

**GEOCHEMICAL AND MINERALOGICAL COMPARISON OF
SOME METASEDIMENTARY ROCKS IN OFU-EBA AND
ENVIRONS, SOUTHWESTERN NIGERIA**

BY

**OGHENEFEGA ISAAC
PSC2008252**

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CERTIFICATION

This is to certify that this project was carried out and researched by Oghenefega ISAAC of the Department of Geology, Faculty of physical sciences, University of Benin, Edo State.

Dr. Mrs. ODOKUMA-ALONGE
(Project Supervisor)

Date

Date

DEDICATION

I dedicate this work to God Almighty for his divine guidance and wisdom. I also thank my parents, MR and MRS ISAAC for their loving support and my siblings and friends for being sources of strength and encouragement. This project s dedicated to you all.

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To my friends, thank you for your camaraderie, understanding, and for being a source of strength during this journey.

And finally, to myself: "Even when the path seemed shrouded in shadow, I never gave up on pushing past my limits, like a protagonist rising to meet their destiny! For believing in my own potential, I acknowledge the strength within."

ABSTRACT

This study examines the geochemical and mineralogical compositions of five meta-sedimentary rock samples from Ofu-Eba, Southwest Nigeria, focusing on marble, metaconglomerate and quartzite. One sample was taken from the Ugbogbo area (UGB5), two from the Ofu-Eba area (EBA3 & EBA4), and two from the Ogbe area (OGB1 & OGB2). Marble (OGB1 & OGB2) had very high CaO (87-88%), which set it apart from metaconglomerates (EBA3 & EBA4), which were enriched in SiO₂ (61-73%) and Al₂O₃ (7-11%), and quartzite (UGB5), which showed high SiO₂ (70%) and K₂O, according to geochemical data (Table 2). These results were supported by mineralogical investigations (Table 1), which showed a carbonate protolith in marble samples with calcite (28–37%) predominating and substantial quartz (35–36%) and orthoclase (27–29%). A siliciclastic origin was suggested by the metaconglomerates' high quartz content (36–38%), feldspars (orthoclase 18–22%, anorthite), and mica (biotite, muscovite). The matrix of quartzite was primarily composed of quartz (63%), which is indicative of a metamorphic rock that is extremely siliceous. These variances show a distinct compositional divergence between the silicate-rich metaconglomerates, the highly siliceous quartzite, and the carbonate-rich marble, reflecting variations in protolith composition and metamorphic grade. The results show the various metamorphic processes that have sculpted these meta-sedimentary rocks, which advances our understanding of the geological evolution of the Ofu-Eba region.

TABLE OF CONTENTS

LIST OF PLATES

LIST OF TABLES

1: The Quantitative Mineralogical Composition	32
2: The Chemical Composition of Major Oxides Within the Area	33

LIST OF FIGURES

- 1.1. Location Map of Study Area
- 2.2. Geological Map of the Study Area
- 3.1. Bragg's Law Diagram

CHAPTER ONE

GENERAL STATEMENT

Nearly equal amounts of crystalline and sedimentary rocks fill Nigeria's 923,768 sq km land. The three primary VIZ groupings of crystalline rocks are as follows: i) the Basement Complex; ii) the Younger Granites; and iii) Tertiary- Recent volcanics.

The Nigerian Basement Complex is situated between the Congo and West African Cratons and is a component of the Pan-African mobile belt. Known as the younger meta-sediments or the schist belts, the Basement Complex of Nigeria is made up of migmatite and migmatic gneisses, slightly migmatized to unmigmatized para schist intercalated by meta and non-meta igneous rocks, and the Older Granites suits, which are primarily composed of various granitic rock types, charnockites (hypersthene granite), syenites, and minor gabbroic and dioritic dykes.

The most common are the migmatized gneissic complexes, which make up around 60% of Nigeria's entire land area (Rahaman and Ocan, 1978). The younger sediments, known as the schist belt, are N-S trending synformal troughs that have been folded into migmatite-gneiss complexes, which are most developed in the country's west. Pelites, semi-pelites, and quartzites are the three most significant lithologies. The structural pattern of the basement rocks can be inferred from these rock suites. Most of the time, there are fault boundaries around the contact between the migamites and meta-sediments (Odeyemi *et al.*, 1999).

1.2 LOCATION MAP OF STUDY AREA

The approximate coordinates of the research region are $7^{\circ}17'0''\text{N}$, $6^{\circ}6'0''\text{E}$ (Figure 1.1). It is encircled by the Kukuruku Hills and is renowned for its rugged topography. The headquarters of the Akoko Edo Local Government Area in Edo State, Nigeria, is located there and is also referred to as Etuno. With a diverse range of rocks, such as migmatite-gneiss complexes, metamorphic rocks (such as metaconglomerate, schist, quartzite, and phyllite), calc-silicate rocks, and sedimentary formations, the region is a geologist's dream come true.

1.3 AIM AND OBJECTIVES

Determining the petrological and compositional characteristics of the metaconglomerates, marble, and quartz mica schist in Efu-Eba and the surrounding area is the goal of this study. Among the objectives are

1. To use X-ray fluorescence (XRF) investigation to ascertain the rock's chemical makeup
2. To use X-ray diffraction (XRD) examination to ascertain the rock's mineralogical makeup.
3. To supplement current studies on the Efu-Eba underground complex and its surroundings.
4. To compare some metasedimentary rocks in the area

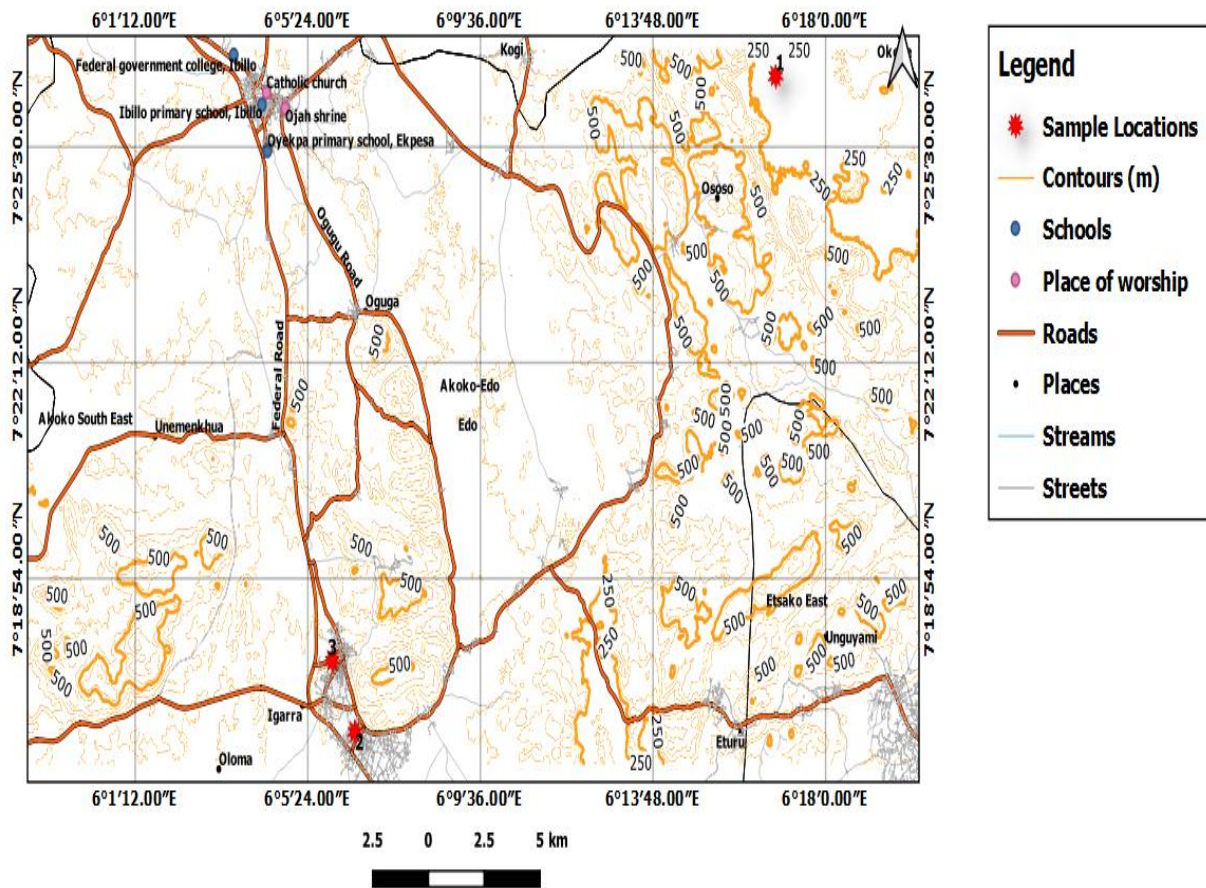


Figure 1.1: Location Map of Study Area

1.4 ACCESSIBILITY

The area's main highway connects Auchi, Sobe Ogbe, Ikpeshi, Igarra, and Ibillo. The experiment was conducted using both the old and new roads as access points. There are other important pathways as well. A system of major and local roads, as well as footpaths, provide access to the area. New outcrops were observed next to walkways and small roadways. As field assistants, a few villagers were incredibly beneficial. Jacob's staff and cutlass were used to gain entry to inaccessible areas.

1.5 TOPOGRAPHY

Ofu-Eba relief in the form of an elongated granite ridge that extends from Igarra to Aiyetoro. This ridge, also called the Igarra pluton, rises to a height of roughly 1550 meters above sea level. The quartzite and its surroundings have distinctive topography. In the northeast, there is a noticeable ridges, which have a maximum elevation of around 1100 feet, are located on the western edge of the region. The lowlands are home to schist and meta conglomerates, which are situated at an elevation of roughly 650 meters above sea level.

1.6 CLIMATE, VEGETATION, AND SOIL

Igarra's climate and those of its surroundings are classified as warm, horrid tropical, with distinct wet and dry seasons. November through February is the dry season, and April through October is the rainy season. With temperatures as high as 36.7°C, the average rainfall ranges from 1000 to 1500 mm (Udo, 1970).

The study location, Igarra and its environs fall into the Guinea Savannah vegetation zone. Vegetation here is prominently made up of sparsely distributed trees, plants, shrubs, and grasses. The vegetation in this area is primarily secondary ie. the natural vegetation is being transformed and such agricultural products such as maize, yam, cocoa, cassava etc. are cultivated. Silty sand and silty clay units are the dominating soil types within the research area These soil types are a consequence of the weathering of the granitic complex rocks and schistose rocks, respectively. Combined with the meteorological circumstances, these soil support farming and animal grazing which is the main profession of the natives of the area investigated.

1.7 HUMAN ACTIVITIES

The predominant activity of the population of Igarra and its environs is mostly subsistence farming and the major crops produced are yams, cassava and pineapples etc. Most of these farming activities are carried out in the lowlands which in most cases have loamy soils with high water table. The process of bush burning is followed by hunting of bush animals by the indigenes. Some of the farmers manufacture palm oil in tiny quantities from the palm plants. There is local quarrying of rocks such as granites and quartzites for construction work by the residents

CHAPTER TWO LITERATURE REVIEW/GEOLOGICAL SETTING

2.1 GENERAL GEOLOGY

Nigeria landmass constitutes part of the Pan African Shield and is located between the West African Craton to the east. The country lies between latitudes 6° N and 12°N and longitudes 4°E and 12°E. The West African Craton and Pan African events present igneous/metamorphic structural framework of Africa comprised of Precambrian rocks that have been subjected to supra crustal plutonics. The Basement Complex of Nigeria lies to the rest of the West African Craton in the zone of late Paleozoic orogenesis. The crystalline rocks are classified into two age groups: Precambrian and Jurassic Younger Granites. The sedimentary sequences range in age from the Cretaceous to the Quaternary and are distributed among five sedimentary basins, as shown in (Fig. 2.1).

The Basement Complex stretches westwards and is contiguous with the Dahomeyan of the Dahomey-Togo-Ghana region. To the east and south, the Basement Complex is covered by the Mesozoic-Recent of the Dahomey and Niger coastal basins. The Nigerian basement rocks are exposed in the north central, south western and eastern part of the country. Usually the basements are separated by sedimentary rocks except in the north central where they are continuous into each other (Rahaman and Ocan, 1978).

The basement rocks are estimated to represent the outcome of at least four main orogenic cycles of deformation, metamorphism and remobilization corresponding to the Liberian (2,700 Ma), the

Eburnean (2,000 Ma), the Kibaran (1,100 Ma), and the Pan-African cycles (600 Ma). (Dada, 2006). The first three cycles were marked by severe deformation and isoclinal folding accompanied by regional metamorphism, which was subsequently followed by significant migmatization. The Pan-African deformation was accompanied by a regional metamorphism, migmatization and significant granitization and gneissification which generated syntectonic granites and homogeneous gneisses (Abaa, 1983). Late tectonic emplacement of granites and granodiorites and related contact metamorphism accompanied the closing phases of this last deformation. The end of the orogeny was marked by faulting and fracturing (Gandu *et al.*, 1986; Olayinka, 1992)

2.2.MAJOR GEOLOGICAL UNITS

The geology of Nigeria is made up of three primary geological components:

1. Basement Complex: Pan-African and Older (Precambrian) > +600 million years
2. Younger Granites: Jurassic 200-145 million years
3. Sedimentary Basins: Cretaceous to Recent 145 million years

The Nigerian Basement Complex forms a section of the Pan-African mobile belt and sits between the West African and Congo Cratons and south of the Tuareg Shield (Black, 1980). Within the Basement Complex of Nigeria four primary petro-lithological units are identifiable as indicated by Dada, (2006).

1. The Migmatite-Gneiss Complex (MGC)
2. The Schist Belt (Metasedimentary and Metavolcanic rocks)
3. The Older Granites (Pan African granitoids)
4. Undeformed Acid and Basic Dykes



Fig 2.1: The regional geologic setting of the Precambrian Basement Complex of Nigeria (Malomo *et al.*, 2004)

2.2.1. The Migmatite - Gneiss Complex (MGC)

About 60% of the Nigerian basement's surface area is made up of the Migmatite-Gneiss Complex, which some researchers also refer to as the "migmatite-gneiss-quartzite complex" (Rahaman and Ocan, 1978). According to Rahman and Lancelot (1984), these rocks document three major geological events. The first, at 2,500 Ma, involved the beginning of crust-forming processes (such as the banded Ibadan grey gneiss of mantle origin) and crustal growth through sedimentation and orogeny. The Eburnean, at 2,000–200 Ma, was followed by the Ibadan type granite gneisses. Rocks from the Migmatite Gneiss Complex cover a large portion of northern, western, and eastern Nigeria. According to Rahman and Lancelot (1984), these regions include, but are not limited to: Ibadan, Ile-Ife, Akure, Ikerre (in western Nigeria); Obudu and the Oban Massif areas in eastern Nigeria; and Abuja, Keffi, Akwanga, Bauchi, Kaduna, Kano, Funtua, Okenne, Egbe, and Ajaokuta (in northern Nigeria).

Nigeria, which is part of the Pan African mobile belt, and a sizable portion of West Africa exhibit signs of the Pan African Orogeny (Turner, 1983). As stated by Ajibade and colleagues (1979). Because it has experienced numerous orogenic processes that were accompanied by deformation and metamorphism, Nigeria's Precambrian basement complex is polycyclic in nature. The Migmatite-Gneiss Complex, a series of Upper Proterozoic metasediments (Igarra Schist Belt), and late-tectonic porphyritic granite, granodiorite, and syenite are the three main rock groups that surround the study area (Odeyemi, 1988; Odeyemi and Rahaman, 1992). The low- to medium-grade metasediments of the Igarra Schist Belt were infolded and unconformably deposited over the basement (*sensu stricto*) formed by the local gneisses and migmatites.

2.2.2. The Schist Belt (Metasedimentary and Metavolcanic rocks)

Coarse to fine-grained clastics, pelitic schists, phyllites, banded iron formation, carbonate rocks (marbles/dolomitic marbles), and mafic metavolcanics (amphibolites) are some of the lithological variants of the schist belts. The schist belts are seen by Oyawoye (1972) and McCurry (1976) as remnants of a single supracrustal cover. The schist belts are fault-controlled rift-like formations, according to Olade and Elueze (1979). According to structural and lithological correlations, Grant (1978), Holt (1982), and Turner (1983) propose that sediments are of varying ages. Ajibade *et al.*, (1979), however, dispute this finding and demonstrate that the deformational histories in the two series were identical. Truswell and Cope (1963) regarded the structural links between the basement and the schist belts as conformable metamorphic fronts, while Ajibade *et al.*, (1979) were the first to trace a structural break.

Both basement rocks and sedimentary basins in Nigeria include marble and limestone (Fatoye and Gideon, 2013). The schist belts in the western half of Nigeria, west of longitude 80E, contain the Precambrian limestone that has been re-crystallized to create marble. When comparatively pure, they are white and grayish. Mica, calc-silicates and occasionally tiny inclusions of gneiss, pegmatite, and quartz are present in varying proportions, along with reaction products.

The carbonate unit of the newer sedimentary Precambrian rocks in the Ubo region of Nigeria's Igarra schist belt is known as the Ubo Marble. A body of biotite schist lies beneath it and forms its eastern edge. In their thorough economic analysis of the marble, Arcelloni and Maranaza (1965) described it as a pure calcitic marble with a purity rating of up to 99.7% CaCO₃.

Streams and rivers to the west of the Study Areas contain several marble outcrops connected to calc-silicate gneiss and phyllite. According to Odeyemi (1988) and Limah *et al.*, (2016), the Igarra Schist Belt's marble deposits were formed during a period of metamorphism from very

pure (silicate-poor) limestone to argillaceous limestone, dolostone, and/or protoliths, along with migration brought on by later deformation, most likely during the Pan-African age.

The most visible example of Pan-African orogamy is seen in the Older Granites, which show substantial material additions to the crust—up to 70% in some locations (Rahaman, 1988). Contact characteristics between members of the Older Granites indicate the coexistence of many magma, and attempts to categorize them based on chronology during an orogenic event are only valid over short distances (Rahaman, 1988).

2.2.4. Undeformed Acid and Basic Dykes

Without Deformation Post-tectonic Pan African acid and basic dykes are late to form. They are thought to be the youngest of the basement rocks and cross the Schist Belts, the Migmatite-Gneiss Complex, and the Older Granites.

2.3. Local Geology of Study Area

The Akoko area of Edo State, Southwest Nigeria, has a polyglot of mining activity due to the remarkable significant deposits of marble and calc-gneiss in the Ubo and Igarra areas. Nonetheless, the Igarra marble has a high LOI (43wt%) composition and a low CaO (53.06wt%) and SiO₂ concentration. The crystalline basement complex of rocks makes up the majority of the study region. Nigeria's Precambrian basement structure is characterized as polycyclic, meaning it has experienced several orogenic processes, together with metamorphism and deformation (Ajibade *et al.*, 1979). The Pan African Orogeny is the most important orogenic event that had an impact on the basement rocks. Earlier structures within the basement rocks

were extensively overprinted and obliterated as a result of this event (Fitches et al., 1985).

Figure 2.2: Geological map of the study area (NGSA, 2015)

The region's northeastern region contains marble, and there may be active quarries all over (Ailegbo et al., 2021). According to Odokuma Alonge, (2019), the marble in the study area has a high calcite classification with $\text{CaO/MgO} > 12$.

The marble in Ubo, as depicted in Plate 2.1, is categorized as non-foliated rocks, albeit some are foliated. Its colors range from whitish to pinkish to gray, and its textures range from fine to medium to coarse. In certain places, banded calcigneis connects the marble within the schist rock unit.

The Basement Complex in the study area is home to seven main types of rocks. These rocks include the following:

1. Granites;
2. Quartzites;
3. Metaconglomerates;
4. Schist, which comes in two varieties: Migmatized schist, quartz-biotite schist
5. Metabreccia,
6. Pegmatite
7. Syenite

Along the northeastern edge of the research region, granites can be found as plutons. The metaconglomerates trend north-south and are primarily found in the southern part of the research area. The research area's northwest and northeast borders are where the schist that was migmatized in some places is found. The two types of quartzites—schistose and massive—form

broad, linear ridges inside the metasediments. There is only one place where the metabreccia exposure can be found, and it takes the form of tiny plutons. Since the Older Granites are intrusions, these rocks have been well examined in the study region by a number of writers (Okeke and Meju, 1985; Ajibade *et.al.*, 1987; Odeyemi, 1988; Ekwere and Ekwueze, 1991; Imeokaparia and Emofurieta, 1991; Ocan *et al.*, 2003)

Marble:

Marble is a metamorphic rock formed from the recrystallization of carbonate rocks, primarily limestone or dolostone, under conditions of heat and pressure. This process transforms the original sedimentary rock into a crystalline metamorphic rock.

Field Observations:

Colour: In the Ofu-Eba region, marble outcrops generally exhibit light colors, ranging from white to creamy hues, sometimes with darker streaks or patches due to impurities.

Texture: The rock displays a noticeable crystalline texture, with visible calcite grains that reflect light. Some outcrops show a slightly layered or banded appearance, indicating foliation.

Mode of Occurrence: Marble occurs as discontinuous outcrops, often forming low ridges or rounded hills. Fractures and veins filled with secondary calcite are common.

Grain Size: The calcite grains are typically medium to coarse-grained.

Grade of Metamorphism: Low to medium-grade. The recrystallization of calcite and the potential presence of accessory minerals like quartz and feldspars suggest moderate temperatures and pressures.

Economic Importance: Marble is widely used as a building and ornamental stone due to its aesthetic appeal and work ability. It is also used in the production of lime and cement. High-quality marble deposits can be economically significant.

Metaconglomerates:

Metaconglomerates are metamorphic rocks derived from sedimentary conglomerates, which are composed of rounded clasts of varying sizes cemented within a matrix. Metamorphism alters the matrix and can affect the clasts, resulting in a rock with a metamorphic fabric but still retaining some of its original sedimentary features. Plate 2.1 shows an Outcrop of Metaconglomerate with Granitic Clast at Efu-Eba, Igarra

Field Observations:

Colour: Metaconglomerates in Ofu-Eba exhibit a range of colors, often gray or brown, reflecting the composition of the original clasts and matrix. Weathered surfaces may have a reddish tint due to iron oxide staining.

Texture: The rock's texture is distinctly clastic, with easily visible rounded clasts of various sizes. The matrix surrounding the clasts appears denser and more compact, showing signs of metamorphic alteration.

Mode of Occurrence: These rocks occur as layers or lenses within the metamorphic terrain, often interbedded with other meta-sedimentary rocks.

Grain Size: The clasts vary in size from pebbles to cobbles, while the matrix is fine-grained, showing recrystallization.

Grade of Metamorphism: Low to medium-grade regional metamorphism. The presence of mica minerals and the metamorphic fabric of the matrix suggest moderate temperatures and pressures.

Economic Importance: While metaconglomerates themselves may not be of significant economic value, their presence can provide insights into the geological history of an area, which can be helpful for identifying other potentially valuable resources. Also the clasts themselves within the matrix could contain valuable minerals.

Quartzite:

Quartzite is a metamorphic rock composed almost entirely of quartz, formed from the metamorphism of quartz-rich sandstone. The metamorphic process causes the quartz grains to fuse together, creating a very hard and dense rock. Plate 2.2 shows An Outcrop of Quartzite Showing Quartz Vein at Ofu-Eba, Igarra

Field Observations:

Colour: Quartzite outcrops in the Ofu-Eba area are typically light-colored, ranging from white to light gray or pale brown. The consistent color reflects the rock's high quartz content.

Texture: The rock is extremely hard and dense, with a massive, non-foliated texture. The quartz grains are tightly packed and fused, giving the rock a glassy or vitreous appearance on freshly broken surfaces.

Mode of Occurrence: Quartzite forms prominent ridges or massive outcrops, indicating its resistance to weathering.

Grain Size: The quartz grains are fine to medium-grained, creating a uniform, tightly packed texture.

Grade of Metamorphism: Medium to high-grade metamorphism. The complete recrystallization of quartz and the dense, massive texture indicate high temperatures and pressures.

Economic Importance: Quartzite is used as a construction material, particularly for road construction and aggregate, due to its hardness and durability. It can also be used for decorative stone. High purity quartzite is used in the production of silicon and ferrosilicon alloys.



Plate2.1: An Outcrop of Metaconglomerate with Granitic Clast at Efu-Eba, Igarra



Plate 2.2: An Outcrop of Quartzite Showing Quartz Vein at Ofu-Eba, Igarra

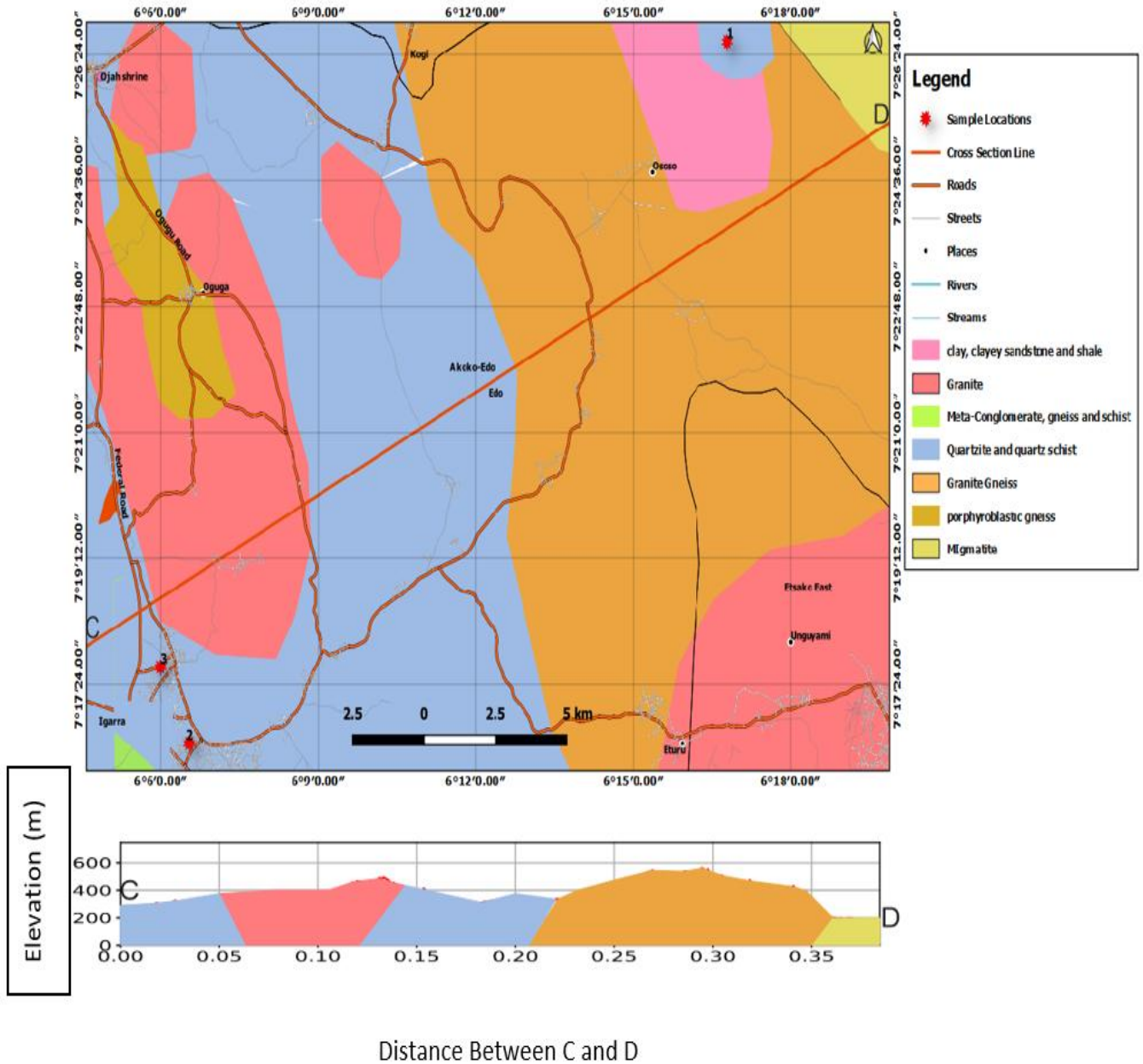


Figure 2.2: Geological map of the study area (NGSA, 2015)

2.4 Previous work

- Aderogbin and Isibor, (2020) carried out petrographic and geochemical investigations in order to evaluate the economic potential of Igbeti Marble in southwest Nigeria. Calcite (88.49%vol) made up the majority of the marble's composition, with lesser levels of dolomite (4.71%vol), pyrite (1.97%vol), muscovite (1.44%vol), tremolite (0.78%vol), quartz, and albite (2.81%vol) present. While MgO, CaO, and LOI were comparatively high, geochemical investigation revealed low amounts of SiO₂, Al₂O₃, Fe₂O₃, Na₂O, K₂O, and P₂O₅. The marble is not a suitable raw material, as indicated by the silica and alumina ratios falling below the acceptable levels for cement manufacturing. It might be improved, nevertheless, to satisfy the composition needed to produce cement.
- Alkali *et al.*, (2018) in "physio-Chemical and Mineralogical Characteristics of Obajana Marble Deposit: Implication for Economic Appraisal," investigated the physical, geochemical, and mineralogical characteristics of Obajana marble in Kogi, Nigeria. The marble's calcitic composition was indicated by its high CaO content and wide variety of physical features. Calcite made up the majority of it, with trace amounts of dolomite, quartz, iron, and magnesium oxide. According to the results, Obajana marble may be used economically in the production of cement, iron and steel fluxes, paint and paper fillers, glass, and water treatment.
- Chaanda et al., (2019) In "Environmental Geochemistry of Igarra Marble Mining District, Southwestern Nigeria" revealed that the marble contains high concentrations of **Cd, Co, Cu, Ni, Pb, and Zn**. High amounts of **Cd, Co, Cu, Ni, Pb, and Zn** were found in water samples taken from wells, streams, and taps; lead levels were also found to be beyond permissible limits. High levels of lead, nickel, and cadmium were discovered in both surface

and subterranean water, suggesting that the water must be treated before use. Protecting the health and welfare of the local populace should be the first priority when it comes to implementing mining rules and corporate social responsibility initiatives.

- Madukwe and Obasi,(2016). Low-grade metamorphic granoblastic rocks are what the study "Geochemical and petrogenetic characteristics of the marble deposit at Ikpeshi southern Nigeria" found. These include pure calcite marbles, pure dolomite marbles, pure calcitic dolomite, and pure dolomitic calcite. Most likely, the protolith was deposited in a salty, shallow environment. The marbles' dolomites are most likely the result of precipitation. While the Mn content is lower, most likely as a result of recrystallization at higher temperatures, the immobile Ni and Cr contents are similar to sedimentary carbonates. The paleo-oxygenation of the protolith depositional environment is typically oxic, with sporadic suboxic circumstances.
- Odokuma-Alonge, (2019) "A geochemical appraisal of some marble physiquies in Ubo Area and Environs, Southwestern Nigeria" have high CaO values (52.98wt.% to 82.18wt.%) and low MgO levels (1.64wt% to 6.95wt%). Every important oxide, including SiO₂, FeO/Fe₂O₃, Na₂O, K₂O, Al₂O₃, and MnO₂, was less than 2 weight percent. Multivariate analysis revealed strong relationships between CaO/Fe₂O₃, MgO/Al₂O₃, and FeO/CaO, indicating comparable ionic values and valencies. Pre-metamorphism deposition in a shallow sea environment is indicated by the amount of lime. Because of their high calcite content, these marbles are utilized in several industrial processes, including the creation of Portland cement and ceramics.
- Onimisi *et al.*, (2013) in "The geochemical investigations of the marble deposit in Itube. Kogi state, central Nigeria" and they proposed co-precipitation with calcium from hyper-

saline waters under anoxic circumstances, supported by microfauna and flora. Itobe marble and other metamorphic carbonate rocks are frequently encountered in continental settings where crustal distension is present. The marble deposit's connection with other rock types suggests that it was deposited in a rift setting where there was concurrent magmatism and fast subsidence, followed by sediment deformation.

CHAPTER THREE

MATERIALS AND METHODS

MATERIALS

The following were used for this study;

- Base map
- Compass clinometer
- GPS
- Cutlass
- Sampling bag
- Field notebook
- Acid bottle
- Peak hammer and sledge hammer
- Measuring tape
- X-ray Diffraction (XRD) Analyzer
- Petrographic microscope

3.1.2. Preparation of Samples

Following collection, five (05) samples were given the following labels: , OGB1, OGB2 (Ogbe area) EBA3, EBA4 (Ofu-Eba area) and UGB5 (Ugbogbo area). Before analysis, the samples were first ground in a disc mill to enhance their surface area and decrease their size. After being allowed to air dry to eliminate any remaining moisture, the samples were transferred to the

NSRMEA (National Steel Raw Material Exploration Agency). Laboratory in Kaduna State for investigation of X-ray Fluorescence and diffraction.

3.2.METHOD:

For this investigation, two analytical techniques (mineral and geochemical) were used:

3.2.1.X-ray Diffraction Method

An effective analytical method for examining a material's atomic and molecular structure and composition is X-ray diffraction (Zhang *et al.*, 2019). There are several crucial steps in the X-ray diffraction methodology:

a. Sample Preparation: Getting a representative sample of the material being studied is the first step. To obtain precise diffraction measurements, the sample needs to be homogeneous and finely powdered.

b. Equipment: An X-ray diffractometer is a specialized instrument used in X-ray diffraction investigations. An X-ray source, a sample holder, and a detector make up the diffractometer.

c. X-ray Source: This source shoots off X-rays with a particular wavelength, usually between 0.1 and 2.5 angstroms. A sealed X-ray tube is the most popular source; when a high voltage is applied across its electrodes, X-rays are produced.

d. Sample Alignment: The powdered sample is positioned in line with the incident X-ray beam after being carefully placed on the sample holder. This guarantees that the sample will be struck by the X-rays at the appropriate angle.

e. Measurement of Diffraction: The X-ray beam interacts with the sample's crystal lattice, causing the diffracted waves to interfere constructively and destructively. The detector collects the diffracted waves and logs the diffracted X-rays' intensity and scattering angle.

f. Data Analysis: The arrangement of atoms or molecules in the sample can be inferred from the recorded diffraction pattern, also called a diffractogram. Structural information is extracted from the diffractogram using sophisticated data analysis methods like Fourier transformation.

3.2.1a. X-ray Diffraction: How Does It Work? Work:

Wave interference is the basis for X-ray diffraction. The periodic arrangement of atoms or molecules in a crystal causes an incident X-ray beam to scatter in various directions when it interacts with the crystal lattice.

Bragg's Law states that when the path difference between the waves is an integer multiple of the X-ray wavelength, the scattered X-rays experience constructive interference. The following is an expression for this condition:

Since $n\lambda = 2d\sin(\theta)$, Equation (1), where θ is the angle of incidence, d is the interatomic or intermolecular spacing in the crystal lattice, n is the order of the diffraction, and λ is the wavelength of the incident X-rays. The orientation of the material's crystal planes and the distances between atoms or molecules can be ascertained by measuring the scattering angles and intensities of the diffracted X-rays. This data sheds light on the sample's crystal structure, crystallographic phases, and the existence of flaws or contaminants. Figure 3.1 shows Bragg's law diagram.

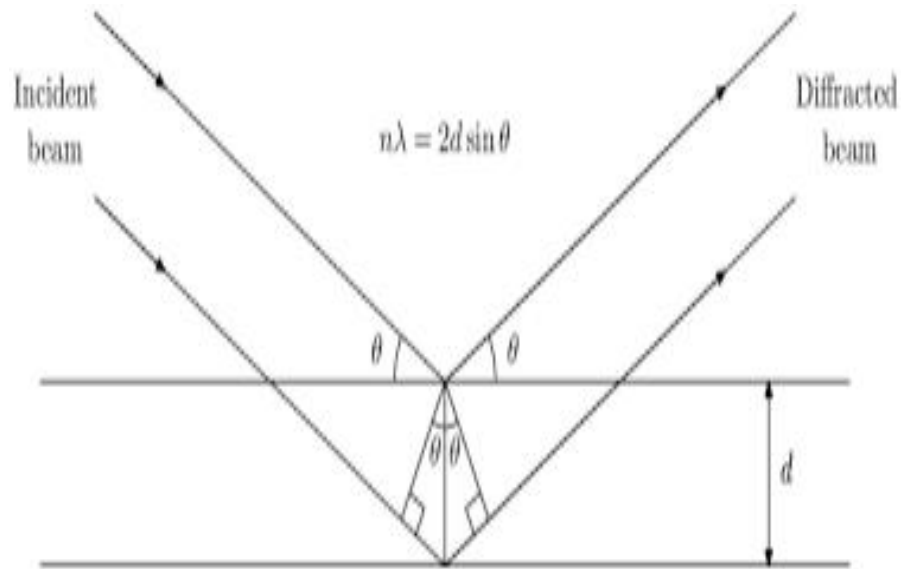


Fig 3.1: Bragg's law diagram

3.2.1b. Basic Principles of X-ray Diffraction

The following are the basic ideas of X-ray diffraction: Bragg's Law, developed in 1912 by Sir William Henry Bragg and Sir William Lawrence Bragg, is the fundamental idea behind X-ray diffraction. This law states that constructive interference between the scattered X-ray waves produces a discernible pattern called a diffraction pattern when an X-ray beam impacts a crystal lattice at a particular angle. Bragg's Law establishes a mathematical relationship between the diffraction angles that come from the angle of incidence, the interatomic spacing in the crystal lattice, and the wavelength of the incident X-rays.

When X-rays interact with a crystal, their wave nature allows them to behave like waves and display diffraction patterns (Putnam *et al.*, 2007). The angle of incidence and the path difference determine whether the diffracted X-rays interfere constructively or destructively. Researchers can ascertain the crystal structure, lattice spacing, and other characteristics by examining the resulting diffraction pattern. Plate 3.1 shows an XRD machine

3.2.1c. X-ray Diffraction Applications:

a. Materials research: To investigate the crystalline structure and characteristics of a variety of materials, such as metals, alloys, minerals, and semiconductors, X-ray diffraction is widely utilized in materials research. It helps with material characterization, phase identification, and quality control by offering insightful information on crystallographic phases, lattice properties, grain sizes, and crystal defects.

b. Crystallography and Structural Biology: The three-dimensional atomic structures of proteins, enzymes, and other biological macro-molecules are determined in large part by X-ray crystallography. Understanding their roles, interactions, and drug design requires knowledge of

these information. Additionally, the structure of tiny chemical molecules, medications, and coordination compounds can be examined using X-ray diffraction (Amch, 2019).

3.2.1d. Geology and Mineralogy:

X-ray diffraction is used in geology and mineralogy to help identify and describe different minerals found in rocks and sediments. In order to help with geological mapping, resource development, and environmental investigations, geologists can ascertain the mineral composition by comparing the diffraction patterns acquired from an unknown sample with published mineral databases.

3.2.1e. Pharmaceutical Analysis:

The pharmaceutical industry uses X-ray diffraction to examine the crystal structures of medicinal substances in both their bulk and final dosage forms. This aids in the research of polymorphism and solvates, which can impact medication formulation and performance, as well as the purification, stability, and bio availability of pharmaceutical goods.

3.2.1f. X-ray Diffraction (XRD) Strengths and Limitations

a. Strength

- i. A powerful and quick (less than 20 minutes) method for identifying an unknown mineral
- ii. It usually yields an unequivocal result
- iv. XRD devices are widely accessible
- v. Sample preparation is minimal. The interpretation of data is quite simple.

b. Limitation

- i. The most effective material for identifying an unknown is homogeneous and single-phase.

- ii. A standard reference file of inorganic substances must be available.
- iii. Needs tenths of a grain substance that needs to be powdered
- iv. The detection limit for combined compounds is 2% of sample volume.
- v. Pattern indexing for non-isometric crystal systems is challenging for unit cell determinations.

3.2.2. X-ray Fluorescence Spectroscopy

An X-ray device used for routine, comparatively non-destructive chemical investigations of rocks, minerals, sediments, and fluids is called an X-ray fluorescence (XRF) spectrometer. Similar to an electron microprobe, it operates on wavelength-dispersive spectroscopic principles. The stability and user-friendliness of x-ray spectrometers, along with the relative simplicity and affordability of sample preparation, make this one of the most popular analytical techniques were employed to determine the major and trace elements composition of rocks, minerals and sediments. Plate 3.2 shows an XRF machine

a. Sample Preparation: The sample must first be made ready for analysis. The material could be a liquid, solid, or powder. In order to guarantee representativeness and homogeneity, solid samples are frequently ground into a fine powder. Liquid samples may require dilution or drying to get the appropriate consistency, or they may be evaluated directly.

b. Equipment: An X-ray fluorescence spectrometer is a specialist tool needed for XRF examination. This device is made up of an X-ray tube that produces high-energy X-rays and a detector system that measures the distinctive X-rays that the sample emits.

c. X-ray Excitation: The X-ray tube produces high-energy X-rays that are exposed to the sample. Inner-shell electrons become energized and are expelled from their orbits when the X-rays impact with the sample's atoms, creating vacancies.

d. X-ray Emission: Outer-shell electrons descend to occupy the empty spaces left by the removal of inner-shell electrons. Characteristic X-rays with energy unique to each element are released during this process. The X-rays that are released will be examined.

e. X-ray Detection and Analysis: The spectrometer's detection system picks up the released X-

rays. A spectrum representing the intensities of various X-ray energies is produced by the detector. Certain elements found in the sample are represented by the distinctive peaks in the spectrum.

f. Data Analysis: Software is then used to process the acquired X-ray spectrum, comparing the intensities and peak positions with a database of established standards. The techniques identifies the elements in the sample and measures their exact amounts by analyzing the strength of the X-ray they produce.

3.2.2a Basic X-ray Fluorescence Analysis Concepts

The foundational ideas of the XRF method are shared by a number of different instrumental techniques that include interactions between electron beams and X-rays and samples, such as X-ray spectroscopy (e.g., SEM-EDS). The way atoms behave when they contact with radiation allows x-ray fluorescence to be used to analyze major and trace elements in geological strata. Materials may ionize when exposed to high-energy, short-wavelength radiation, such as X-rays. The atom becomes unstable and an outer electron takes the place of the missing inner electron if the radiation's energy is high enough to loosen a securely held inner electron. When this occurs, energy is liberated because the inner electron orbital's binding energy is lower than that of the outer one. Fluorescent radiation is the name given to the radiation that is released, which has a lesser energy than the original incident X-rays. The generated fluorescence X-rays can be used to determine the abundances of elements present in the sample because the energy of the photon that is released is characteristic of a transition between particular electron orbitals in that element.

3.2.2b HOW X-RAY FLUORESCENCE FUNCTIONS

The way atoms behave when they encounter with X-rays enables XRF to analyze the major and trace elements in geological materials. The reason an XRF spectrometer functions is because when an incident beam of X-rays shines on a sample, some of the energy is dispersed and some is absorbed by the sample in a way that is dependent on its chemistry. Usually, a Rh target produces the incident X-ray beam, but depending on the application, W, Mo, Cr, and other materials may also be utilized. The sample is said to be stimulated when this primary ray beam illuminates it. The heated sample then releases X-rays along a range of wavelengths that correspond to the different kinds of atoms that are contained within it. How does this occur? By ionizing and expelling electrons from the lower (often K and L) energy levels, the atoms in the sample absorb X-ray energy. Electrons from an outside, higher energy orbital take the place of the expelled electrons. When this occurs, energy is liberated because the inner electron orbital's binding energy is lower than that of the purer one. This energy release manifests as the emission of distinctive X-rays that reveal the type of atom in question. A wavelength disperse spectrometer similar to that used in an EPMA can be used to separate a complex emission X-ray spectrum into distinctive wavelengths for each element present in a sample that contains a large number of elements, as is common for the majority of minerals and rocks. The intensity of the emitted beam is measured using a variety of detector types, including scintillation and gas flow proportional detectors. For measuring long wavelength (0.15mm) X-rays, which are characteristic of K spectra from elements lighter than Zn, the flow counter is frequently used. Shorter wavelengths of the X-ray spectrum (K spectra of elements from Nb to I, L, spectra of Th and U) are frequently analyzed using scintillation detectors. Both detectors are typically used in tandem to measure intermediate wavelength X-rays, such as K spectra from Zn to Zr and L.

spectra from Ba and the rare earth metals. The element's abundance in the sample is directly correlated with the energy intensity detected by these detectors. Each element's precise proportionality value is determined by comparing it to standards of minerals or rocks whose composition has been determined by previous analysis using different techniques.

3.2.1c.Application of X-ray diffraction

The most common application of X-ray powder diffraction is the identification of unknown crystalline materials, such as minerals and inorganic chemicals. Geology, environmental science, material science, engineering, and biology all depend on the determination of unknown solids.

Additional uses include:

- i. Characterizing crystalline materials;
- ii. Identifying fine-grained minerals that are challenging to identify optically, such as clay and mixed layer clays.
- iii. Calculating unit cell measurements .
- iv.Purity measurement using specialized methods. XRD is useful.
- v.One hundred Retrieved refinement is used to determine crystal structures.
- vi. Quantitative analysis to determine the median quantities of minerals
- vii. Using rock curve measurements to determine the dislocation density and film quality
- viii. Super lattice measurement in multilayered epitaxial systems
- ix. Calculating the film's density, thickness, and roughness by glancing incidence X-ray reflectivity measurements
- x. Measure the texture of a polycrystalline sample, such as the grain orientation.

3.2.2c. X-RAY DIFFRACTION (XRD) STRENGTHS AND LIMITATIONS

a. Strength

- i. A strong and quick (less than 20 minutes) method for identifying an unidentified mineral
- iii. It usually yields a clear result;
- iv. Sample preparation is minimal;
- v. XRD units are generally accessible.
- v. Interpreting data is rather simple.

b. Limitation

- i. The best material for identifying an unknown is homogeneous and single-phase.
- ii. A standard reference file of inorganic substances must be available.
- iii. needs to be processed into a powder using tenths of a grain material
- iv. The detection limit for combined compounds is 2% of sample volume.
- v. Pattern indexing for non-isometric crystal systems is challenging for unit cell determinations.

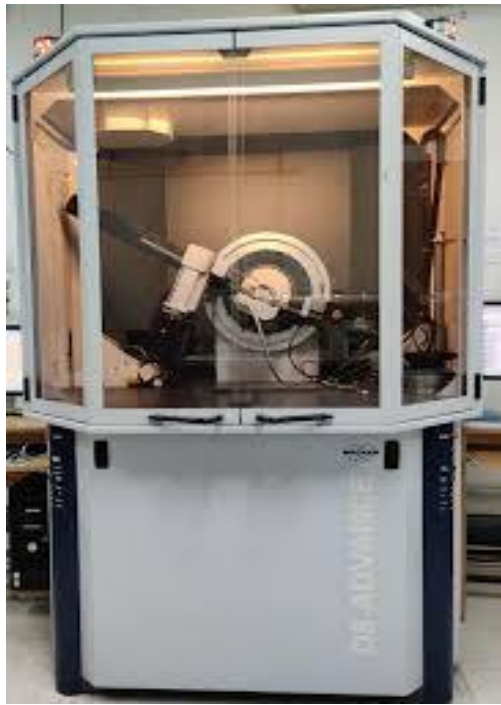


Plate 3.1: An XRD machine



Plate 3.2: An XRF machine

CHAPTER FOUR

PRESENTATION AND DISCUSSION OF RESULTS

4.1. PRESENTATION OF RESULTS

The mineralogical and geochemical findings are summarized in Table 4.1, 4.2 and Figures 4.1a-4.5b, which include X-ray diffractograms and pie charts illustrating mineral distribution.

Table 1: The Quantitative Mineralogical Composition (%)

Mineral name	Formula	OGB1	OGB2	EBA3	EBA4	UGB5
Calcite	CaCO ₃	28.00	37.00	---	---	---
Quartz	SiO ₂	36.00	35.00	38.00	36.00	63.00
Orthoclase	KAlSiO ₃	29.00	27.00	22.00	18.10	15.00
Rutile	TiO ₂	7.00	1.20	3.20	6.50	4.50
Albite	NaAlSiO ₃	---	---	---	---	5.00
Anorthite	CaAl ₂ (SiO ₄) ₂	---	---	18.00	15.00	9.00
Biotite	K(Mg,Fe) ₃ (AlSi ₃ O ₁₀)(F,OH) ₂	---	---	4.50	8.50	---
Muscovite	KAl(Si ₄ O ₁₀)(OH,F) ₂	---	---	---	---	3.00
Others		---	---	14.30	15.90	0.50

Table 4.2: The Chemical Composition of Major Oxides Within the Study Area (wt.%)

Major oxides	OGB1	OGB2	EBA3	EBA4	UGB5
SiO ₂	3.23	4.33	73.81	61.62	70.09
Fe ₂ O ₃	0.38	0.52	2.14	5.59	4.76
CaO	88.10	87.51	2.24	5.95	5.46
MgO	2.12	1.31	ND	0.49	ND
Al ₂ O ₃	3.98	3.84	7.30	11.23	9.23
TiO ₂	0.07	0.22	0.39	1.02	0.56
K ₂ O	0.09	0.09	1.28	2.43	4.59
MnO	0.03	0.03	0.05	0.17	0.08
TOTAL	98.00	97.85	87.21	88.5	94.77

ND = Not Detected

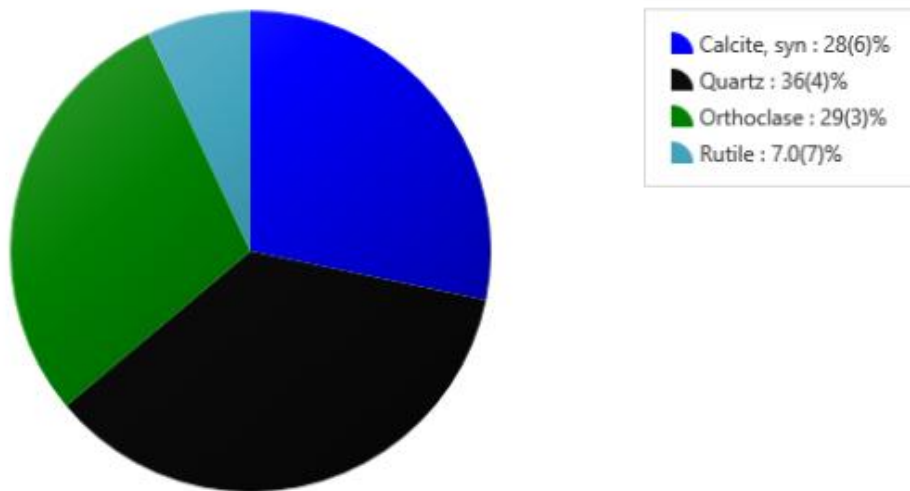
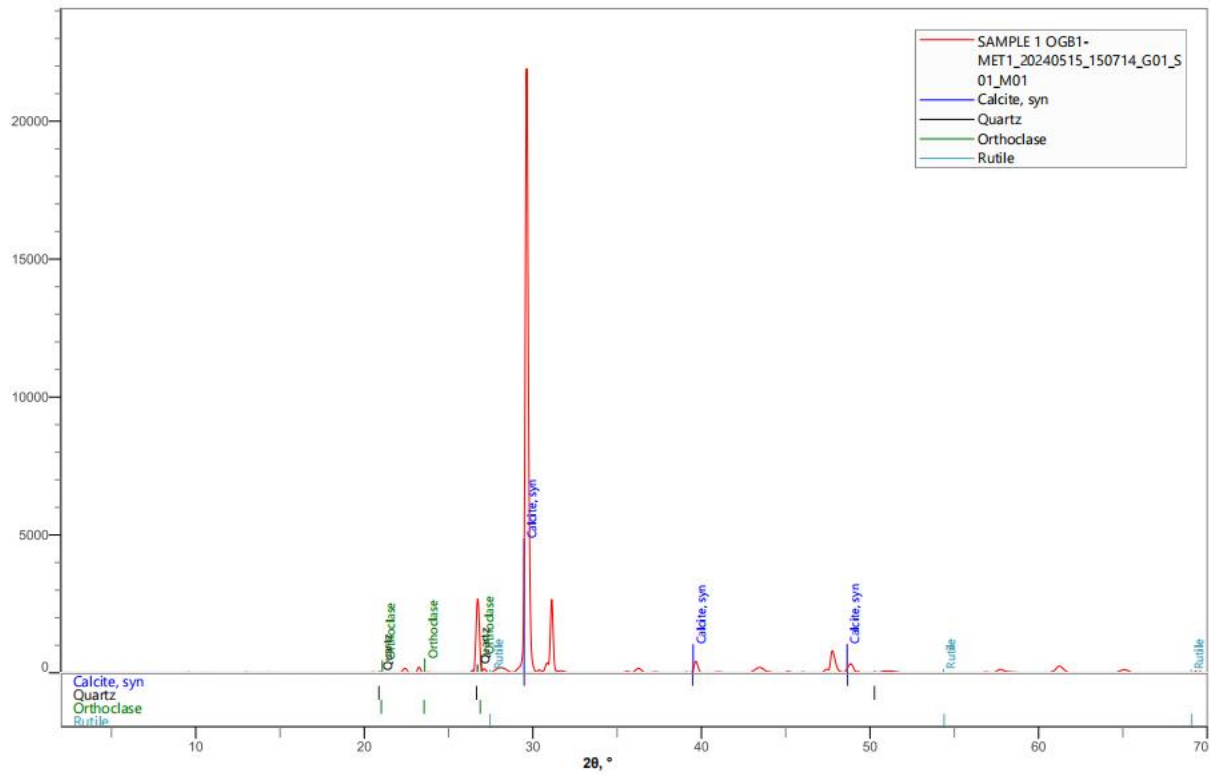


Figure 4.1a and 4.1b: An X-ray Diffractogram and Pie Chart Showing Mineral Distribution in

OGB1

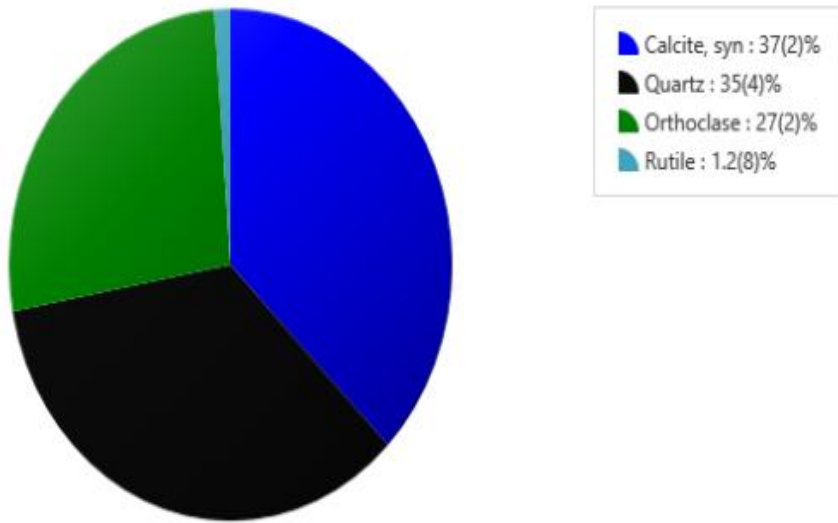
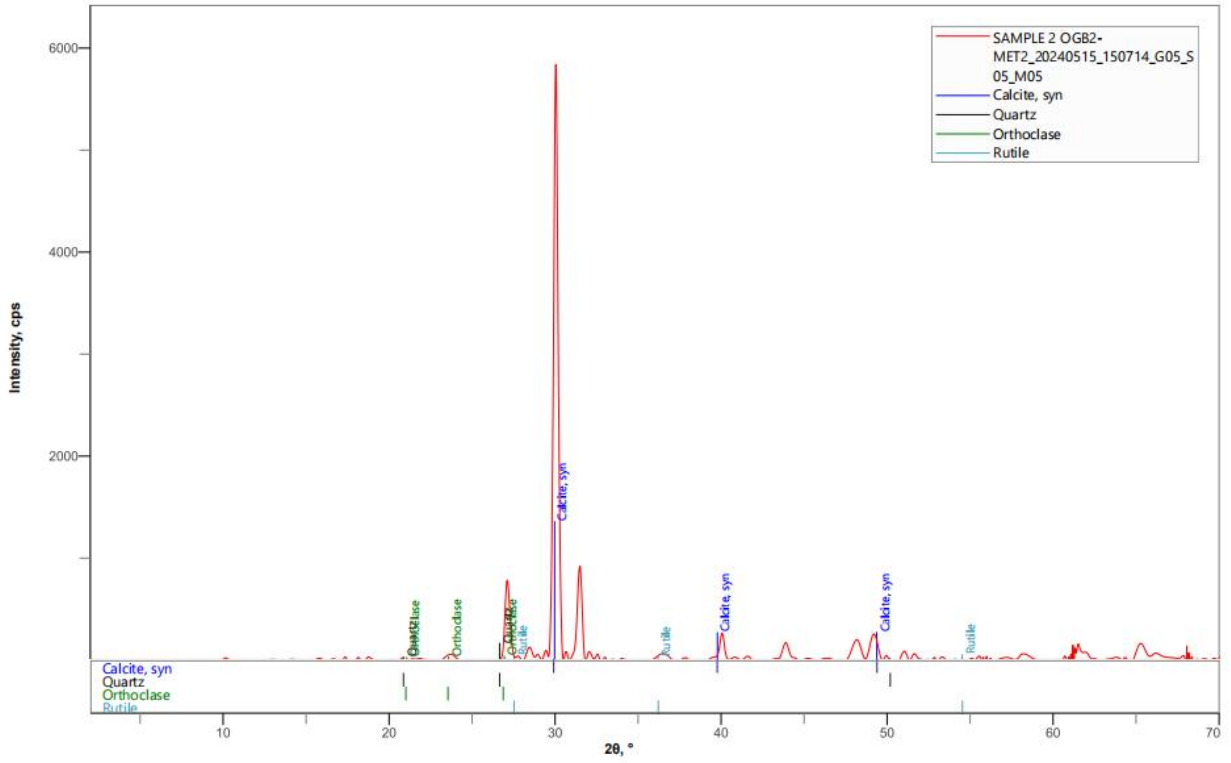


Figure 4.2a and 4.2b: An X-ray Diffractogram and Pie Chart Showing Mineral Distribution in OGB2

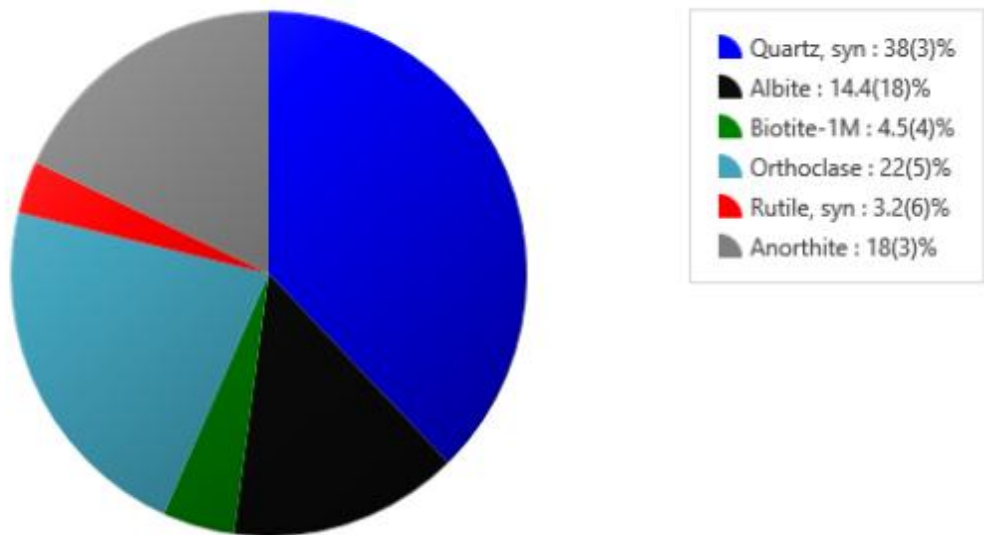
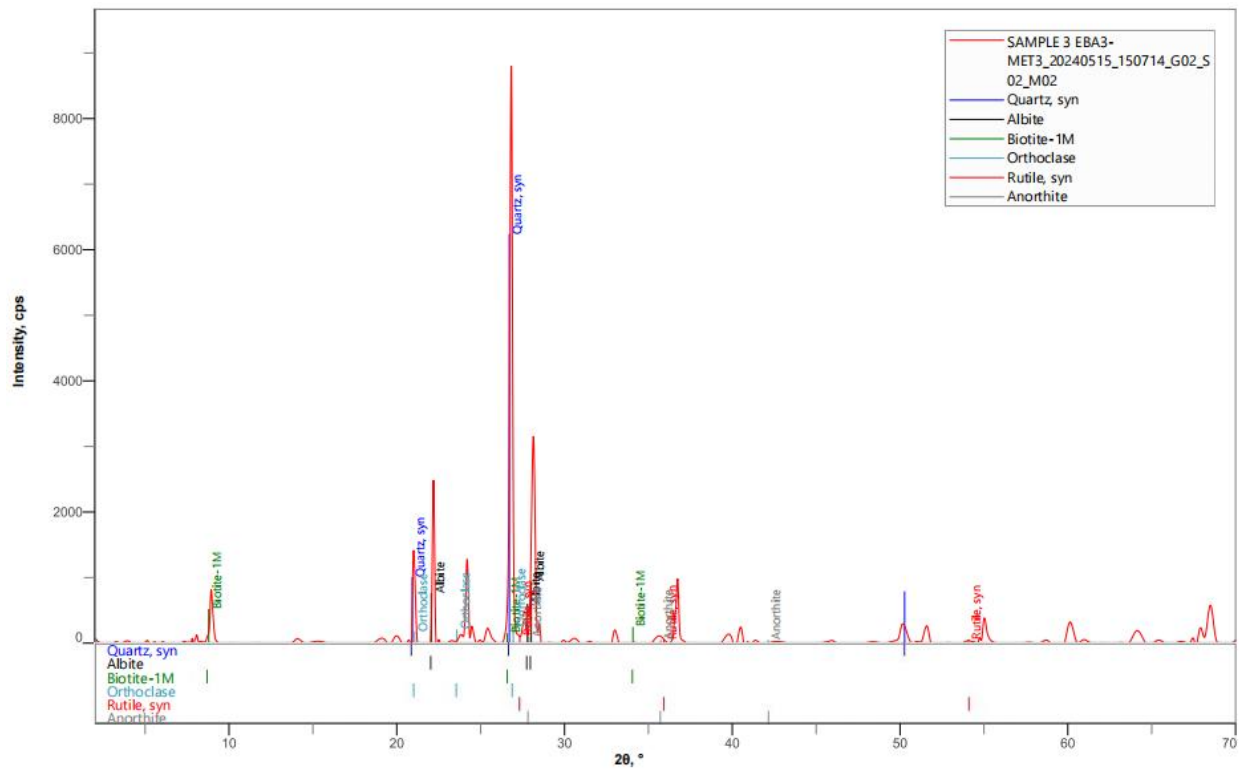


Figure 4.3a and 4.3b: An X-ray Diffractogram and Pie Chart Showing Mineral Distribution in EBA3

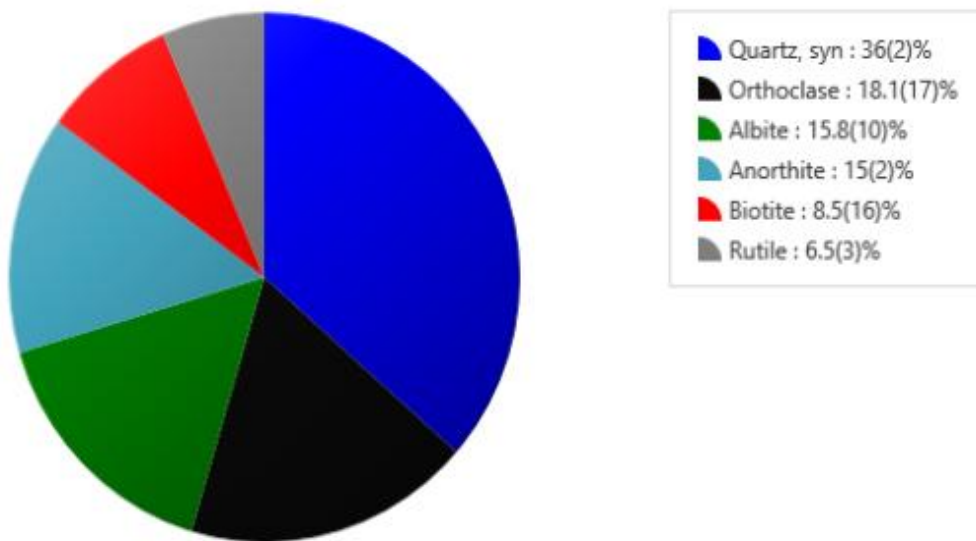
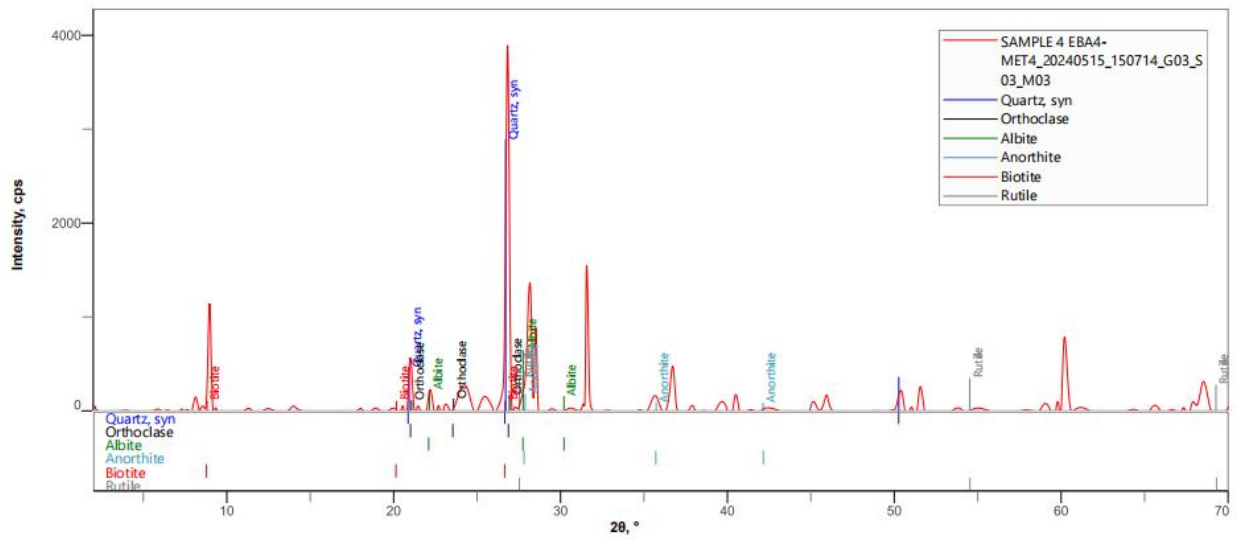


Figure 4.4a and 4.4b: An X-ray Diffractogram and Pie Chart Showing Mineral Distribution in EBA4

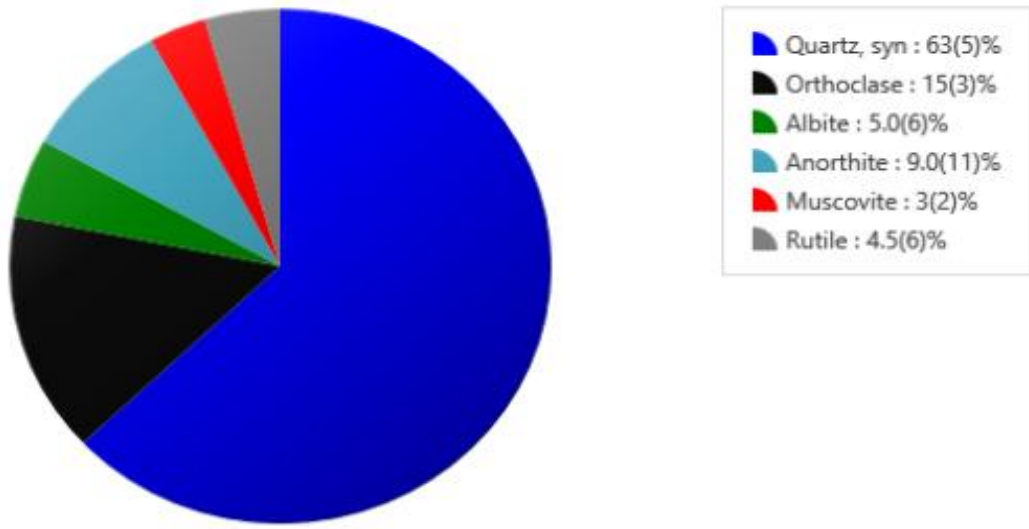
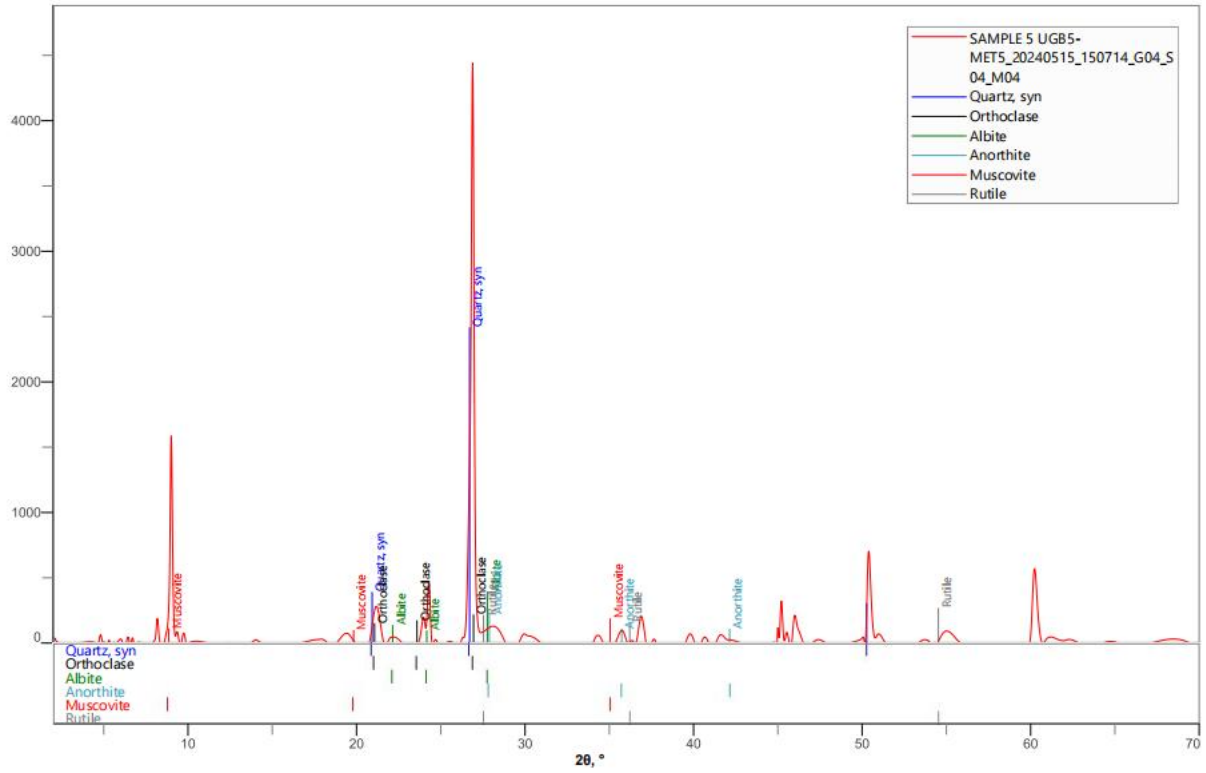


Figure 4.5a and 4.5b: An X-ray Diffraction Spectra and Pie Chart Showing Mineral Distribution in UGB5

4.2 DISCUSSION OF MINERALOGICAL AND GEOCHEMICAL RESULTS

This study looked at the geochemical and mineralogical makeup of meta-sedimentary rocks from Ofu-Eba, southwest Nigeria, including quartzite, metaconglomerate and marble.

4.2.1 OGB1:

The mineral composition of this sample shows that calcite (CaCO_3) makes up 28% of the total. Marble's distinguishing mineral is calcite, and its abundance strongly implies that it underwent metamorphism from a carbonate protolith, like limestone. The rock would have a distinctive crystalline texture due to the calcite crystals that interlock within it.

At 36% of the sample, quartz (SiO_2) is also present in considerable amounts. Although calcite makes up the majority of marble, the presence of quartz suggests that there may have been some silica impurities in the original limestone (). The calcite matrix would probably contain tiny, dispersed grains of this quartz.

A potassium feldspar called orthoclase (KAlSi_3O_8) makes about 29% of the sample. Orthoclase's existence indicates that clay minerals or other feldspars may have been present in the original limestone and undergone metamorphism.

Seven percent (7%) of the sample is made up of the metamorphic mineral rutile (TiO_2). Its existence indicates that metamorphism has occurred.

Five percent of the sample is made up of albite ($\text{NaAlSi}_3\text{O}_8$).

Other minor minerals, such as other feldspars, clays, or accessory minerals, probably make up the remaining 14.30%.

Geochemical Analysis

The mineralogical results are supported by the geochemical analysis. The remarkably high CaO (calcium oxide) concentration (88.10%) is the most notable characteristic. The quantity of the calcium carbonate mineral calcite is immediately reflected in this high concentration.

In line with a carbonate rock, the SiO₂ (silicon dioxide) percentage is low (3.23%).

In accordance with the mineralogical data, the low Al₂O₃ (aluminum oxide) percentage (3.98%) suggests little clay or feldspar.

The presence of magnesium oxide (MgO) at 2.12% may indicate that the initial protolith was a dolomitic limestone.

The extremely little amounts of other oxides further support calcium carbonate's supremacy.

4.2.2 OGB2

Mineralogy:

OGB2 is classified as marble because, like OGB1, it is primarily made of calcite (37%).

Even now, quartz (35%) remains a substantial component.

There is orthoclase (27%).

There's rutile (1.2%).

There is anorthite (18%).

Other minor minerals make up the remaining 15.90%.

Geochemistry Analysis:

With a very high CaO (87.51%), the geochemical data closely resembles that of OGB1.

At 4.33%, SiO₂ is marginally higher.

The presence of MgO is 1.31%.

3.84% is Al₂O₃.

4.2.3EBA3:

Mineralogy:

There has been a notable change in mineralogy with this sample. The predominant mineral, quartz (38%) suggests a siliciclastic protolith. These quartz grains are probably bonded together in the rock matrix.

There is still orthoclase (22%).

3.2% rutile is found.

There is 15% anorthite.

The presence of biotite (4.5%) and muscovite (3%) indicates that metamorphism has taken place.

Other minor minerals make up the remaining 14.30 percent.

Geochemical Analysis:

The higher quartz content is reflected in the much higher SiO₂ (73.81%).

CaO is 2.24%, which is relatively low.

7.30% is Al₂O₃.

2.14% is FeO.

1.28% is K₂O.

4.2.4 EBA4:

In minerals, quartz (36%) is still the most common mineral.

There is orthoclase (18.10%).

There's rutile (6.5%).

There is anorthite (9%) found.

There is biotite (8.5%).

Other minor minerals make up the remaining 15.90%.

Geochemical Analysis:

61.62% SiO₂.

5.95% is CaO.

11.23% is Al₂O₃.

5.59% is FeO.

2.43% is K₂O.

4.2.5 UGB5:

Mineralogy:

Metamorphic rocks rich in quartz have a preponderance of quartz (63%) in their composition.

There is 15% orthoclase.

There's rutile (4.5%).

Other minor minerals make up the remaining 0.5%.

Geochemical Analysis:

70.09% SiO₂.
5.46% is CaO.
9.23% is Al₂O₃.
FeO is 4.76%.
4.59% is K₂O.

3 4 COMPARISON AND JUSTIFICATION OF ROCK NAMES:

1. OGB1 and OGB2:

COMPARISON

Mineralogy:

Calcite (CaCO₃) makes up the majority of the mineral composition, with notable amounts of quartz and orthoclase. Metamorphic minerals are present.

Geochemistry:

Low SiO₂, Al₂O₃, and other oxides, and extremely high CaO.

Justification for the name “Marble”

Marble is distinguished by its high calcite concentration, which is evident from the mineralogical data (28–37% calcite).

This is supported by the incredibly high CaO content (88.10% and 87.51%) in the geochemical analysis.

Under heat and pressure, carbonate rocks (limestone or dolostone) recrystallize to become

marble, a metamorphic rock. This origin is strongly suggested by the geochemical and mineralogical evidence.

Metamorphism has occurred when metamorphic minerals are present.

Thus, these samples are properly categorized as marble due to the high calcite concentration and geochemical data.

2. EBA3 and EBA4

Comparison

Mineralogy:

Orthoclase, anorthite, biotite, and muscovite are present, together with a high quartz percentage.

Metamorphic minerals are present.

Geochemistry:

Low CaO, FeO, K₂O, moderate to high Al₂O₃, and high SiO₂.

Justification for the name "Metaconglomerates"

A siliciclastic sedimentary protolith, like sandstone or conglomerate, is indicated by the high quartz concentration.

Based on the mineralogical data, the term "meta" denotes that these rocks have undergone metamorphism.

The differences in mineralogy amongst the samples show that there were a wide range of clast sizes in the original parent rock.

Sedimentary rocks with rounded clasts of different sizes make up conglomerates. The prefix "meta" indicates that the original conglomerate has undergone metamorphosis, which has altered

its texture and mineralogy.

Thus, the presumed sedimentary protolith and the mineralogical and geochemical data support the categorization of these samples as metaconglomerates.

3. UGB5

Comparison

Mineralogy:

Primarily composed of quartz, with muscovite and orthoclase present. Metamorphic minerals are present.

Geochemistry:

Low CaO, high K₂O, moderate Al₂O₃, FeO, and very high SiO₂.

Justification for the name "Quartz-Mica-Schist"

The quartz-mica-schist is characterized by its predominant quartz content and the presence of muscovite.

The alignment of mica minerals under pressure causes foliation, or layering, in schist, a metamorphic rock. The richness of mica is reflected in the high K₂O level.

Metamorphism is also indicated by the presence of rutile.

This sample's classification as quartz-mica-schist is supported by its high quartz content, the presence of mica, and the suggested metamorphic texture.

CHAPTER FIVE

SUMMARY, CONCLUSION AND SUGGESTION FOR FURTHER STUDIES

5.1 SUMMARY

Three different meta-sedimentary rock types from Ofu-Eba, Southwest Nigeria—marble, metaconglomerates, and quartz-mica-schist—were examined in this study for their mineralogical and geochemical makeup. According to mineralogical investigations, the marble samples were mainly made of calcite, with notable amounts of quartz and orthoclase, suggesting that the carbonate protolith had undergone metamorphism. A siliciclastic sedimentary origin was suggested by the metaconglomerates' high quartz concentration, feldspars, and mica. As is typical of higher-grade metamorphic rocks, quartz and muscovite predominated in the quartz-mica-schist. The mineralogical results were supported by geochemical data, which showed that the quartz-mica-schist had high SiO₂ and K₂O, the metaconglomerates had increased SiO₂ and Al₂O₃, and the marble samples had an exceptionally high CaO content. This study highlights the various geological processes that have sculpted the Ofu-Eba region and offers insightful information about the protoliths and metamorphic histories of these rocks.

5.2 CONCLUSION

The three main rock types from Ofu-Eba have been satisfactorily identified and described using mineralogical and geochemical comparisons of the meta-sedimentary rocks. It is established that the metaconglomerates came from a siliciclastic sedimentary protolith, the marble samples from a carbonate protolith, and the quartz-mica-schist from a siliceous sedimentary protolith that

experienced higher-grade metamorphism. Differences in protolith composition and metamorphic conditions are reflected in the reported changes in mineralogy and geochemistry. The regional geology and metamorphic evolution of the Ofu-Eba area are better understood as a result of this study.

5.3 SUGGESTION FOR FURTHER STUDIES

The following recommendations for more research are put forth in order to improve our comprehension of the geological history and processes in the Ofu-Eba region:

Detailed Petrographic Analysis: Use optical microscopy to perform in-depth petrographic investigations to look at the rock samples' textures, grain sizes, and mineral connections. More accurate details about the metamorphic grades and processes would result from this.

Geochronological Dating: To ascertain the ages of the metamorphic episodes and limit the timeframe of protolith production, use radiometric dating methods (such as Ar-Ar dating of mica or U-Pb dating of zircon).

Trace Element Analysis: To provide more specific information about the origin of the sedimentary protoliths and the fluid-rock interactions during metamorphism, conduct trace element and rare earth element (REE) analyses.

Isotopic Studies: To improve knowledge of the fluid sources and metamorphic conditions, perform stable isotope analyses (such as those of the oxygen and carbon isotopes in marble).

Regional Geological Mapping: To gain a better understanding of the spatial distribution and relationships among the various rock units, enlarge the study area and carry out thorough geological mapping.

Conduct a structural analysis to ascertain the region's tectonic setting and deformation history.

Examine fluid inclusions in minerals to learn about the temperature and composition of the fluids used in metamorphic processes.

Environmental Impact Assessment: Examine the possible effects on the environment of these rock types' weathering and erosion, especially with regard to the discharge of trace elements into the environment.