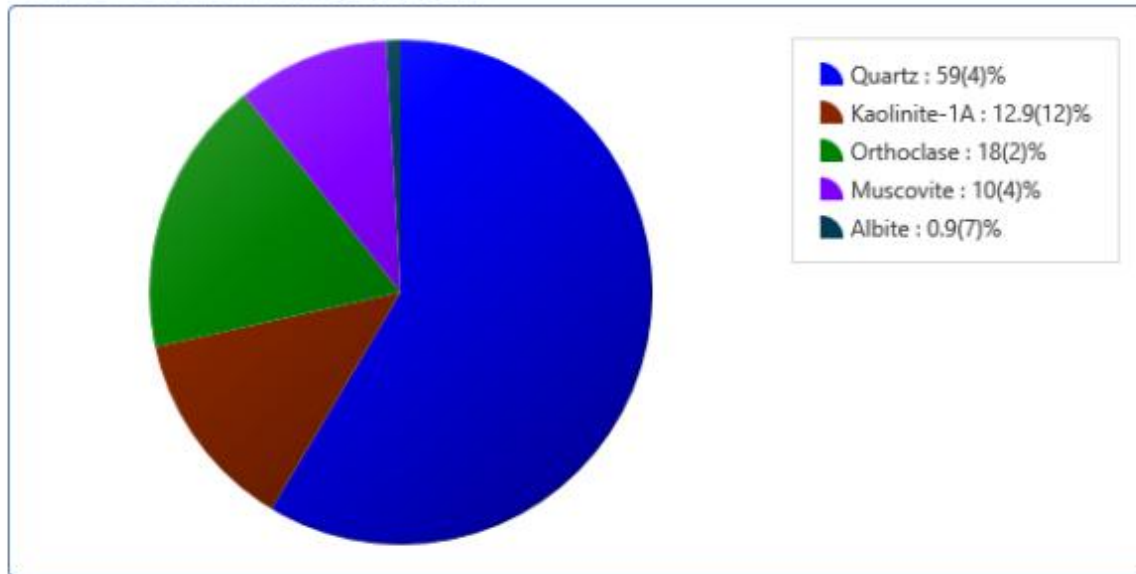
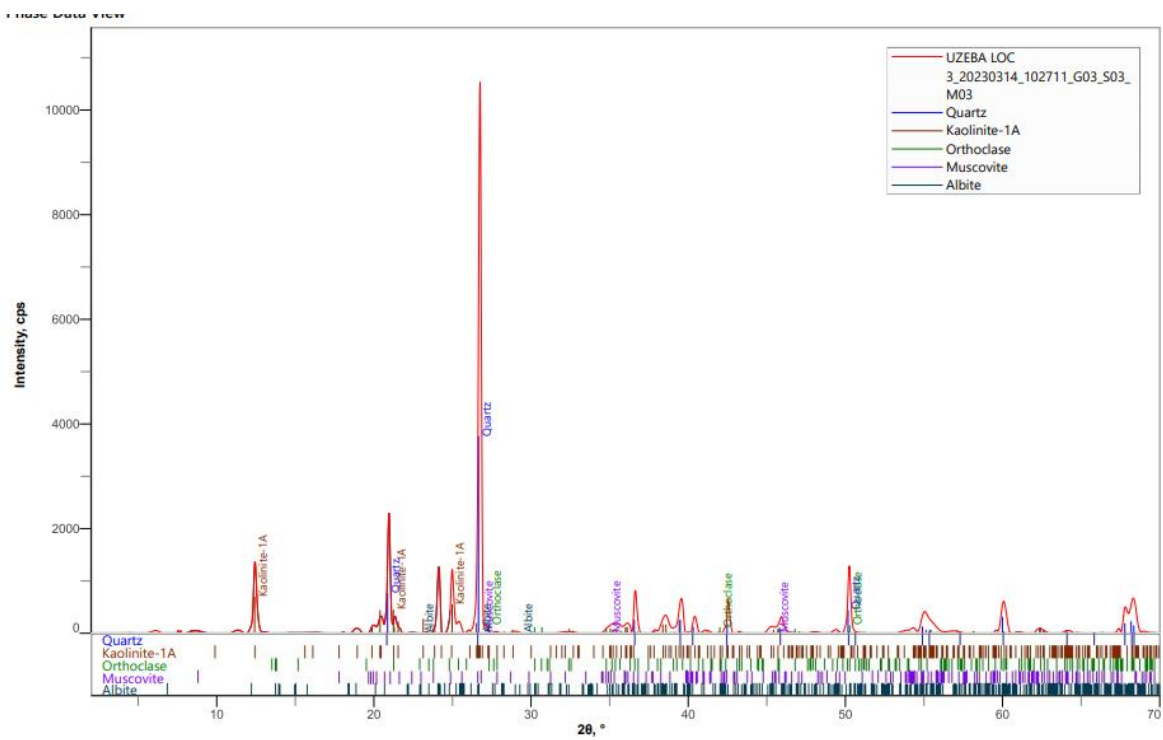


Figure 4.2a and 4.2b: X-ray diffractogram and Pie chat for UZ2



UZEBALOC 320230314_102711_G03_S03_M03

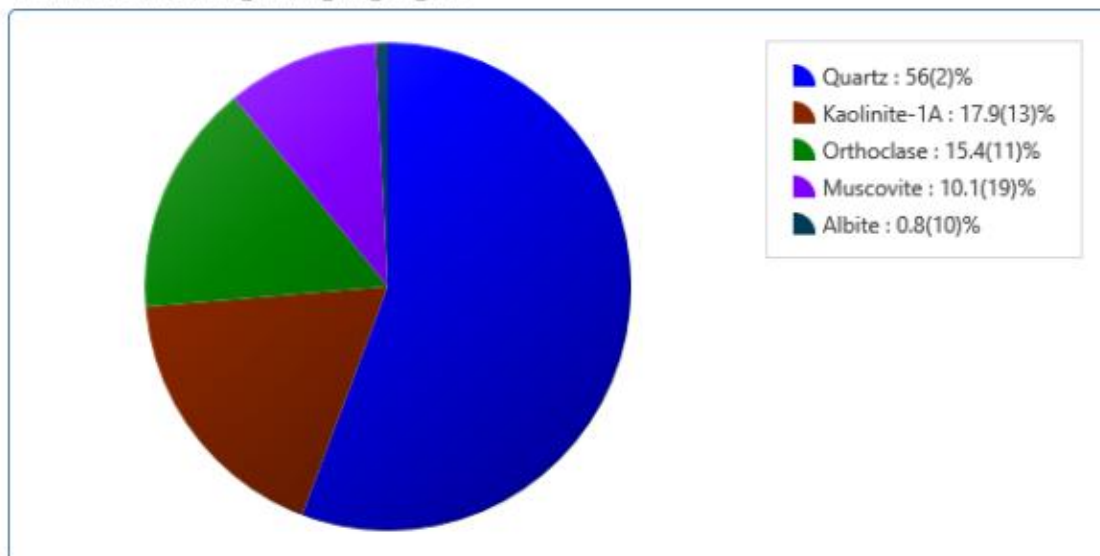
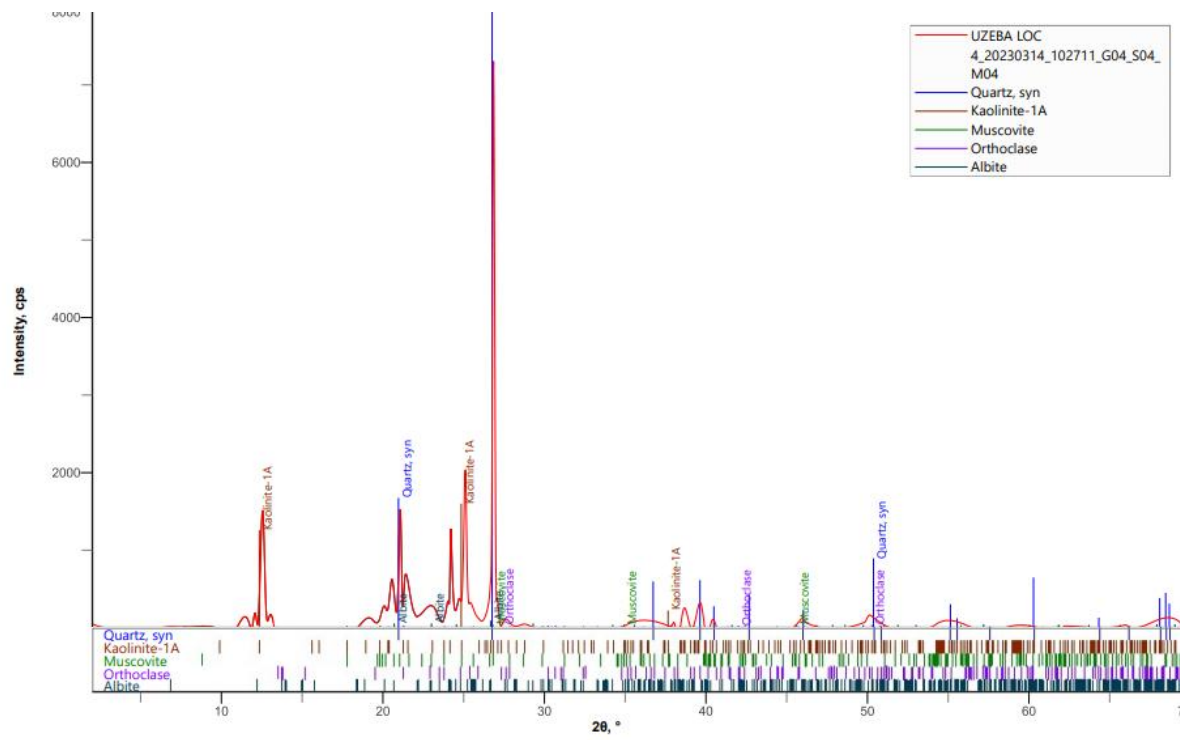


Figure 4.3a and 4.3b: X-ray diffractogram and Pie chat for UZ3



UZEBALOC 420230314_102711_G04_S04_M04

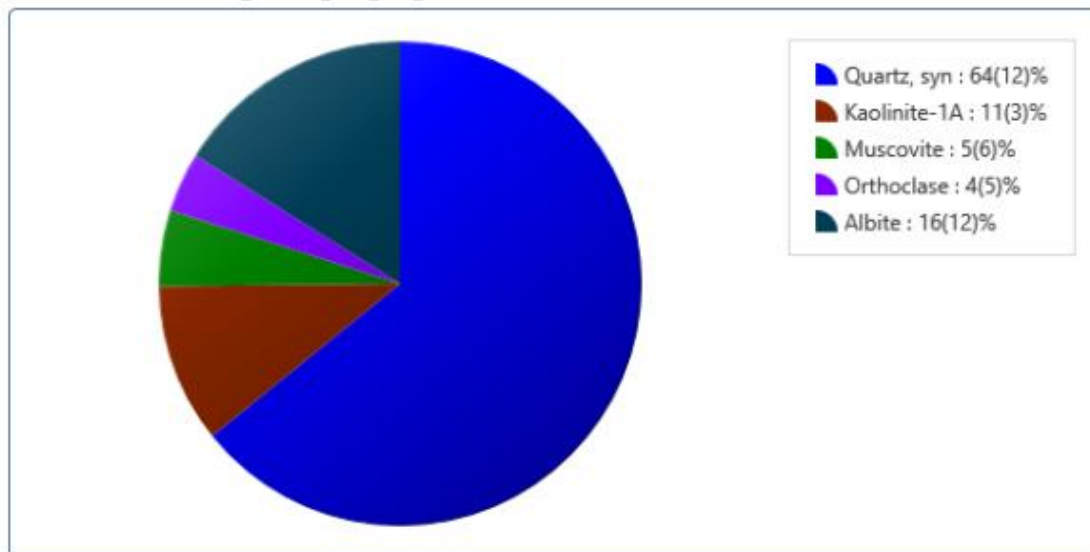
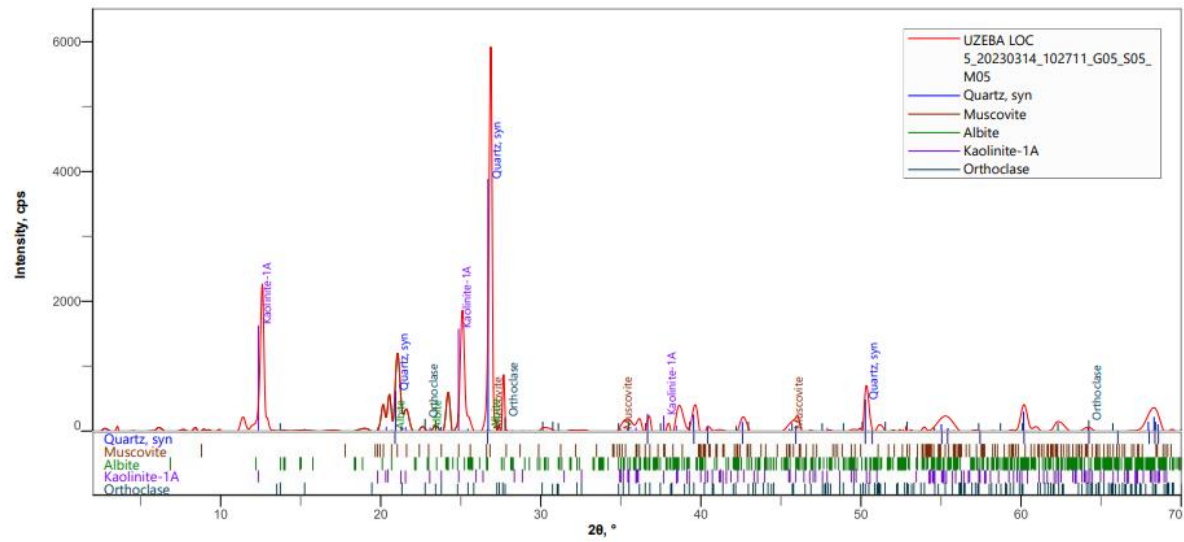


Figure 4.4a and 4.4b: X-ray diffractogram and Pie chat for UZ4



UZEBALOC 520230314_102711_G05_S05_M05

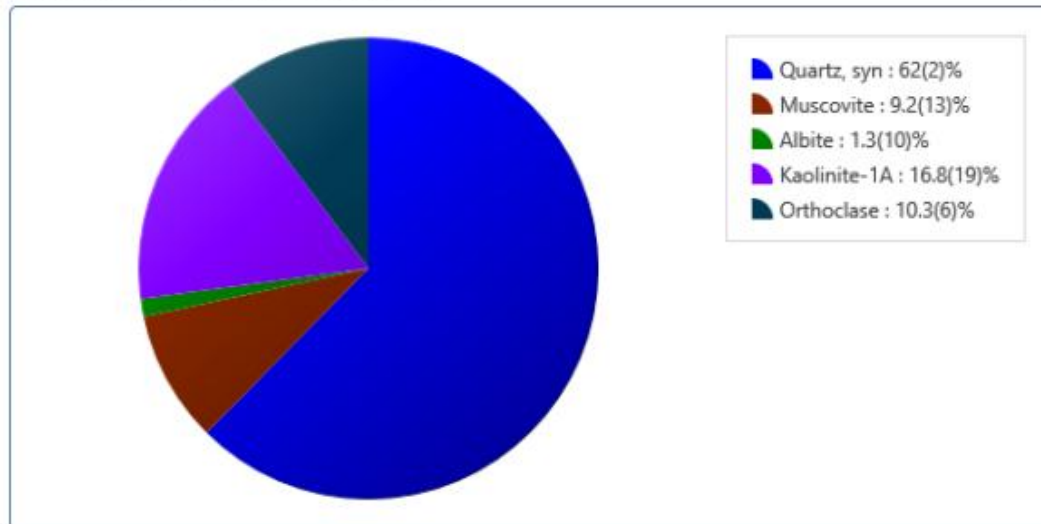


Figure 4.5a and 4.5b: X-ray diffractogram and Pie chat for UZ5

4.3 Mineralogical Analysis of Clay Samples

Table 4.5: The modal composition of clays from Uzebba

Major Minerals	Formula	UZ1	UZ2	UZ3	UZ4	UZ5	Average Value
Quartz	SiO ₂	51.00	59.00	56.00	64.00	62.00	58.40
Orthoclase	KAlSi ₃ O ₈	25.10	18.00	15.20	4.00	10.56	14.56
Muscovite	KAl ₃ Si ₃ O ₁₀ (OH) ₂	8.90	10.00	10.10	5.00	9.20	8.50
Albite	NaAlSi ₃ O ₈	2.00	0.90	0.80	16.00	1.44	4.20
Kaolinite	Al ₂ Si ₂ O ₅ (OH) ₄	13.60	12.10	17.90	11.00	16.80	14.44
TOTAL		100.00	100.00	100.00	100.00	100.00	

From the mineralogical analysis of the clay samples from Uzebba town, the following minerals were observed: Quartz had the highest percentage of 67.00% at UZ4, while UZ1 had the lowest percentage of silicon oxide in the clay sample, Orthoclase had its highest value of 25.10% at UZ1, while UZ4 as its lowest value of 12.60%.

For Muscovite, it was seen that UZ3 had the highest vales with 10.10%, while UZ4 had the lowest vales with 5.00%. No traces of Illite is obsevered in the clay sample.

Albite had the highest percentage of 16.00% at UZ4, while sample UZ3 had the lowest percentage of Albite in the clay sample with 0.80%. Kaolinite had its highest value of 16.80% at UZ5, with UZ4 as its lowest value of 11.6%.

For quartz, it was seen that it constituted the highest mineral in the clay samples with UZ4 having 64.00% and UZ1 having lowest value of 51.00%. Therefore, significant amounts of quartz occur in the clay rocks of the project site and it makes good sense to investigate the mineral as a possible co-product of the clay.

4.3.1 Discussion

From the mineralogical and geochemical analyses, it was observed that the clay samples have different characteristic and mode of formation. An analysis of the clay samples contains high amount of quartz with an average value of 58.40% which suggest that quartz is a major constitute of the clays in the study area.

The geochemical analysis of clay samples in Uzebba contain quartz, orthoclase, muscovite, and albite which undergoes chemical weathering to form clay. 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 suggests that the clays could be of

residual origin (Okosun, 2013). Quartz is needed in the production of adhesives and sealants, adsorbents, ceramic, porcelain, corrosion inhibitors, anti-adhesives, dyes and paint additives. In addition, silicon dioxide production occurs in agricultural chemicals. It is obtained as a transparent to grey, in its crystalline or amorphous powdered form. It is odourless and tasteless compound

Muscovite is one of the additional clay minerals that are also classified as fine-grained mica. While it's commonly known as a mineral that makes up the composition of a variety of rocks, muscovite is a standalone material. It comes with its own properties and uses that are worth noting (Okpiafoh, 2016). The muscovite value was seen to be in good amount. Orthoclase, a common alkali feldspar, usually occurs as variously coloured, frequently twinned crystals in granite. Orthoclase is used in the manufacture of glass and ceramics; occasionally, transparent crystals are cut as gems. Therefore, the clay samples is suitable for its use as ceramics and glass.

Albite, common feldspar mineral, occurs most widely in pegmatites and felsic igneous rocks such as granites. It may also be found in low-grade metamorphic rocks and as authigenic albite in certain sedimentary varieties. However, the rock from the same locations showed it to be mafic igneous in nature with acceptable Albite percentage. Prominent basal reflections, strong and sharp peaks are indicators of moderate to well-formed crystalline mineral components. The sample studied showed brooding of kaolinite reflection suggesting the presence of poorly ordered kaolinite (Semiz 2017). Clays often contain considerable amounts of organic matter just as the samples obtained from Uzebba which also influences their firing properties (Osayuki, 2016).

4.3.2 Potential industrial value of the clay deposits

The assessment of the clay deposits as potential raw materials was achieved by a comparative analysis of the chemical composition of the clay deposits in the study area with those of some notable clays and industrial specifications of some clays in other areas (Tables 4.1, 4.2 and 4.4). It is observed that the average chemical compositions of the studied clay deposits were similar to the Maastrichtian clay of Bida Basin as reported by Olushola *et al.*, (2011) and Afikpo shale as reported by Arua and Onyeok, (1978) in terms of silica, alumina, iron oxide and calcium oxide content.

The clay samples in the study area were also compared with the Afam clay as reported by Jubril and Amajor (1991). The Afam clay was observed to be less enriched in silica than those from the study area. The study sample from Uzebba was observed to be less in Fe_2O_3 and CaO with the study area showing a relatively lower iron and calcium oxide content than the Afam clay. The clay from the study area also compared similarly with Dukku clay from Kebbi State as reported by Salihu and Suleiman (2018), however, Al_2O_3 and CaO were high in comparison with standard chemical specifications for the requirement of kaolin to be used for refractory bricks (Parker, 1967). The average chemical compositions of the sampled clay deposits in Uzebba are fairly suitable for refractory bricks and ceramics production but there will be appreciable beneficiation in some oxide so as to meet the full requirement Table 4.4. Appreciable amounts of oxides of sodium, calcium and magnesium recorded in the deposits would also lower the vitrification of the clays (Obribe. 2012).

Comparison of this average study with other industrial application in Table 4.4, it is deduced that this study sample can also find application in refractory bricks (Parker, 1967) in terms of its silica, alumina, lime content. The high iron oxide may pose a problem but can be beneficiated to meet with the standard requirement for refractory bricks. The industrial

specification for ceramics (Singer and Simla, 1971) reflects the oxide requirement for clay sample in ceramics- The chemical data from the clay sample in this study area correlated with the specification for ceramics (Singer and Sunja, 1971) emphasized on the need for reduction in Iron oxide content. In correlation with other ceramic specification, there is also a compositional deficiency in iron oxide content so therefore the iron oxide content of this study needs to be beneficiated to meet the ceramic requirement.

CHAPTER FIVE

SUMMARY, CONCLUSION AND SUGGESTION FOR FURTHER STUDIES.

5.1 SUMMARY

Clay samples were collected from Owan East Local Government Area of Edo State with the aim of determining the geochemical and mineralogical analysis of clay from Uzebba and environs to determine their suitability for ceramic and refractory purposes. This was achieved using XRD and XRF methods. The analysis showed that the Uzebba clay is kaolinitic in composition. Table 1 shows the chemical composition of the major oxides of the clays in the study area. Table 2 shows the origin, quality and major minerals present in the sample clay. Table 3 disclose the chemical composition of some known reference clay samples. Table 4 divulge the Standard Specifications of the Concentrations in (wt.%) of Oxides in Clays for various Industrial Uses. Table 5 shows the modal composition of clays from Uzebba which confirms that it's a kaolinitic clay.

Nigeria is known to have over 710 million metric tons of clay deposit which is majorly seen around the southern western parts of the country. This huge amount of clay deposits is believed to meet the increasing demand for clay in the country.

The clay samples were seen to be with the accepted range when compared with clay samples from other locations across the country

5.2 CONCLUSION

The clay is a part of the Benin Formation, which stretches beyond the current coastline to the south and west throughout the whole Niger Delta region. The varieties of clay at Uzebba and environs is kaolinitic in composition. From the XRD analysis the dominant mineral is quartz (51-64%). Uzebba clay is used for ceramics, glass, refractive bricks etc.

5.3 SUGGESTION FOR FURTHER STUDIES

- i. Further geochemical analysis should be carried known if they can be exploring for commercial purpose.
- ii. Further studies should be done on the engineering properties of both clay and its suitability in the construction industries.

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