GEOCHEMICAL AND PETROGRAPHIC STUDIES OF THE NEWLY EXPOSED FACIES IN THE IBESE QUARRY, DAHOMEY BASIN

by

AKINPELU, Racheal Anuoluwapo

GLY/14/2251

BEING A PROJECT SUBMITTED TO THE DEPARTMENT OF GEOLOGY, FACULTY OF SCIENCE, IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF BACHELOR OF SCIENCE DEGREE (B.Sc.) IN GEOLOGY, FEDERAL UNIVERSITY, OYE EKITI, EKITI.

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CERTIFICATION

FEDERAL UNIVERSITY, OYE-EKITI, EKITI,

DEPARTMENT OF GEOLOGY

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THE BOARD OF EXAMINERS CERTIFY AS FOLLOWS:
HIS IS THE ORIGINAL WORK OF THE CANDIDATE; THAT THE IS ACCEPTED IN PARTIAL FULFILLMENT OF THE ENT FOR THE AWARD OF THE DEGREE OF BACHELOR OF Sc.) IN GEOLOGY

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25/02/19
Signature/Date

Prof. O.J. Ojo
HOD

Signature/Date

DEDICATION

I dedicate this project first and foremost to God Almighty who has given me the strength and enablement to complete this work. I also dedicate this work to my Parents, Mr. and Mrs. O. Akinpelu, for their prayers and support throughout the course of this work. Finally, I dedicate this project to every lecturer in the Department of Geology for their selfless efforts directed towards making me a better person as well as a competent geologist.

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ABSTRACT

Limestone, glauconite, ferruginized sandstone and shale samples were obtained from the mapped newly exposed part of the Dangote Cement quarry located in Ibese, eastern Dahomey Basin, Nigeria. The Paleocene–Eocene Akinbo and Ewekoro Formations were studied, logged and sampled from the Northern (NM) and Southwestern (SWM) sections quarried to depths of about 12*m* and 23*m* respectively. Twelve samples taken from these sections have been examined in an attempt to determine the geochemical characteristics of the sediments, mineralogical composition and provide petrographic interpretation in order to understand the associated diagenetic processes and depositional conditions. A combination of analytical techniques including: geochemical analysis for major and trace elements, X-ray diffraction (XRD), thin section and scanning electron microscopy (SEM) were used in this study. Results show that the major elements present in the analyzed samples include CaO, SiO_2, SO_3, Al_2O_3, Fe_2O_3, P_2O_5, MgO, K_2O, TiO_2 and MnO respectively. CaO is the dominant oxide with concentration ranging from 4.13 – 94.67*wt.*% (*Av.* 65.85*wt.*%), followed by SiO_2 with values ranging from 1.38 – 59.78*wt.*% (*Av.* 30.58 *wt.*%). The high Ca/Mg ratio defines a high calcite purity, which is significant in cement production. LOI values range from 5.13 – 12.20% and trace elements are generally below 0.7ppm in all the samples. Calcite (56.69 – 57.96%) dominates the mineralogy followed by quartz, dolomite and kaolinite. Petrographic observations identify four facies: unsorted biosparite, sparse biomicrite, fossiliferous micrite, and packed biomicrite, which have been affected by various diagenetic processes such as cementation, neomorphism, micritization, compaction, dissolution and dolomitization. Due to the presence of fossils such as sponges, molluscs, the limestones are typically deposited in a freshwater environment, probably upper deltaic (non-marine) environment or brackish water, (probably lower deltaic (marginal-marine) environment). The studied limestones are suitable for cement production based on the elemental composition.

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INTRODUCTION

1.1 General Statement

The Dahomey basin is an arcuate extensive basin located within the Gulf of Guinea, West Africa. It extends from the eastern part of Takoradi height in Ghana through Togo, Republic of Benin and then terminates at the Okitipupa ridge, southwestern Nigeria separating it from the Niger Delta. (Boboye and Omotosho, 2017, Whiteman, 1982). The Basin cuts across four coastal states in Nigeria covering a total area of 49,209km^2 and contain sediments as thick as 3000m. The basic lithologies include: limestone, mudstones, shale, siltstone, sandy shale, marl and laterite with notable presence of beds of phosphatic and glauconitic nature, and an occasional occurrence of lignite.

In this study, rocks freshly encountered within the Ibese limestone deposit quarry, operated by Dangote Industries Limited were mapped, sampled and analyzed. Lithologies encountered are limestones, glauconitic rock, shale and sandstone.

Limestones are carbonates which are the most abundant non-terrigenous sedimentary rocks, consisting roughly one-tenth of the Earth's sedimentary shell, the most common being limestone, which is dominant in the study area in various forms. Three classification systems are currently used, each with a different emphasis, but that of Dunham, which is based on texture is now more widely used (Tucker, 2001). The first scheme, which is very simple, divides limestones on the basis of grain size into calcirudites (>2mm), calcarenite (2mm-62μm and calcilutite (<62μm). The scheme of R.L. Folk (Folk, 1959; 1962) is based on composition and it distinguishes three components; the grains (allochems), matrix (chiefly micrite), and cement (usually drusty sparite). The third classification of R.J. Dunham divides limestones on the basis of texture into: grainstone, packstone, wackestone and mudstone (Dunham, 1962). Additional terms such as: boundstone, crystalline, floatstone, rudstone, baffle stone, bindstone and framestone are often also used. Most limestone form in shallow, calm, warm marine water, the type of environment where organisms capable of forming $CaCO_3$ shells and skeleton. Calcite ($CaCO_3$) can easily be extracted along with every other needed ingredient from the ocean water. When these animals die their shell and skeleton debris, as sediments, lithifies into limestone. Their waste products can also contribute to the sediment mass. Limestone formed from this type of sediment are biological sedimentary rocks. Their biological origin is often revealed in the rock by the presence of fossils. Limestones formed by chemical processes are: oolithic limestone, travertine, chalk, tufa and the diatomaceous marl, which is particularly obvious type in Ibese (Boboye and Omotosho, 2017).

Shale is another lithology present significantly in the Ibese quarry. They are any mudrock that exhibits lamination or fissility or both (Prothero and Schwab, 2014). Black shales are mudrock that typically contains 1%-15% Organic Carbon (Wignall, 1994). The Black or dark grey color is probably due to the presence of the organic matter and because of finely disseminated pyrite (iron sulphide), which is formed under reducing conditions.

The sandstones of the Ibese quarry, are fine grained with the ferruginized sandy clay units significantly different from the upper units (Boboye and Omotosho, 2017). The clay and shale units are laterized occurring as concretions ranging from brown to reddish brown and grey in color respectively. The purpose of this study is to understand the geochemistry and petrography of the newly exposed section of the Paleocene-Eocene Akinbo and Ewekoro Formations at Ibese area.

1.2 Aim and Objectives of the Study

The aim of this project is to attempt a genetic description of the various lithofacies of the Ewekoro and Akinbo Formation with emphasis on the limestones.

The specific objectives include:

1. to determine the geochemical characteristics of the sediments and integrating the major and trace element geochemistry with the mineralogical composition.

2. to provide petrographic interpretation in order to understand associated diagenetic processes to which the rocks have been subjected and infer the depositional conditions.

1.3 Scope of the Study

The scope of this study includes:

1. detailed geological field mapping and sample collection from quarried sections of the Akinbo and Ewekoro Formations, exposed around the North and Southwestern mine location of the Dangote Industries limestone quarry, Ibese, Ogun state.
2. laboratory studies involving geochemical, mineralogical and petrographic analyses of the samples collected.
3. integration and interpretation of the results obtained from the laboratory studies.

1.4 Relevance of the Work

Geochemistry of sedimentary rocks is a valuable tool to infer factors that control sediments characteristics during and after their deposition and to delineate the relationship between specific units of both clastic and carbonate strata (Nagarajan, *et al.*, 2008; Armstrong-Altrim *et al.*, 2009; Frimmel, 2009; Madharavaju and Lee, 2009). Carrying out geochemical studies on the rock samples obtained from the Ibese quarry, and integrating the results with the mineralogical composition and petrographic evidences would serve as a veritable tool to understand the geological controls and economic viability of the limestone resource of the Ibese quarry.

1.5 The Study Area

The study area is the Ibese limestone deposit quarry, operated by Dangote Industries Limited located within the Dahomey basin, which lies between latitudes 6^0 9' N and 7^0N and longitudes 3^0E and $3^0$3'E. The area is located about 40km southwest of Abeokuta where quarrying activities have revealed rocks of diverse ages and lithologies within the basin (Fig. 1.1). The geology as exposed at the Dangote cement quarry shows the occurrence of lateritic sandy horizon at the topmost layer, with basal units consistent with the geology of the Dahomey basin. The area covers about 50% of the Ogun State portion of the Dahomey basin, and is accessible by easily noticed roads marked by the rigid pavement of concrete.

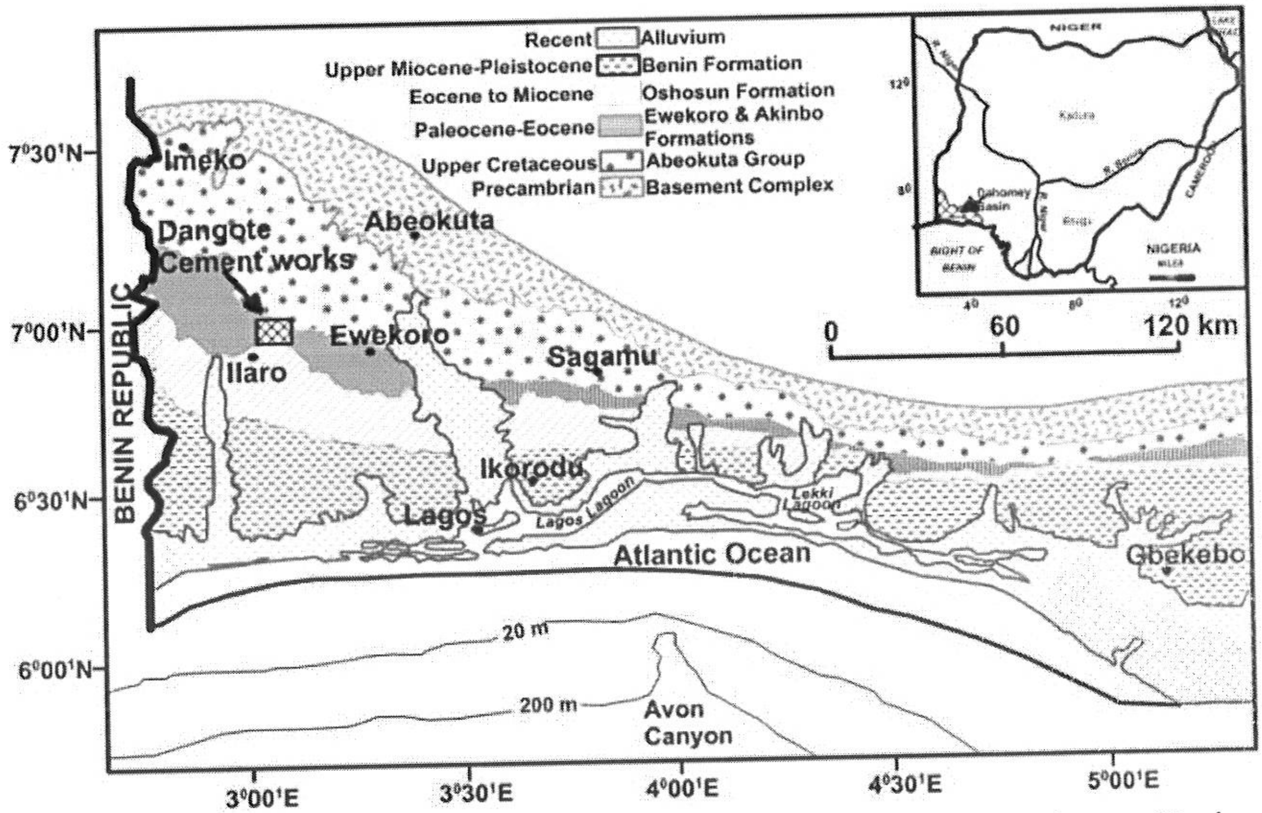

Fig. 1.1: Dangote Cement works, Ibese at the eastern portion of the Dahomey Basin.

1.5.1 Physiography

Ogun State has a wide area of undulating lowlands belonging to the coastal sedimentary rocks of western Nigeria. There are scattered hills that are interfluves between the different river valleys. Some remnants of a large planation in the state include the outcrop inselbergs found at Abeokuta the Olumo Rock at the southern edge of the Western uplands. One implication of this location of the state is seen in all the rivers that traverse the state which flow southward either as tributaries or main rivers into the coastal lagoons and the Atlantic Ocean. These include Ogun, Osun, Yewa, Yemoja, Ona, Sasa, Oni, Ohu, Ohia, Abafon, Oyan, lju and others. Most of the state is well-drained by these streams and rivers (Fig. 1.2), much of which dry up during the dry season. The area is relatively gentle, gradient-wise with elevation of about 42m at the highest point.

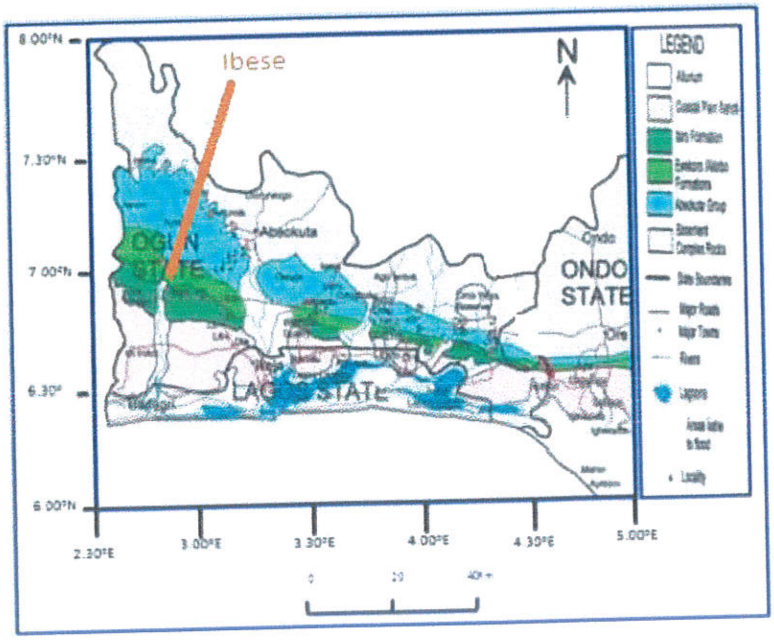

Fig. 1.2: Map showing the drainage and waterways of Lagos and Ogun state.

1.5.2 Climate and Vegetation

The study area is located in the tropical savannah region with its closeness to the ocean and distance from the Sahara Desert. Ogun State is located in the moderately hot, humid tropical climatic zone of southwestern Nigeria. There are two distinct seasons in the state, namely, the rainy season, which lasts from March/April to October/November and the dry season, which lasts for the rest of the year, *i.e.* October/November till March/April. The mean average temperature of about 18.45^{0}C – 36.9^{0}C, wind speed of 9mph from the south, and cloud cover of 59% is typical of the area. Visibility can be as clear as 12miles with humidity of about 46% and annual rainfall maxima of 1500m around September.

The area is marked by the presence of tall evergreen trees typical of rain forests, shrubs and grasses. Ogun State has two main types vegetation, namely: tropical rain forest and guinea savanna. The tropical rain forest is found in the coastal areas in Ogun Waterside and the southern part of Egbado South LGAs. Rain forests are found in some parts of the eastern LGA it such as ljebu-lgbo, Odogbolu, Sagamu and ljebu Ode. Guinea and derived savanna are found most of the western and northern LGAs including in Egbado North and South, lfo, Ewekoro, Abeokuta, Owode and Ado-Odo/Ota.

1.6 Review of Previous Work

The structural setting, general geology and hydrocarbon potential of Eastern Dahomey basin have been summarized by Jones and Hockey (1964), Reyment (1965), Adegoke (1971), Nwachukwu and Adedayo (1987), Ekweozor and Nwachukwu (1989), Okosun (1998), and Nton *et al.,* (2006) among others.

The importance of Geochemistry in determining source area of sedimentary rocks, paleo-weathering source area of sedimentary rocks, paleo-weathering conditions as well as tectonic evolutions of sedimentary basins is well established in many literatures (Taylor and McLennan, 1985; Cullers, *et al.,* 1988; McLennan, 1993; Nagarajan, *et al.,* 2007b). Consequently, there are quite a number of geological studies focusing largely on stratigraphy, paleontology, petrology and geochemistry of the Limestone of Dahomey (Jones and Hackey, 1964; Reyment 1965; Adegoke, *et al.,* 1971; Fayose and Azeez, 1972; Ogbe, 1972; Okosun, 1998; Nton, and Elueze 2005; Adekeye *et.al.,* 2006; Akaegbobi and Ogungbesan, 2016; Boboye and Omotosho, 2017; Olatinsu *et al.,* 2017).

Petrographical study of Ewekoro carbonate rocks (Akinmosin and Osinowo, 2010) revealed the limestone constituents observed belongs to that of Wackestone; asserting that there are allochems of sponges, echinoids, bivalves, coralline algae, and pelecypodds which represents the skeletal content with the intraclast pellets and ooids representing the non-skeletal grains. The orthochemical components were carbonate mud matrix and sparry calcite cement. The authors asserted that the constituent observed belong to the wackestone and could have been deposited in a quiet and low energy environment. Other than the fact that other carbonates rocks like marl wasn't studied, classification of all the limestones in Ewekoro as wackestone is too ambiguous as described the Formation as consisting of shaly limestone 12.5m thick which tends to be sandy and divided it into three microfacies.

Akaegbobi and Ogungbesan (2016), carried out geochemistry of the drill cores limestones of Paleocene age of the Ewekoro Formation, using Inductively Coupled Plasma Mass Spectrometric (ICP-MS) method to get the main oxides, rare earth elements and trace elements. The limestones where normalized with the Post Archean Australian shales (PAAS) which revealed significant enrichment in Nb, Sr and U whereas Ba, Th, Rb and other trace elements contents were depleted. The PAAS normalized REE+Y of the limestones displayed relatively uniform patterns of (1) slightly enriched REE, (2) positive Gd anomaly, (3) positive Ce anomaly, (4) positive Eu anomaly, and (5) high Y/Ho ratio which suggested non-seawater-like attributes. REE contents, high La_N/Yb_N ratios and high Y/Ho ratios suggested that variations in REE content where brought about by terrigenous contaminations of the limestones. The La/Sc, La/Co, Th/Co, Th/Cr, Cr/Th and Th/Sc suggest that the terrigenous inclusions in these limestones has their sources in felsic rocks. In summary, his work indicated that the Ewekoro Formation sediments were deposited in open shallow marine environments under the influence of oxic to anoxic conditions.

Nuclear Magnetic Resonance (NMR) technique are valuable tools for pore structure description and fluid discrimination in logging application and core-lab analysis. Pore scale characterization in particular utilizes fluid-rock interaction to evaluate pore-size distributions and pore volume of rocks. Pore characteristics have been linked with rock mechanical properties which often affects their use for certain purposes such as building stones (Buj and Gisbert, 2010).

Olatinsu *et al.,* 2017, used Nuclear Magnetic Resonance (NMR) to characterize three rock samples: shale, limestone and sandstone, in no particular order. Results obtained from this study showed that the most useful characteristics used to discriminate the different facies come from full saturation ore size distribution from clay bound water component (T_{2s}) coupled to capillary bound water component (T_{2i}) centered on 2ms. T_{2i} didn't exist in Glauconite facies. The Basic difference between the limestone and sandstone facies is related to the longer T_2 coupling. T_{2i} and T_{2l} population are coupled in sandstone generating a single population, which convolves. Both population limestones exhibit trimodal distribution attesting to the facts that limestones has a more complex pore system than siliciclastic rocks. The degree of pore connectivity is highest in sandstone, followed by limestone and least in Glauconite. Therefore, a basic and quick NMR log run on a samples along a geological Formation can provide precise lithofacies characterization with quantitative in Formation on pore size, structure and distribution.

Boboye and Omotosho (2017), logged and collected samples from wells and using X-ray fluorescence was able to decipher the amount of CaO, SiO_2, Fe_2O_3, MgO, Al_2O_3 and establish them as the most abundant elements. Increase in CaO, MgO down the sequences show depth of diagenetic re-distribution. Percentage of Al_2O_3, Fe_2O_3 and K_2O are significant to indicate continental influence during diagenesis and many depositional episodes. The three different microfacies from the petrographical indices for the indication of lithofacies, chemistry and viability done where; shelly biomicrite, shelly biosparmicrite and algae biosparmicrite. The predominance of micrite as a cementing matrix once again show the quiet shallow marine inner shelf environment occasioned by storm waves. This work factors in X-ray diffraction (XRD) and scanning electron microscopy (SEM) as a better determinant to corroborate the work of all these authors.

CHAPTER TWO

REGIONAL GEOLOGY AND TECTONIC SETTING OF DAHOMEY BASIN

2.1 Location

Dahomey Basin comprises of island coastal and offshore basins that extend from southeastern Ghana through Togo and Benin republic to southeastern Nigeria. It is separated from the Niger Delta basin by the Okitipupa ridge (Ogbe, 1972; Omatsola and Adegoke, 1981). The Nigerian sector of the basin extends from the boundary between Benin Republic and Nigeria to the Benin hinge line, consisting of an arcuate belt roughly parallel to the coastline (Fig. 2.1) (Whiteman, 1982). It runs parallel to the coastal states of these countries covering a total area of approximately 49,209 km^2 (Whiteman, 1982). The states within the basin in the Nigerian sector include Lagos, Ogun, Ondo, Edo, and the sediment thickness increases toward the sea. The northern part of the basin in Nigeria is the exposure of the Abeokuta group which unconformably overlies the basement while towards the sea, there is a progressive increase in the thickness of chemical and biochemical sediments such as gypsum, glauconite, limestone and other associated marine deposits which is then capped by coastal sands (Boboye and Omotosho, 2017).

2.2 Basin Evolution

The Dahomey Basin has been described as an extensive wedge of Cretaceous to Recent sediments, which lies unconformably on the basement (Whiteman, 1982). These rocks are upper Cretaceous Post Santonian also outcrops in the River Niger, Bida Basin and the eastern sector part of the Anambra Basin down to the Okitipupa high. It is a marginal sag or pull-apart basin. (Klemme, 1975; Kingston, *et al.*, 1983) which developed in the Mesozoic as a result of the drifting of the African and south American plate (Burke *et al.*, 1971; Whiteman, 1982).

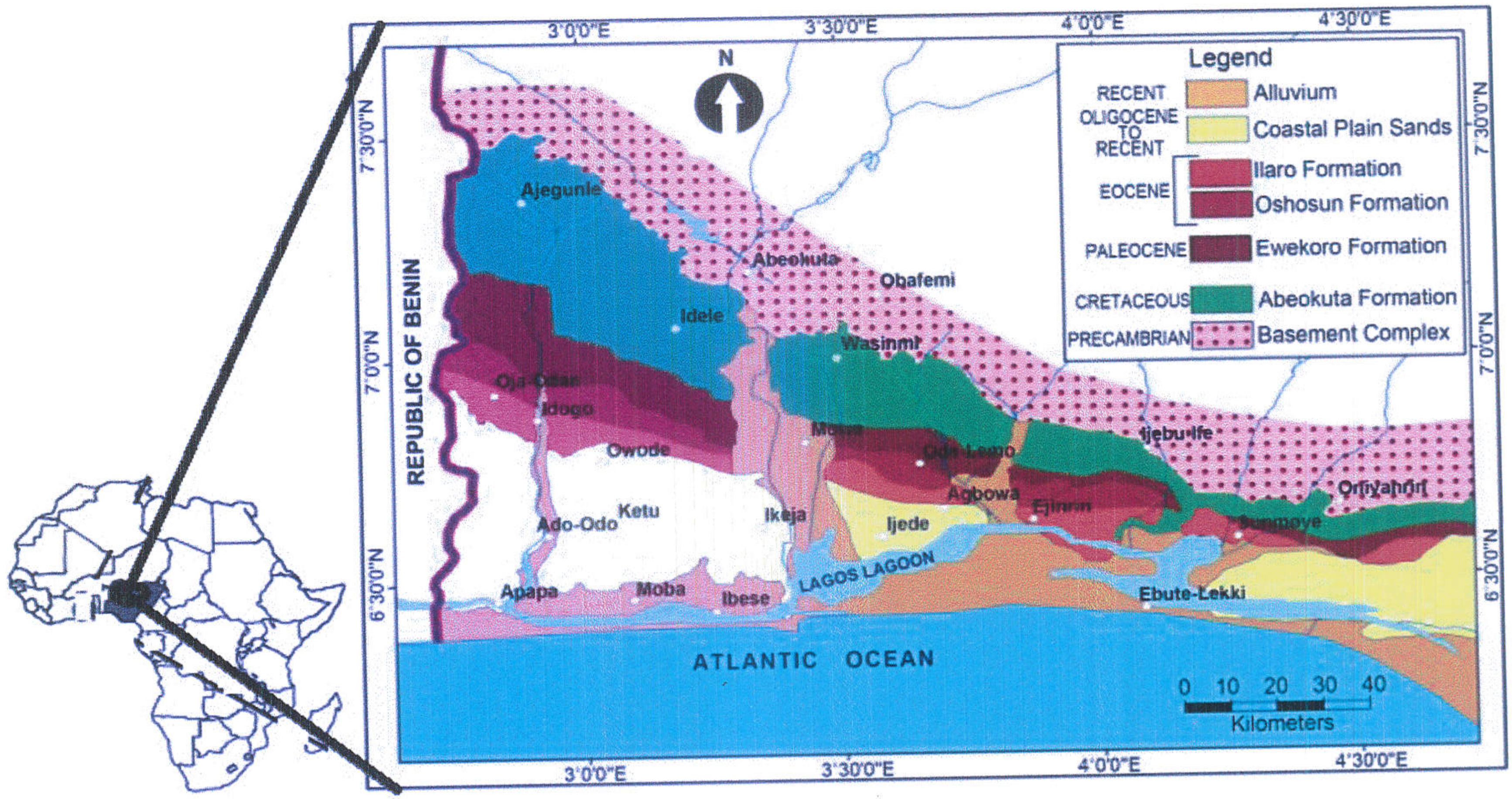

Fig. 2.1: Map of Africa showing the location of Nigeria and the generalized geological map of the eastern. Dahomey Basin (*modified after* Gebhardt *et al.*, 2013)

In the Dahomey Basin, the three-stage tectonic evolution allows the stratigraphic sequence to be divided into three sequences namely: (1) Precambrian to Triassic intracratonic rocks and Jurassic to lower Cretaceous, Continental to marginal marine rocks representing the pre-transform stage; (2) the Lower Cretaceous to late Albian rocks representing the syn-transform stage; and (3) Cenomanian to Holocene rocks representing the post-transform stage (Boboye and Nwosu, 2013).

2.3 Stratigraphy

Jones and Hockey (1964); Reyment (1965); Adegoke (1981); Nwachukwu and Adedayo (1987); Ekweozor and Nwachukwu (1989); Okosun (1998); and Nton *et al.*, (2006) discussed the various aspects of the stratigraphy of the Dahomey basin. Billman (1992) divided the stratigraphy of the entire basin into three chronostratigraphic packages: Pre-lower Cretaceous folded sediments, Cretaceous folded sediments and Tertiary sediments. The oldest dated sediments onshore are the Cretaceous sediments, which rest unconformably on the crystalline Basement Complex. Further offshore, the Cretaceous sediments thicken and rest unconformably on the pre-lower Cretaceous sediments. In Nigerian sector of the basin, the Cretaceous sequence referred to as Abeokuta Group is sub-divided into three Formations: Ise, Afowo and Araromi Formations (Omatsola and Adegoke, 1981).

The outcrops studied are in the Tertiary section consisting of the Ewekoro, and Akinbo Formations. The Tertiary sediments consist of the Ewekoro, Akinbo, Oshosun, Ilaro and Benin Formations. Ewekoro Formation (Paleocene) is made up of fossiliferous limestone while Akinbo and Oshosun Formation are made up of flaggy grey and black shales, Glauconitic bands and phosphatic beds define the boundary between the Ewekoro and Akinbo Formation. Ise Formation (Neocomian-Albian) is the oldest lithic fill and is unconformably disposed on the basement complex. It comprises of conglomerate and grit at the base, overlain by coarse-grained loose sand interbedded with kaolinitic clays (Omatsola and Adegoke, 1981). Afowo Formation is the middle layer of the Cretaceous sequence. It is composed of transitional sand and sandstone with variable but thick interbedded marine shales, siltstone and claystones. The sandy facies are tar bearing, while the shales are organic-rich. The Formation has been dated Turonian – Maastrichtian. Araromi Formation (Maastrichtian – Paleocene) is the uppermost unit and is made up of fine to medium-grained sandstone at the base, overlain by shale and siltstone with interbeds of limestone, marl and lignite.

Next in the Tertiary is the Ewekoro Formation which consists of thick fossiliferous limestone. It is Paleocene in age and associated with shallow marine environment due to abundance of coralline algae, gastropods, pelecypods, echinoid fragments and other skeletal debris (Maduekwe, *et al.,* 2015). The Akinbo Formation overlies Ewekoro Formation and it consists of shale, flaggy glauconitic rock bed, and gritty sand to pure grey shale and with little clay. Limestone lenses from Ewekoro Formation grades literally into the Akinbo shale towards the base. The base is characterized by the presence of a glauconitic rock. The age of the Formation is Paleocene to Eocene. The Oshosun Formation overlies the Akinbo, which is a sequence of mostly pale greenish-grey laminated phosphatic marls, light grey white-purple clay with interbeds of sandstones. It also consists of claystone underlain by argillaceous limestone of phosphatic and glauconitic materials in the lower part of the Formation and were deposited during Eocene (Agagu, 1985). The sedimentation of the Oshosun Formation was followed by a regression phase, which deposited the sandstone unit of Ilaro Formation (Kogbe, 1976). The sequence represents mainly coarse sandy estuarine deltaic and continental beds, which show rapid lateral facies change. The coastal plain sands are the youngest sedimentary unit in the eastern Dahomey basin. It conceivably unconformably overlay the Ilaro Formation but lack convincing evidence (Jones and Hockey, 1964). It consists of soft, poorly sorted clayey sand and pebbly sands deposited during Oligocene to Recent.

Regionally, the limestone of the Ewekoro Formation is traceable for a distance of about 320km from Ghana to the eastern margin of Dahomey basin. Akinbo Formation

(Paleocene and Eocene) consists of shale and clay overlying Ewekoro Formation (Ogbe, 1972). Its base is defined by the glauconitic block. Oshosun Formation overlies the Akinbo Formation and consists of phosphate bearing, greenish-grey or beige-clay and shale with interbeds of sandstone (Okosun, 1998). Ilaro and Benin Formations are predominantly coarse, sandy, estuarine, deltaic and continental beds which are difficult to identify in the field. The summarized stratigraphy is shown in Fig 2.2 below.

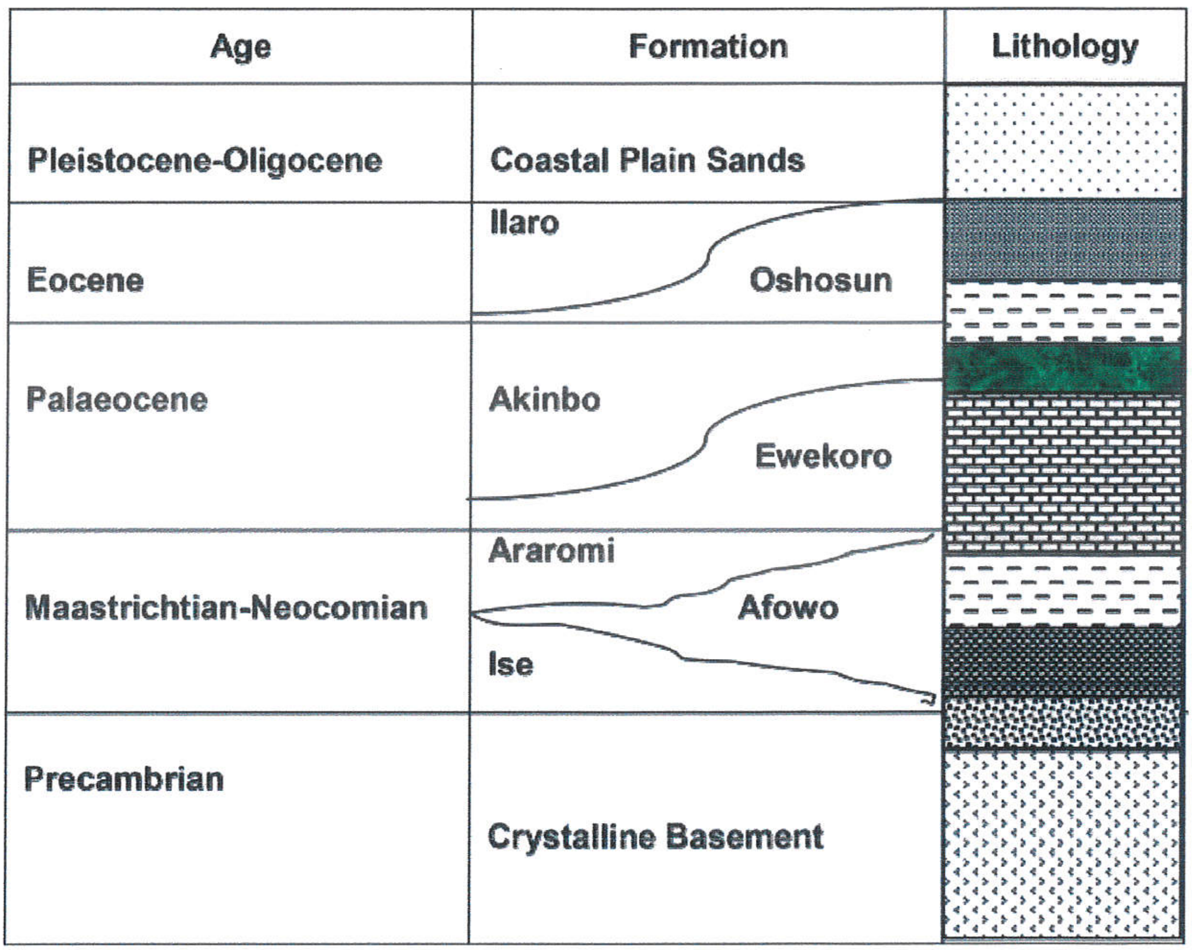

Fig. 2.2: Stratigraphy of Nigerian (Eastern) sector of Dahomey Basin (*modified after* Omatsola and Adegoke, 1981).

2.4 Petroleum Geology

(Boboye and Raji, 2013) reported that the biostratigraphy and petrography of the sequences were deposited in quiet shallow marine environments and that the presence of Toweiuscallosus and Coccolithusformosus indicated that Oshosun and Ewekoro Formations were Ypresian and Thanetian to Ypresian age. This rock is a potential hydrocarbon reservoir for the overlying shale while this character of the rock is seen in the Benin Republic sector of the basin, where petroleum is produced at marginal level (Boboye and Raji, 2013; Adeonipekun, *et al.,* 2012; Fakolade and Obasi, 2012). The aspect geochemical evaluation, depositional environment and reserve estimation of limestone deposit were also reported around Orimogija and Okeluse areas of eastern Dahomey Embayment (Abayomi and Dare, 2016; Ehinola, *et al.,* 2012). However, this study focus on the distribution of diverse lithofacies, genetic description of the Ewekoro Formation with emphasis on the limestone and what characterize the environment of its deposition, the extent of diagenesis, biological influence on the chemistry of the units, major elemental composition, post depositional processes and their viability in determining its economic values. Known Oil fields are the Toju-Ejanla and the Ajibutan field offshore in Ondo state owned by Conoil. The petroleum system being the Akinbo-Ewekoro petroleum system and the Ise-Afowo petroleum system.

CHAPTER THREE

METHODOLOGY

A desk study including review of previous literatures was undertaken ahead of the field work and laboratory studies, details of which are presented in this chapter.

3.1 Materials

3.1.1 Field Work and Sample Collection

Field mapping of the study area was carried out in February, 2018, around the Northern and Southern mines of Ibese quarry Dangote cement quarry. Rocks were examined in their natural locations in the field, geologically relevant observations of rock features were made and field relationships established. These observations are recorded in a field notebook along with sketches of the observed features where applicable. The Garmin 72 handheld Global Positioning System (GPS) was used in taking locations of the studied area, which was subsequently plotted on a map. In addition to the GPS, the following instruments and/or equipment were used in the course of the fieldwork: Compass Clinometer, Sample bag, Hammer, Field notebook, Cutlass, Hand Lens, Measuring Tape, Camera, Topographic Map of the area, Grain size comparator and Chisel.

3.2 Laboratory Techniques/Methods

3.2.1 X-Ray Fluorescence (XRF) Spectrometry

Samples for whole-rock geochemical analysis were pulverised to fine homogeneous size and then pelletized. Platinum gold crucible, Lithium tetraborate flux, Platinum mold and Jiangsu Skyray instrument were used in the preparation and analysis of the pellets. About 5.0g of rock powder of each of the sample was weighed out and mixed with few drops of polyvinyl alcohol and the sample placed in a die, and spread out to form a "puck". Subsequently, boric acid (backing) was placed on top of the rock powder and a pellet formed by applying pressure of 15 tons for about 15 seconds. After drying, the pellets were placed in the sample holder of the XRF spectrometer, and the fluorescence measured. The elements measured (as oxides) include SiO_2, Al_2O_3, Fe_2O_3, MgO, CaO, P_2O_5, K_2O, TiO_2, MnO and SO_3.Each channel was calibrated using certified international reference rock materials. Major and trace elements were determined using Energy Dispersive X–Ray Fluorescence (ED-XRF) Spectrometer (EDX3600B model) at the Centre for Engineering and Material Development, Akure, Ondo State.

3.2.2 Loss on Ignition (LOI)

The Equipment and materials used in the preparation of this test include: crucibles, evaporating dishes, weighing balance, desiccator and oven. LOI is the sum of the organic matter, inorganic carbon, sulfur and water in crystalline form. A predetermined amount of sample is roasted to determine the loss on ignition (LOI) by heating for some times at temperature as high as 100^0C in these case 6 days until there is no change in weight. The final weight was taken after the oven-dried samples were ignited for 2hrs at about 950°C and the LOI was determined.

3.2.3 X-Ray Diffraction (XRD)

The Equipment and materials used in the preparation of the XRD pellets were: pulverizer, Shimadzu XDS 2400H diffractometer, Cu anode, and ≤ 80μm Sieve.The samples are crushed, and sieved to a particle size ≤ 80μm. The sieved samples now in powdered form are used for X-ray diffraction analysis (XRD). XRD analysis was performed on the sample by a Shimadzu XDS 2400H diffractometer with Cu anode, λ_{CU} = 1.5406 [A°], on uncompressed powders in order to collect the maximum of the diffraction lines and a better identification of the phases. The prepared sample was placed in a Lucite holder on the goinometer of the instrument, which was configured with a graphite monochromator. The diffraction beam monochromator operated at 40 KVA and a current of 30 mA with the 2θ values varying from 2^0 to 60^0 with step size of 0.02^0 for 120 minutes to create x – ray patterns with enough intensity to produce lines to identify minerals at the 2θ angles. Scanning rate was 0.75 degree per minute. Minerals were identified using the JCPDFWIN software of the Joint Committee on Powder Diffraction Standard (JCPDS).

Most sample always possessed high background readings due to high degree of overlapping peaks, and this must be corrected to remove the background noise from the XRD patterns. Full profile fitting of the diffractogram was made by progressively refining parameters such as intensities of individual mineral phases, unit cell dimensions, peak widths, preferred orientations, and global parameters like zero setting of the diffractometer. All these parameters were used for the simulation of peak shapes, which in turn, would be used to generate a calculated diffraction pattern. The calculated diffraction pattern is then compared with the observed data, and the differences between the observed and calculated were minimized.

3.2.4 Thin Section

The Equipment and materials used in the preparation of the petrographic slides include: hammer, grinding plate ($30cm^2$ and 6mm thick). Abrasive power (F360 and F800), araldite A and B, glass slide (76mm X 25mm X 12mm thick), cover glass (40mm X 22mm), abrasive dispensers, petrographic microscope, mounting jig, methylated spirit for cleaning prepared slide, slide label pencils, wash bottle, forceps, spatula and silicon carbide. The procedure adopted in thin section preparation is briefly explained below:

1. The rock samples were air-dried at room temperature for 72 hours.
2. A little quantity was taken from each of the sediment samples.
3. Araldite A and B were mixed and 5g of the mixture smeared on the surfaces of the heated samples with the aid of a spatula.
4. After the surface impregnation, the samples were removed from the hot plate and allowed to cool at room temperature.

5. Surfaces of the cooled samples were polished with abrasives F360 and F800, washed and placed on the hotplate.
6. Surface of glass slides were polished with F800 abrasive, washed and placed on a hotplate.
7. Araldite A and B were mixed with 5g of the mixture taken with a spatula and smeared on the dried glass slides and then allowed to flow.
8. The dried polished samples were picked with the aid of a forcept and placed on the smeared glass slide and corked to remove air bubbles.
9. The mounted samples were placed on the mounting jig and allowed to remain on the hot plate for 10 minutes.
10. The mounted samples were removed from the mounting jig and allowed to cool at room temperature.
11. The prepared thin section slides were washed and cleaned with methylated spirit and excess araldite removed with the aid of razor blade.
12. The slides were labelled and later observed for their petrographic properties under the microscope.
13. In cases where by the slide is not thin enough and light is not passing through it, some silica carbide is smeared on a glass plate and the slides are filed till its thin enough.
14. The properties of the finished thin section are then checked under the petrographic and snapped for interpretation.

Thin section slides were prepared at the Department of Geology Laboratory of the Obafemi Awolowo University, Ile Ife, Osun State.

3.2.5 Scanning Electron Microscopy (SEM)

The Equipment and materials used in the preparation of the SEM were: ethanol, coverslips, carbon adhesives, discs, FESEM, 7500F and P+ sputter coater. For SEM, there is no special preparation other than to attach the mounting stub for insertion into instrument, and provide a conductive path using silver paint to avoid non-conductive, one must coat with a conducive thin layer (Au, C, Cr). To produce the SEM image, the electron beam is swept across the area being inspected, producing many such signals. These signals are then amplified, analyzed and translated into images of the rocks being inspected.

Finally, the image shown on CRT is collected for interpretation. JEOL JSM 840 electron microscope was used in this study. Each element in the sample produces X-rays with characteristic energies and wavelengths which can be analyzed using an energy sensitive Si (Li) detector in an energy dispersive system (EDX) or by dispersing the X-rays according to wavelength using the crystal detector of a wavelength dispersive system (WDX). In this study the WDX was used for quantitative analyses.

3.3 Processing the Results obtained from the Analyses of the Samples

Microsoft Office™ Suites, Grapher 12, and the IBM SPSS Softwares were used in processing reults obtained from the laboratory analyses of the samples. Geochemical Analysis results was prepared and bivariate plots of the major oxides was plotted in Microsoft Excel. Microsoft Powerpoint was used in preparing the lithologs and presentation of the findings from this study. Microsoft Word was used in documentation of the entire research, while the Grapher 12 software was used in the preparation of Ternary plots of major oxides composition. Statistical analyses performed on the geochemical data were done using SPSS software.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1 Field Characteristics

Limestone, glauconite, ferruginized sandstone, and Shale samples were obtained from freshly exposed quarry section of the Ewekoro and Akinbo Formations. The Ewekoro Formation as exposed at the Ibese mines –Northern Mine (NM) section (Fig. 4.1), about 12m thick (Fig. 4.2),is made up predominantly of limestone in various forms, dipping regionally towards the southern direction. The South-western Mine (SWM) section (Fig. 4.1) is made up of the Ewekoro and Akinbo Formations, quarried to the depth of about 23m of quarried section (Fig. 4.3), containing shales, ferruginized sandstone, glauconitic bed and limestones.

4.1.1 Limestone

The Limestones occur as beds of pure, whitish to dirty white, and in impure argillaceous forms. They are typically very fine to coarse grained in the NM section, while those of the SWM section are very fine grained. Macroscopic examination of the limestones show the presence of several fossils such as gastropods and bivalves. There were no visible sedimentary structures observed in the studied sections. Detrital input are basically localized within the Akinbo Formation. The limestone samples are labeled NM-02, NM-03, NM-04, NM-05, SWM-06, SWM-07 and SWM-08 respectively. These samples have been described and classified based on Folk (1959) and Dunham (1962) schemes (Table 4.1).

According to Folks (1959), we have: Sparry Micrite or Mic-calcite (NM-02, SWM-06 and NM-05), which are characterized by lime mud matrix alone with no visible fossils. Oo-Micr-Calcite or Rounded packed Biomicrite (SWM-07and NM-03).

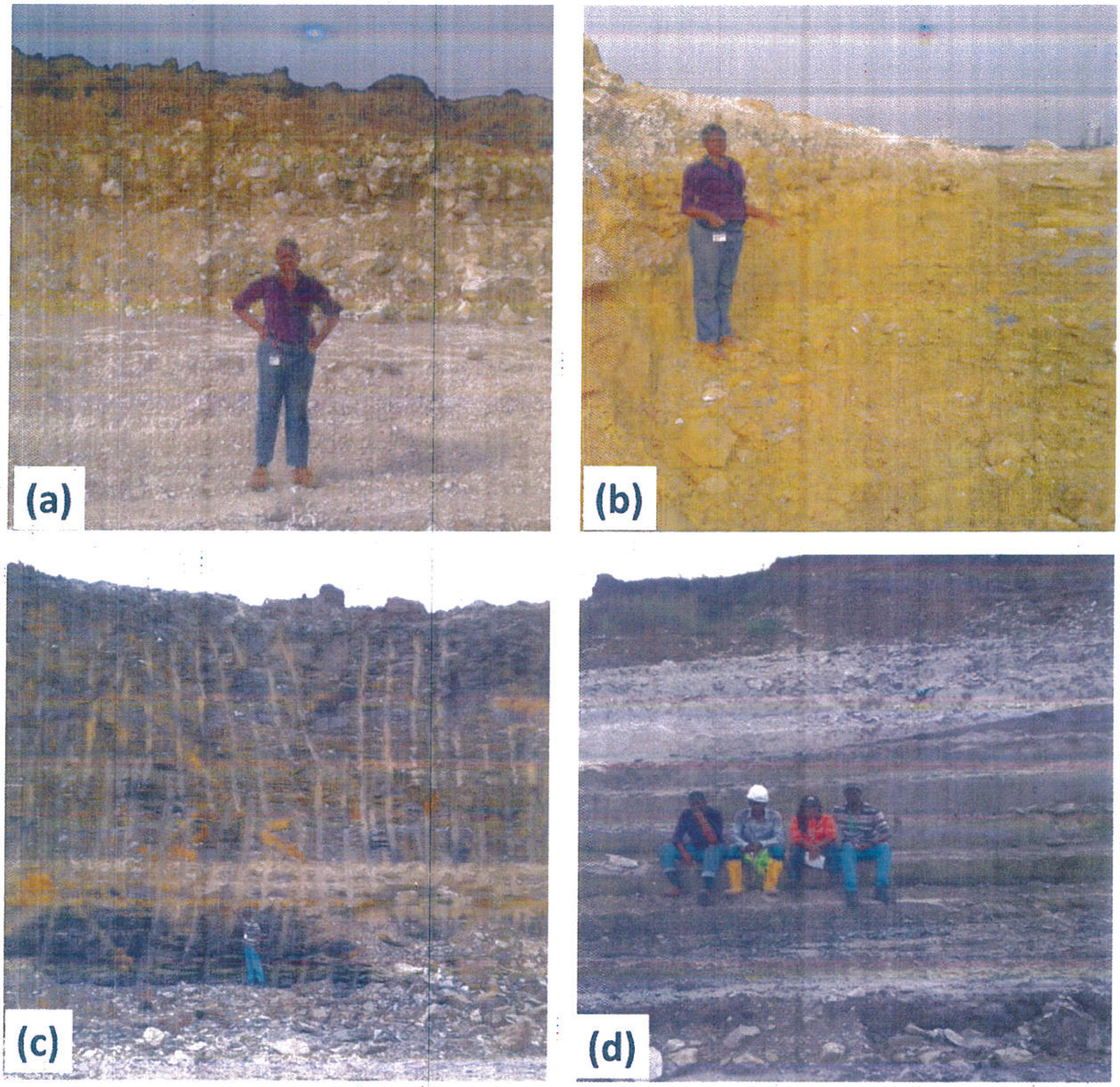

Figure 4.1: (a) Northern mines showing NM-01, 02 and 03. (b) Northern mines showing NM-04, 05 and 06. (c) Southern mine showing shales and ferruginized beds. (d) Southern mines showing the impure limestones.

Oo-calcite or Rounded micrite, which consists of Micrite with Oolithic grains (NM-04), Micrite (NM-06). SWM-08 of the Ewekoro Formation is layered with intervals of soft to hard marl, and crystalline limestone, such as calcite (SWM-07).

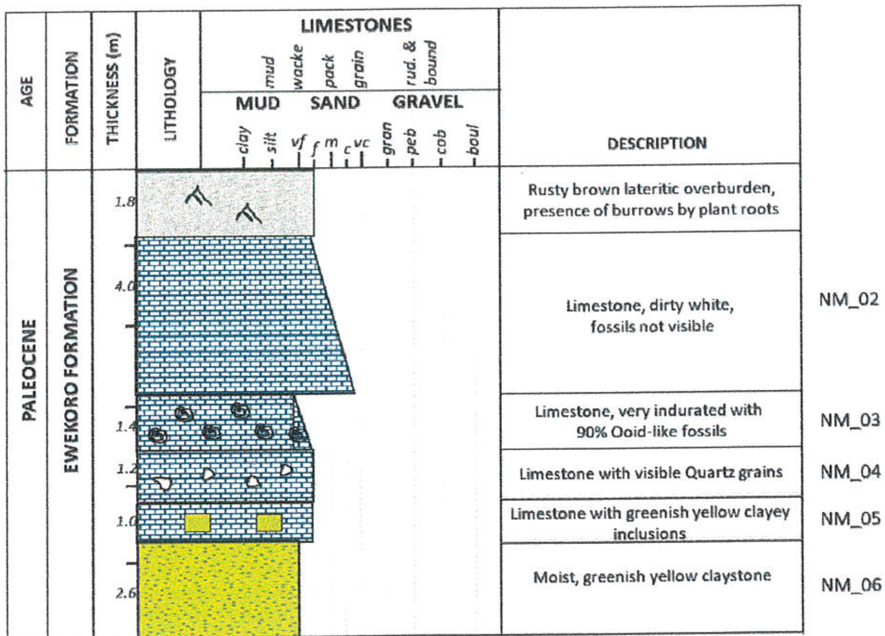

Fig. 4.2: Litholog of the Northern mine (NM) section [E: 003^0 01.692', N: 07^0 00.075'].

The individual layers are usually thin, around 10 cm, and the marls themselves are seldom thicker than a few cm. The impure argillaceous limestone contains small to large fossil fragments and is grey to ash in colour. The Dunham classification emphasizes limestone texture, especially grain (allochem) packing and the ratio of grains to matrix. Allochem type is ignored. Five types of limestones are identified: mudstone, Wackestone, Packstone, Grainstone, and Boundstone. All except Boundstone accumulate as elastic carbonates; individual components are not bound together during deposition.

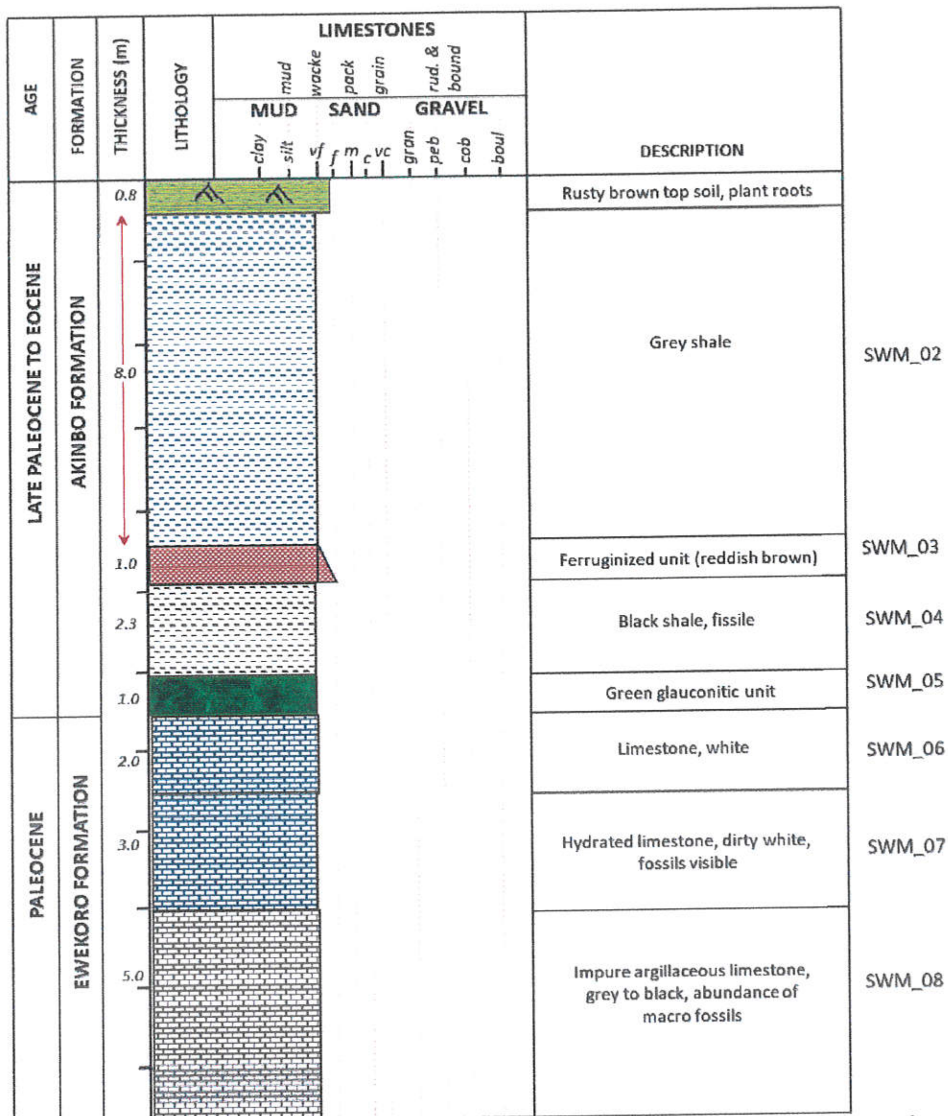

Fig. 4.3: Litholog of the South-western (SWM) mine section [E: 003^0 01.614', N: 06^0 59.440'].

Table 4.1: Description and classification of the studied limestones samples.

Sample ID	Description	Folk (1959)	Dunham (1962)
NM-02	Yellowish to creamy white fine to coarse grained limestone with visible fossils here and sparry calcite cement, unsorted biosparite, 1-10% Micrite	Micr-calcite	Wackestone
NM-03	Yellowish to creamy white, very fine to fine limestone with visible quartz grains and presence of a lot of visible fossils. Rounded packed biomicrite with 10-50% micrite.	Oo-micr-calcite	Packstone
NM-04	Creamy white to whitish, fine grained limestone with visible presence of fossils, packed biomicrite, poorly sorted and unsorted biomicrite	Oo-calcite	Wackestone
NM-05	Dirty white to greenish yellow, fine limestone with mud matrix and visible presence of fossils, sparry calcite cement, unsorted biosparite, 1-10% micrite matrix	Micr-calcite	Packstone
NM-06	Wet, brownish, clayey limestone that is very fine	Micrite	Mudstone
SWM-07	Dirty white, very fine limestone with a lot of tiny visible fossils present, sparry calcite cement, unsorted biosparite, 1-10% micrite matrix	Oo-micr-calcite	Boundstone
SWM-08	Impure argillaceous limestone greyish impure argillaceous very fine limestone and visible presence of fossils.	Sparse biosparite	Grainstone

4.1.2 Glauconite

The glauconitic bed is a green to greenish-yellow rock with perfect cleavage, with a dull glistering luster and a hardness of two (2). The shale units are rich in Glauconite and the limestone units constitute the weathered, poorly fossiliferous partly to the telltale of weathering on the layer and unweathered units which grades from the upper layer in a diffuse manner. The diagenetic process has had its toll on this layer due to pore spaces (mouldic and vuggy porosities) which shows casts of the disintegrated fossils. (Boboye, *et al.*, 2016).

Fig. 4.4: Glauconite Sample (SWM-05)

4.1.3 Ferruginized Sandstone

The ferruginized sandstone tagged SWM-03 is a 1 metre thick bed, sandy in texture, reddish to brownish in colour (Fig 4.4), the Fe/Mg ratio indicates this at 1.85 in Table 4.2.

The average Fe/Mg in SWM-03 measured from XRF is 145ppm indicating sudden increase suggesting a continental influence. The moderate amount (1m) of both reflect the ferruginous nature of these sediments by surface meteoric waters flushing rapidly through ferromagnesian rich minerals overlying the limestone (Ewekoro Formation).

4.1.4 Shale

Two shaly beds (SWM-02 and SWM-04) are observed within the Akinbo Formation, one of which is the black shale while the other is greyish to yellowish, fissile, clayey and concretionary (Fig. 4.4). Their black colour is due to a very high content of unoxidized organic matter. They are usually finely laminated, indicating little bioturbation or storm disturbance. Typically, they contain no bottom-dwelling marine organisms, but they may contain planktonic (floating) organisms, such as microfossils or graptolites, or nektonic (swimming) organisms, such as fish and marine reptiles, which sank to the bottom after they died.

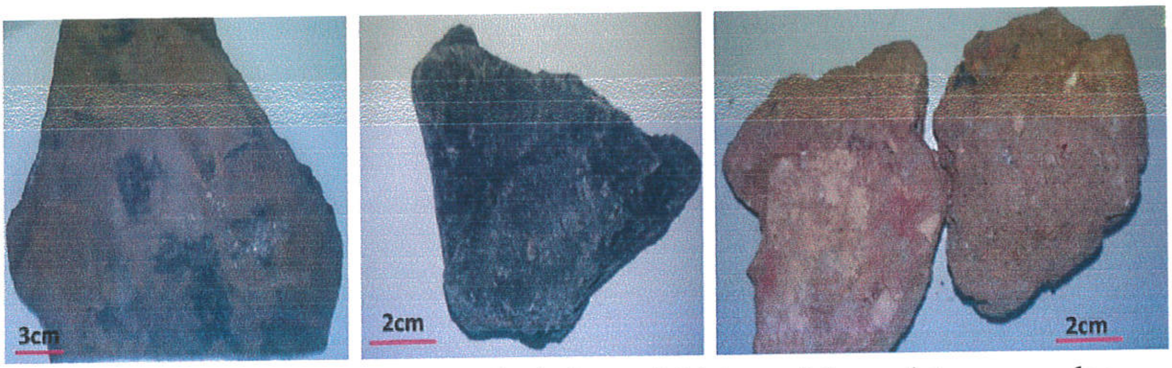

Fig. 4.5: (a) grey shale, (b) black shale, and (c) iron-rich sandstone samples.

4.2 Geochemical Analysis Results

The chemical composition of the samples is presented in Tables 4.2 and 4.3 respectively. 28 parameters were analyzed consisting of 10 major elements, loss on ignition (LOI), and 17 trace elements.

For the major elements, CaO and SiO_2 were the dominant oxides in most of the samples, with the exception of SWM-03 where Fe_2O_3 is present in significant amounts. CaO concentration ranges from 4.13 – 94.67 *wt.* %, with an average of 65.85%, while SiO_2 values range from 1.38 – 59.78 *wt.* % with an average of 30.58%. The chemical composition of the major elements present in the samples is presented in (Fig. 4.5). Results obtained shows the major elements from the twelve samples within the Ibese quarry, with average percentages of the other major oxides across the formation as SO_3 (6.61%), Al_2O_3 (4.62%), Fe_2O_3 (4.38%), P_2O_5 (1.05%), MgO (0.72%), K_2O (0.59%), TiO_2 (0.03%) and MnO(0.01%) respectively. CaO concentration is typically below 50% in SWM-02, 03, 04 and 05

(Fig. 4.4) due to the fact that they are argillites and not carbonates. Conversely, SiO_2 concentration is generally greater than 20% in these samples. This result is consistent with the geology of the Akinbo Formation. The high ratio observed in calcium and magnesium defines a high calcite purity, which is significance in the cement production. The presence of magnesium in carbonate rocks is a function of the temperature of deposition and time due to its removal by interstitial solution while dolomitization process could also contribute to high magnesium content. (Fayose and Azeez, 1972). LOI values range from 5.13 – 12.20%.

Table 4.2: Major elements composition and concentrations of the samples

ELEMENT/ OXIDE	NM-02	NM-03	NM-04	NM-05	NM-06	SWM-02	SWM-03	SWM-04	SWM-05	SWM-06	SWM-07	SWM-08	Min.	Max.	Av.	St. Dev.
MgO	0.00	0.00	0.09	0.00	0.00	0.99	2.66	0.00	3.23	0.21	0.82	0.65	0.00	2.66	0.72	1.10
Al_2O_3	1.13	1.40	2.90	4.88	4.55	13.61	9.39	9.37	4.12	1.24	1.13	1.71	1.13	13.61	4.62	4.09
SiO_2	1.38	1.94	5.10	10.09	9.12	59.78	36.74	31.66	19.89	1.79	1.47	2.36	1.38	59.78	15.11	18.61
P_2O_5	0.72	0.75	0.68	0.58	0.64	0.51	2.13	0.66	3.82	0.70	0.79	0.64	0.66	2.13	1.05	0.97
SO_3	1.19	1.19	1.01	0.49	1.28	11.48	4.87	40.41	9.34	2.30	3.38	2.42	0.49	40.41	6.61	11.20
K_2O	0.00	0.00	0.00	0.00	0.00	1.13	1.54	0.69	3.72	0.00	0.00	0.00	0.00	1.54	0.59	1.12
CaO	94.67	93.63	89.07	76.02	82.59	4.13	14.21	11.92	48.82	92.81	91.40	90.95	4.13	94.67	65.85	35.94
TiO_2	0.00	0.00	0.00	0.00	0.00	0.19	0.10	0.01	0.00	0.00	0.00	0.00	0.00	0.19	0.03	0.06
MnO	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.01	0.02	0.00	0.00	0.00	0.00	0.06	0.01	0.02
Fe_2O_3	0.34	0.47	0.48	5.76	1.09	6.57	26.26	4.09	6.14	0.35	0.48	0.55	0.35	26.26	4.38	7.34
LOI	6.85	7.69	7.63	5.13	9.26	10.20	12.20	10.10	8.62	7.14	7.14	6.25	5.13	12.20	8.18	1.97
Total	106.28	107.07	106.96	102.95	108.53	108.59	110.16	108.92	107.72	106.54	106.61	105.53	13.27	241.31	107.16	1.86
MgO/CaO	0.00	0.00	0.00	0.00	0.00	0.24	0.19	0.00	0.07	0.00	0.01	0.01	0.00	0.24	0.04	0.08
Fe_2O_3/MgO	0.00	0.01	0.01	0.08	0.01	1.59	1.85	0.34	0.13	0.00	0.01	0.01	0.00	1.85	0.34	0.66
SiO_2/CaO	0.01	0.02	0.06	0.13	0.11	14.47	2.59	2.66	0.41	0.02	0.02	0.03	0.01	14.47	1.71	4.14
Fe_2O_3/MgO	0.00	0.00	5.33	0.00	0.00	6.64	9.87	0.00	1.90	1.67	0.59	0.85	0.00	9.87	2.24	3.27
SiO_2/Al_2O_3	1.22	1.39	1.76	2.07	2.00	4.39	3.91	3.38	4.83	1.44	1.30	1.38	1.22	4.83	2.42	1.33
CaO/MgO	0.00	0.00	989.67	0.00	0.00	4.17	5.34	0.00	15.11	441.95	111.46	139.92	0.00	989.67	142.30	296.04

LOI = Loss on Ignition

Table 4.3: Trace elements composition of the samples

Trace Elements	NM-02	NM-03	NM-04	NM-05	NM-06	SWM-02	SWM-03	SWM-04	SWM-05	SWM-06	SWM-07	SWM-08
V	0.0000	0.0000	0.0000	0.0000	0.0011	0.0019	0.0120	0.0054	0.0000	0.0000	0.0009	0.0000
Cr	0.0000	0.0000	0.0000	0.0000	0.0000	0.0101	0.0228	0.0013	0.0181	0.0000	0.0000	0.0000
Co	0.0000	0.0005	0.0026	0.0688	0.0000	0.1577	0.5545	0.0793	0.1301	0.0000	0.0000	0.0004
Ni	0.0008	0.0000	0.0023	0.0168	0.0042	0.0257	0.0398	0.0192	0.0054	0.0022	0.0028	0.0022
Cu	0.0027	0.0026	0.0073	0.0180	0.0046	0.0150	0.0134	0.0121	0.0058	0.0038	0.0036	0.0034
Zn	0.0088	0.0097	0.0164	0.0493	0.0201	0.0589	0.0599	0.0598	0.0200	0.0105	0.0122	0.0152
As	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Pb	0.0000	0.0000	0.0000	0.0026	0.0000	0.0095	0.0002	0.0124	0.0014	0.0000	0.0000	0.0000
W	0.0000	0.0000	0.0000	0.0000	0.0000	0.0380	0.0317	0.0495	0.0000	0.0000	0.0000	0.0000
Au	0.0131	0.0000	0.0000	0.0000	0.0172	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Ag	0.0000	0.0001	0.0000	0.0000	0.0000	0.0052	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Rb	0.0003	0.0003	0.0000	0.0002	0.0000	0.0061	0.0042	0.0045	0.0055	0.0007	0.0000	0.0005
Nb	0.0000	0.0031	0.0013	0.0116	0.0003	0.0000	0.0054	0.0067	0.0000	0.0054	0.0007	0.0000
Mo	0.1093	0.1191	0.1329	0.2073	0.0916	0.2354	0.2247	0.2181	0.1553	0.1395	0.0329	0.1368
Cd	0.0000	0.0000	0.0002	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Sn	0.1668	0.2119	0.2008	0.4204	0.2515	0.6791	0.4922	0.5786	0.2558	0.1678	0.1906	0.2274
Sb	0.1810	0.1702	0.2170	0.4292	0.2638	0.6396	0.4474	0.5270	0.2875	0.1803	0.2052	0.2310

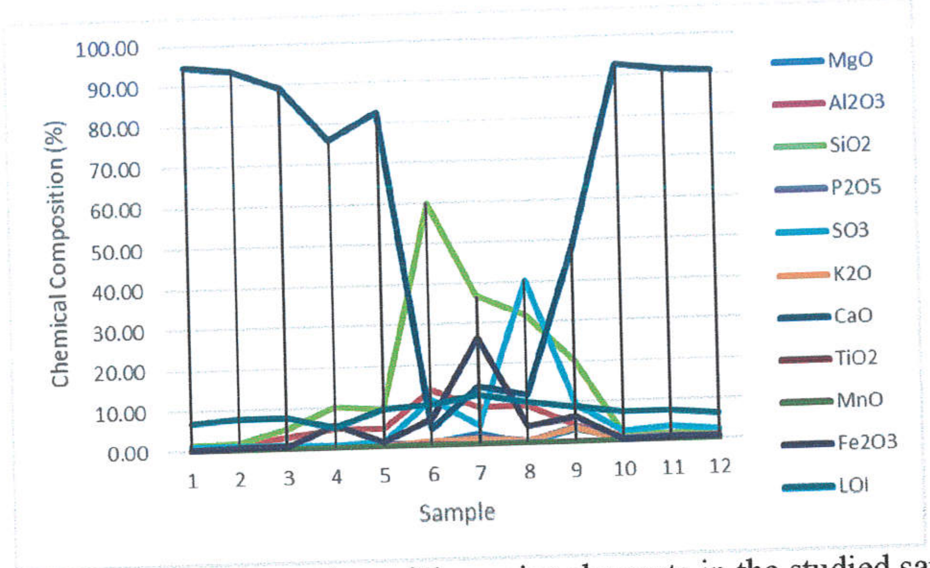

Fig. 4.6: Chemical Composition of the major elements in the studied samples.

4.2.1 Depth Relationship

Interpreted from (Fig. 4.5), silica is low from 15m – 23m and consistently increasing around depth 5m to 15m, with notably higher concentrations (>10%) between 8 and 14 m. By contrast, magnesium oxide concentration is very low all

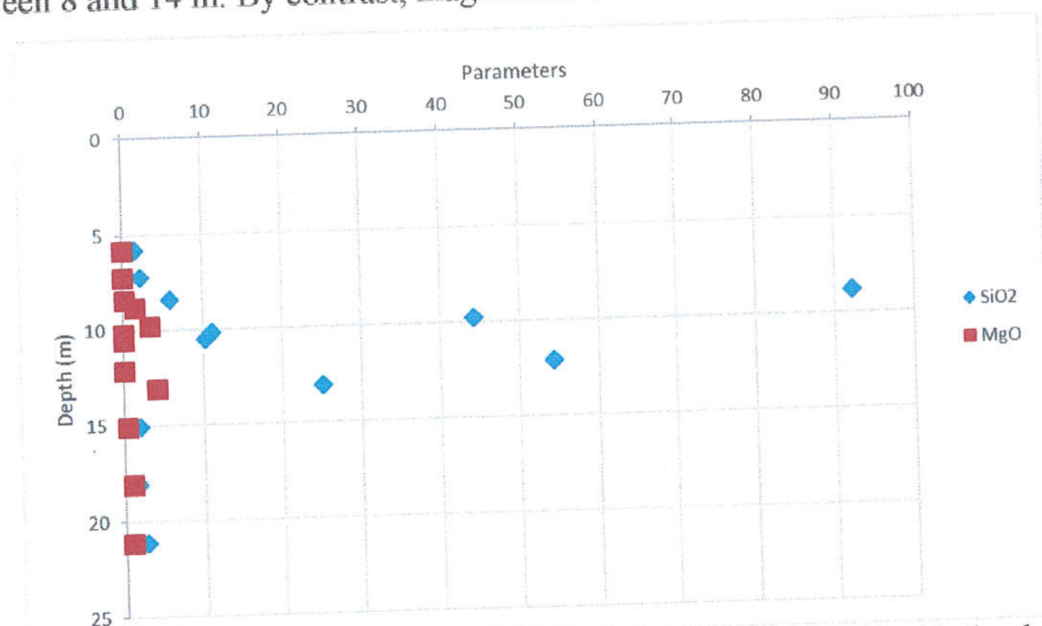

Fig.4.7: Depth plot showing the variation of SiO_2 and MgO with depth

through, generally <6m. A magnesium rich limestone is called a dolomite (Krauskopf, 1967). XRD analysis results revealed that two out of fifteen minerals identified are dolomite, indicating that the limestone is indeed very pure and not being altered by post depositional alteration of lime mud or magnesium-rich water, but remains essentially mostly calcite. Inclusion of SiO_2 at depths between 5m to 15m, could essentially be caused by the percolation of silica-rich waters from rainfall. Boboye and Raji, (2013) asserted that the biostratigraphy and petrography of the sequences were deposited in quiet shallow marine environment. Since it is overlain by shale, there could be detrital influence of sands and other arenites in the limestone as seen in X-ray diffractogram, marked by the presence of quartz and a little argillic alteration of feldspar (a tectosilicate) manifesting as kaolinite, plus shale being detrital with phyllosilicates contributes to this silica enrichment.

The almost absence of magnesium in carbonate rocks can be a function of the temperature of deposition and time, due to its emplacement by interstitial solution (Fayose and Azeez, 1972) along with the dolomitization process earlier mentioned. Also lack of coralline algae can be implied as the reason for low magnesium content (Boboye and Omotosho, 2017), as these beds are deposited in shallow marine environment where these species are not abundant. The low composition of magnesium can be attributed to an increase in organism complexities (advanced phyla) based on this premise, a shallow marine environment of deposition is suggested (Boboye and Omotosho, 2017).

4.2.2 Ternary Plot

Aluminium occurs in low amounts ranging from 0 – 15% as it is easily weathered and pretty unstable, meanwhile leaching from the laterized top soil could induce a ferruginization process responsible for the high iron content seeing that the silica is also high and is not so farfetched as it is not easily weathered (Fig. 4.6). Fe in

SWM-02 to SWM-05, especially in SWM-03, which is obviously ferruginized, and SWM-02 and 04 are shales while SWM-05 is Glauconite and this is due to the percolation of meteoric waters through shale and glauconitic layers into the underlying limestone units while the little found in the other Limestones are leachates (Boboye and Omotosho, 2017).

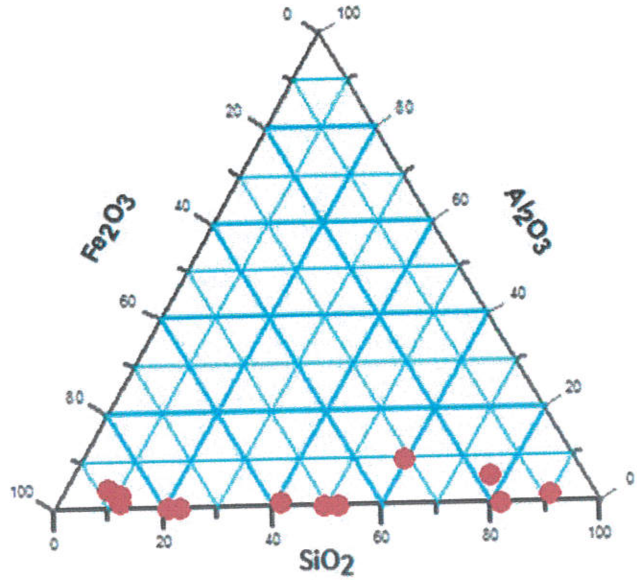

Fig. 4.8: Fe_2O_3–Al_2O_3–SiO_2 Ternary plot

4.2.3 CaO/SiO_2 Distribution and Relationship

At the shallow part of the upper continental crust where silica is readily made available through weathering and erosion, calcite reacts with it to form a calc-silicate at low temperature and pressure. Also, at all stages of diagenesis, dissolved silica (derived from dissolution of siliceous tests of marine organisms) replaces calcite. Fluvial silica input often augment dissolved silica in the basin at the unset of shallow marine condition. However, $CaCO_3$ production dominates as shallow marine conditions become fully established and silica supply from the hinterland reduces. Hence the higher the SiO_2 input, the more the continental influence, whereas the $CaCO_3$ production signals shallow marine incursion (Ehinola, *et al.*, 2012). Hence, the very high amount of CaO observed in and low silica is indicative of the Shallow marine environment and reduced continental influence on the Marine sediments comprising on non-calcareous shells. The low alumina content confirms a low index of weathering of the alumino-silicates such as feldspars and micas in the adjacent basement areas during transportation and deposition prior to diagenesis (Brand, 1983). Fe_2O_3 is usually derived from intense chemical weathering of heavy mineral such as the ferromagnesians. Its low value indicates that the environment of deposition is a reducing one that does not favour the precipitation of Iron (II) to Iron (III) and thus leached away (Brand, 1983). SO_3 is low probably because anoxic conditions prevailed in such quiet, low energy environments and there is rapid rate of sulphate reduction.

4.2.4 Correlation between Variables

The measured chemical parameters analyzed have been presented as bivariate plots and further subjected to Pearson's correlation analysis in IBM SPSS 20 Software. The correlation coefficient were determined in order to pinpoint relationships between variables, which serves as indicators of the overall coherence of the data. Initial information about the correlation structure was obtained from the correlation matrix of the 11 variables (Table 4.4). Correlations fluctuate -0.295 (for CaO *vs*P_2O_5) to 0.976 correlation between SiO_2 *vs* Al_2O_3). Only correlation coefficients larger than 0.500 were considered to have high mutual correlation, while those below –0.500 high indirect mutual correlation. The two-tailed Pearson's correlation plot is observed to have strong positive correlation between MgO and P_2O_5, MgO and K_2O, MgO and MnO, MgO and Fe_2O_3, Al_2O_3 and SiO_2, Al_2O_3 and SiO_2, Al_2O_3 and Fe_2O_3, SiO_2 and TiO_2, SiO_2 and Fe_2O_3, P_2O_5 and MnO and lastly, MnO and Fe_2O_3. Also, a strong negative correlation exist between Al_2O_3 and CaO,

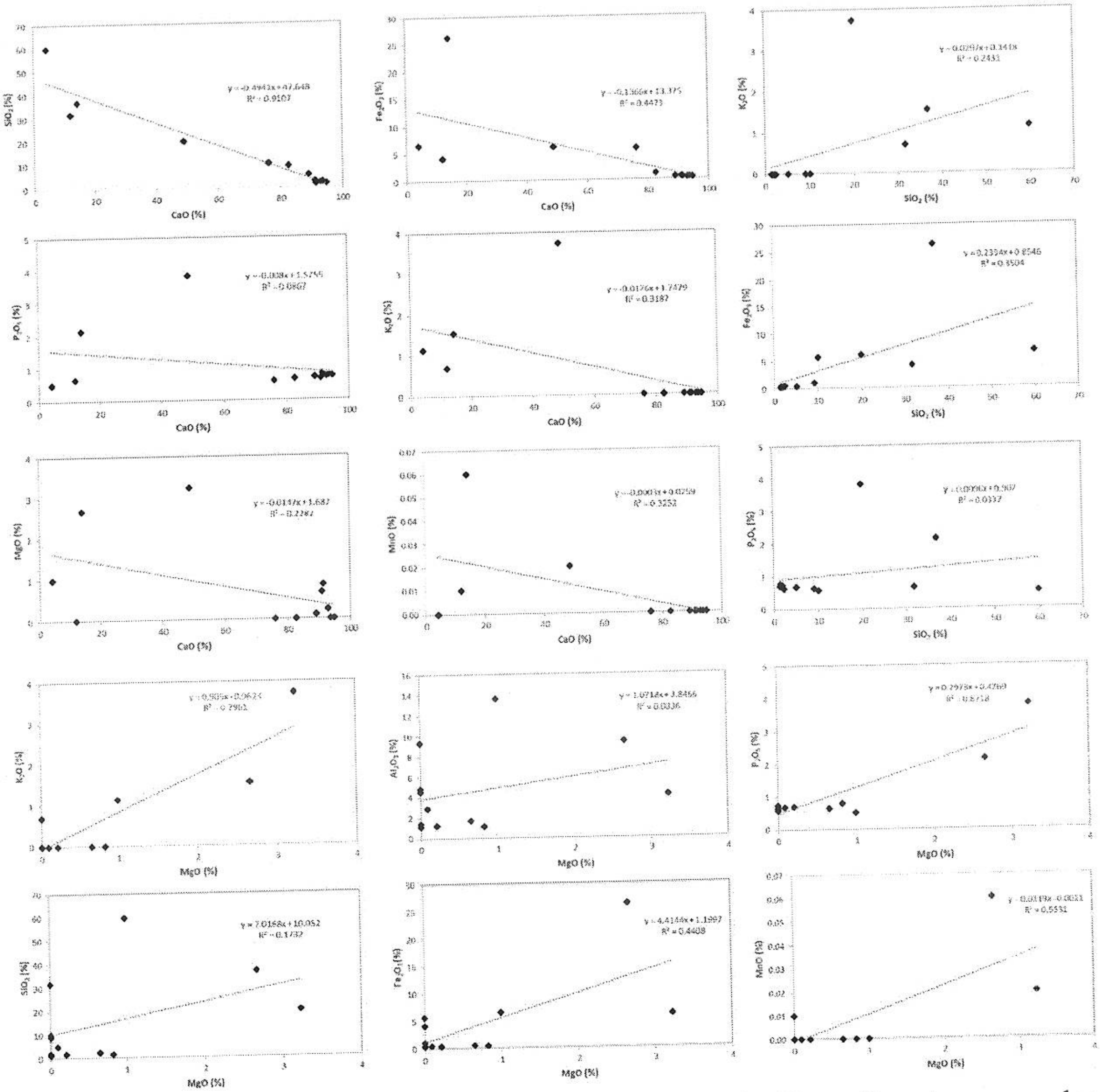

Fig. 4.9a: Bivariate scatter plots of the major oxides in the Ibese limestone samples

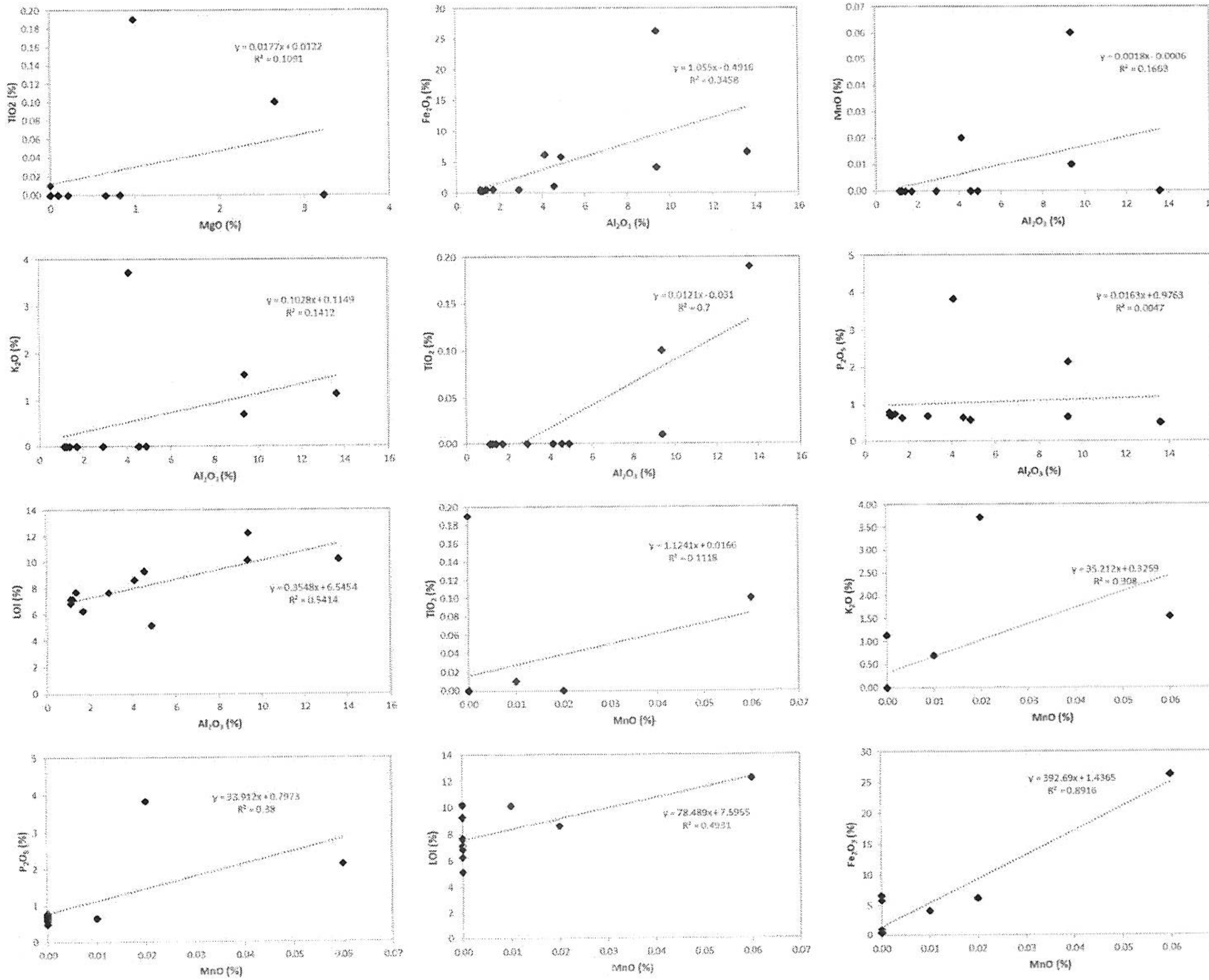

Fig. 4.9b: Bivariate scatter plots of the major oxides in the Ibese limestone samples

Table 4.4: Correlation matrix for the major element geochemistry.

	MgO	Al_2O_3	SiO_2	P_2O5	SO_3	K_2O	CaO	TiO_2	MnO	Fe_2O_3	LOI
MgO	1.000	-	-	-	-	-	-	-	-	-	-
Al_2O_3	0.289	1.000	-	-	-	-	-	-	-	-	-
SiO_2	0.416	0.976^{**}	1.000	-	-	-	-	-	-	-	-
P_2O_5	0.907^{**}	0.069	0.183	1.000	-	-	-	-	-	-	-
SO_3	0.016	0.551	0.511	0.038	1.000	-	-	-	-	-	-
K_2O	0.892^{**}	0.376	0.493	0.927^{**}	0.263	1.000	-	-	-	-	-
CaO	-0.478	-0.947	-0.954	-0.295	-0.671	-0.565	1.000	-	-	-	-
TiO_2	0.330	0.837^{**}	0.891^{**}	0.002	0.149	0.272	-0.743	1.000	-	-	-
MnO	0.744^{**}	0.408	0.432	0.616^{*}	0.132	0.555	-0.570	0.334	1.000	-	-
Fe_2O_3	0.664^{*}	0.588^{*}	0.592^{*}	0.460	0.097	0.470	-0.669	0.543	0.944^{**}	1.000	-
LOI	0.489	0.736^{**}	0.750^{**}	0.329	0.456	0.470	-0.800	0.625^{*}	0.702^{*}	0.690^{*}	1.000

**. Correlation is significant at the 0.01 level (2-tailed).

*. Correlation is significant at the 0.05 level (2-tailed).

SiO_2 and CaO, SO_3 and CaO, K_2O and CaO, CaO and TiO_2 and lastly, CaO and MnO.

4.3 Mineralogical Analysis Results

Two samples taken from limestone units of the Ewekoro Formation, one from each litho-section, have been analysed with XRD technique in order to assess the mineralogical composition. The NM-03 sample is composed of calcite (57.96%), quartz (26.17%), dolomite (9.88%) and kaolinite (5.99%). The SWM-07 sample however, is made up of calcite (56.69%), quartz (29.00%), dolomite (8.91%) and kaolinite (5.40%). Figure 4.8 and 4.9 shows the XRD patterns of the limestones. Results revealed that the limestones contains calcite as the predominant mineral phase followed by quartz, while dolomite and kaolinite are minor. The kaolinite may serve as a cementing material for the calcite in limestone. The summary of the mineralogical composition of the sample form the XRD analysis are presented in Tables 4.5 and 4.6 respectively.

4.4 Petrography and Diagenesis

The petrological studies of the thin sections were used to evaluate the limestone framework which consists of the allochems, micrite, sparry cement and ferruginous fragments and also, samples obtained from the glauconitic layer (SWM-05). Attempts to impregnate and lap the shale samples were futile. The allochems constitute about 70% of the rock fabric. Micrite cement is next in abundance at about 15%, sparry calcite is low at 5% and ferruginous fragments are sparsely present at about 10%. The result of the petrological classification of the limestones is presented in Table 4.5. The petrological classification of these limestones shows that they are mainly biosparites and fewer pelsparites and intrasparites (Folk, 1959) or packstones and grainstones (Dunham, 1962).

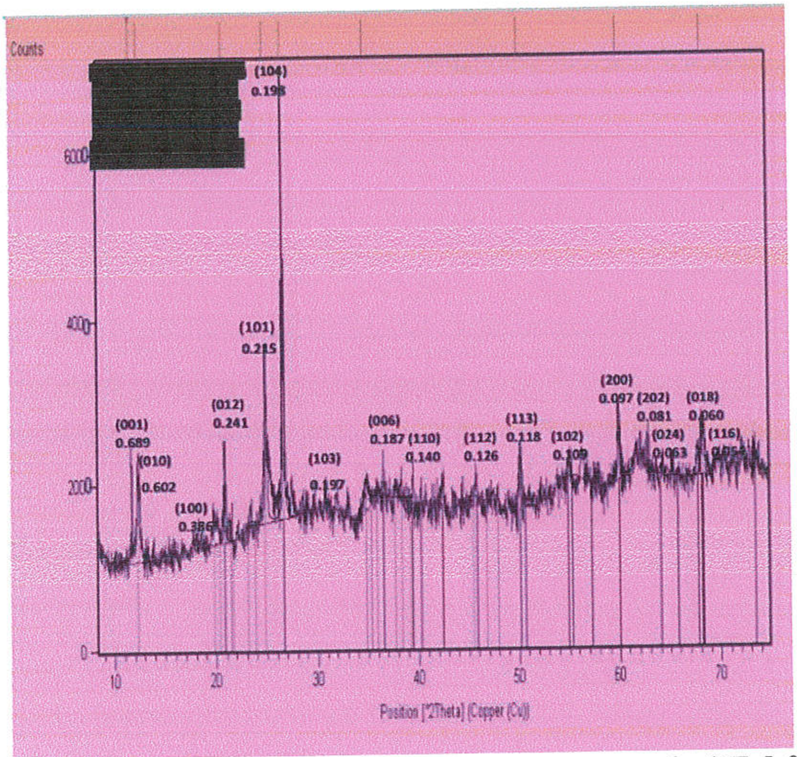

Fig. 4.10: X-ray diffractogram of limestone sample (NM-03)

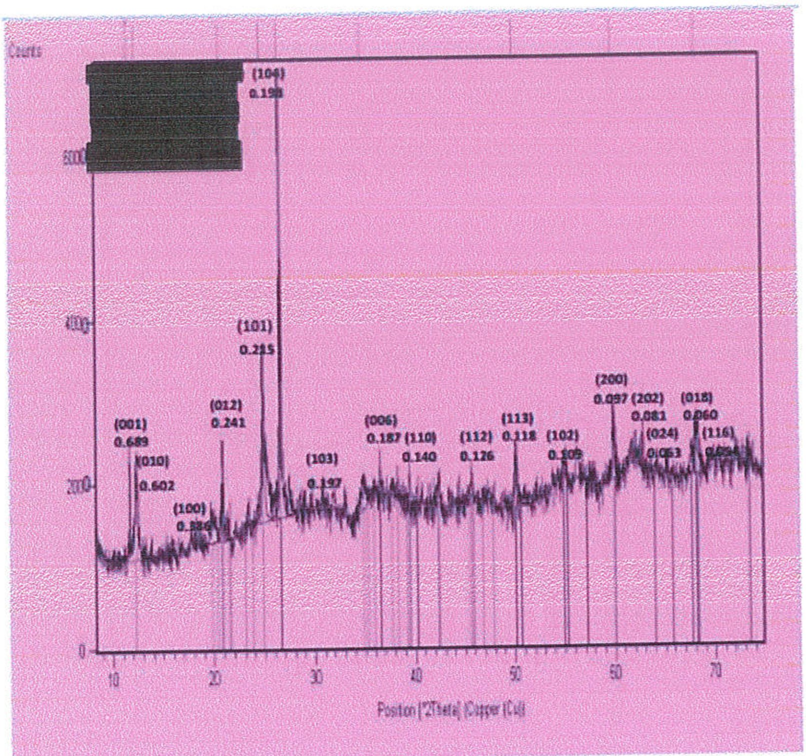

Fig. 4.11: X-ray diffractogram of limestone sample (SWM-07)

Table 4.5: XRD Diffraction Pattern for NM-03

Peak	2θ/degree	Plane	d-Valve (A°)	Intensity (%)	Compound	Formula
1	11.96	001	0.689	2621	Kaolinite	$Al_2Si_2O_5(OH)_4$
2	18.34	100	0.386	1736	Dolomite	$CaMg(CO_3)_2$
3	20.92	012	0.241	2842	Calcite	$CaCO_3$
4	24.98	101	0.215	3724	Quartz	SiO_2
5	26.74	104	0.198	7970	Calcite	$CaCO_3$
6	36.69	006	0.187	2523	Calcite	$CaCO_3$
7	39.22	110	0.140	2268	Calcite	$CaCO_3$
8	45.96	112	0.126	2212	Quartz	SiO_2
9	50.06	113	0.118	2591	Dolomite	$CaMg(CO_3)_2$
10	55.28	102	0.109	2275	Quartz	SiO_2
11	60.00	200	0.097	3248	Quartz	SiO_2
12	62.13	202	0.081	2624	Calcite	$CaCO_3$
13	64.07	024	0.063	2206	Calcite	$CaCO_3$
14	68.03	018	0.060	2742	Calcite	$CaCO_3$
15	69.51	116	0.054	2198	Calcite	$CaCO_3$

Table 4.6: XRD Diffraction Pattern for SWM-07

Peak	2θ/degree	Plane	d-Valve (A°)	Intensity (%)	Compound	Formula
1	11.96	001	0.689	2621	Kaolinite	$Al_2Si_2O_5(OH)_4$
2	12.42	010	0.602	2581	Quartz	SiO_2
3	18.34	100	0.386	1736	Dolomite	$CaMg(CO_3)_2$
4	20.92	012	0.241	2942	Calcite	$CaCO_3$
5	24.98	101	0.215	3764	Quartz	SiO_2
6	26.74	104	0.198	7920	Calcite	$CaCO_3$
7	32.07	103	0.190	2092	Calcite	$CaCO_3$
8	36.69	006	0.187	2523	Calcite	$CaCO_3$
9	39.22	110	0.140	2268	Calcite	$CaCO_3$
10	45.96	112	0.126	2212	Quartz	SiO_2
11	50.06	113	0.118	2591	Dolomite	$CaMg(CO_3)_2$
12	55.28	102	0.109	2275	Quartz	SiO_2
13	60.00	200	0.097	3248	Quartz	SiO_2
14	62.13	202	0.081	2620	Calcite	$CaCO_3$
15	64.07	024	0.063	2216	Calcite	$CaCO_3$
16	68.03	018	0.060	2742	Calcite	$CaCO_3$
17	69.51	116	0.054	2198	Calcite	$CaCO_3$

In most of the thin sections studied, the allochems are generally elongate and poorly sorted. The allochems consists mainly of fossils and pellets. Intraclasts are abundant in the limestones. Some of the Limestones contain detrital quartz grains (NM-04). The fossils (macroscopically speaking) consist mainly of, gastropods and bivalves shell fragments, some of which have been recrystallized to calcite. The fossils contain some nucleus of calcitic replacement or recrystallization of aragonitic shells which is known as neomorphism (NM-05). The calcitic replacement as well as the absence of micrite suggests some considerable amount of solution in the limestones. Some of the gastropods and bivalves shells have been recrystallized to calcite. Foliated structures were also observed in some of the thin sections. The foliated structures observed in the limestones are characteristic of oysters. In some thin sections, bivalve fragments were observed to consist of equant calcite spars. The limestones also contain some peloids, which are in some cases are micritized gastropod and bivalve fragments.

Table 4.7: Petrographic classification of limestones in this study

Sample Location		Composition of Limestone (in wt%)				Composition of allochems (in wt%)				Dunham (1974) Classification scheme	Folk (1962) Classification scheme
Label	Descriptio	Allochems	Micrite	Sparry cement	Ferruginized fragment	Intraclasts	Oolites	Fossils	Pellets		
NM02	Limestone	70	20	10	30	30	0	27	43	Wackestone	Sparse Biomicrite
NM03	Limestone	33	3	50	24	40	2	40	28	Packstone	Unsorted Biosparite
NM04	Limestone	30	50	5	15	50	5	30	15	Wackestone	Sparse Biomicrite
NM05	Limestone	20	10	50	20	20	10	20	50	Packstone	Unsorted Biosparite
NM06	Limestone	2	80	2	16	0	0	5	95	Mudstone	Fossiliferous micrite
SWM06	Limestone	65	20	5	10	50	10	20	20	Wackestone	Packed Biomicrite
SWM07	Limestone	10	0	60	30	20	30	10	40	Boundstone	Unsorted Biosparite
SWM08	Impure Lst.	40	3	20	37	30	20	30	20	Grainstone	Unsorted Biosparite

4.4.1 Diagenetic Features of the Limestones

Diagenesis encompasses all the processes, which affect the sediments after deposition. Due to the soluble nature of calcium carbonate, diagenesis occurs more readily in limestones than in terrigenous siliciclastic rocks. In fact, diagenesis can take place at surface conditions through changes in the pore water chemistry (Prothero and Schwab, 2014). Petrological studies of thin sections of limestones from the study area have revealed that the limestones have experienced six different types of diagenetic effects. It includes processes such as cementation to produce limestone, dissolution to form cave systems, development of micro porosity and changes in the trace elements. Diagenesis in the limestones is observed from the textural and mineralogical characteristics of limestones. The diagenetic influences on the limestones include cementation, micritization, neomorphism, dissolution, dolomitization and compaction which lead to fracturing.

1. Cementation: Cementation is defined as the process of precipitation of space filling crystals (Adams and McKenzie, 2001). Cementation was also defined by as one of the most important diagenetic processes that help in forming a hard limestone from weak sediment (Saffar, *et al.,* 2010). In the study area, the cement type observed include mainly the carbonate type (calcite and aragonite) and some iron oxide. The main cement types in these limestones include blocky or equant mosaic calcite cement, drusy mosaic calcite cement, acicular aragonite cement and micritic calcite cement (NM-04). High carbonate supply appears to favour aragonite precipitation too, so that where fluid-flow rates are higher, as in very permeable reef-rocks and lime sands, aragonite will be precipitated in preference to high-Mg calcite, which will tend to occur in less permeable sediments.

2. Micritization: Micritization is the process whereby the margins of carbonate grains are replaced by micrite at or just below the sediment/water interface. The process involves microbes attacking the outside of grains by boring small holes in them, which are later filled with micrite cement (Adams and MacKenzie, 2001). In (NM-03, 04, 05, SWM-06 and 08) parts of the bioclast surfaces are micritized.

3. Neomorphism: is defined as the process leading to the formation of large sized crystals (Saffar, *et al.,* 2010). Neomorphism is an important process in the overall diagenetic transformation of these limestones as observed in thin sections with large crystals of calcite (NM-04).It is marked by:(1) Irregular or curved intercrystalline boundaries, commonly with embayments (contrasting with the plane intercrystalline boundaries of sparite cement) (2) very irregular crystal-size distribution and patchy development of coarse mosaic;(3) gradational boundaries to areas of neomorphic spar;(4) presence of skeletal grains floating in coarse spar (Tucker, 2001).

4. Compaction: is an important aspect of diagenesis because it is a direct function of overburden pressure. In this study compaction processes is suggested by the fracturing of coarse recrystallized calcite crystals as observed in NM-06. Mechanical compaction in grainy sediments leads to a closer packing of the grains and a rotation of elongated bioclasts towards the plane of the bedding. As the pressure increases the bioclast can even become fractured as in the case of NM-04 (XPL). Another type of chemical compaction is the result of increased solubility at grain contacts and along sedimentary processes when under applied stress. Mostly this is the result of overburden but tectonic stresses also give rise to pressure solutions (Tucker, 2001).

5. Dissolution: in terms of porosity and permeability, some parts of the rock unit under study can be said to be very porous and permeable while some other sections may not be as porous. This factor is a direct function of the solution cavities (dissolution). All samples show good porosity with the exception of NM-05 and SWM-05, where porosity is thought to be impaired due to the intense sparry cementation and micritization.

6. Dolomitization: Dolomitization is the process by which calcium carbonate rock alters to dolomite. This process obscures the original texture, which makes classification and genetic interpretation difficult as seen in NM-06, NM03 and SWM07. This observation supports the evidence provided by the XRD results with 14% dolomite (Table 4.5 and 4.6) and this is seen with micrite along with drusy cement as its matrix in thin section.

4.4.2 DIAGENETIC AND ENVIRONMENTAL IMPLICATION

The fossil contents include sponges, echinoids, bivalves, coralline algae, pelecypod which are typical of an open shelf environment. The presence of echinoid indicates deeper waters. The dominant support is mud and muddiness is generally a property of rocks deposited in quiet water and a low energy environment, while the presence of large gastropod, bivalves, and Cephalopods shells indicated sudden increase in velocity of the water and rapid deposition from the continent. The absence and presence of shell fragments in some slides is indicative of this. There is absence of shells in NM-03 and SWM-07 as showing in the scanning electron microscope.

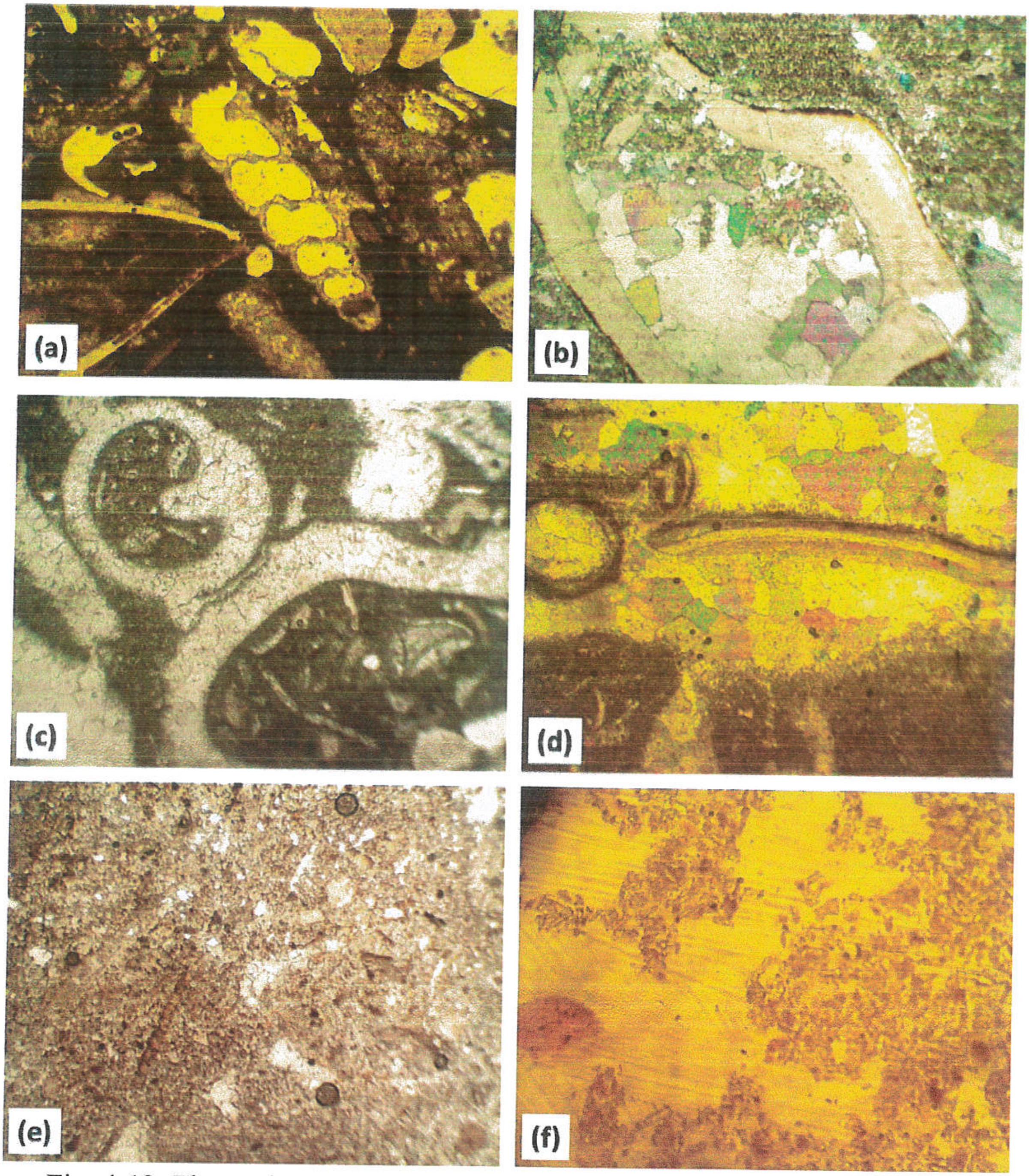

Fig. 4.12: Photomicrograph showing the different features of the limestone samples (a) NM-02 (XPL): evidence of mouldic forms with cements in pore space cementation, (b) NM-04 (XPL): evidence of compaction, (c) NM-04 (PPL): neomorphic features observed, (d) NM-03 (XPL): cementation features, (e) NM-05b (PPL): Dissolution, (f) NM-06 (PPL): Twinning is typical of calcite while drusy cement is probably due to dolomitization.

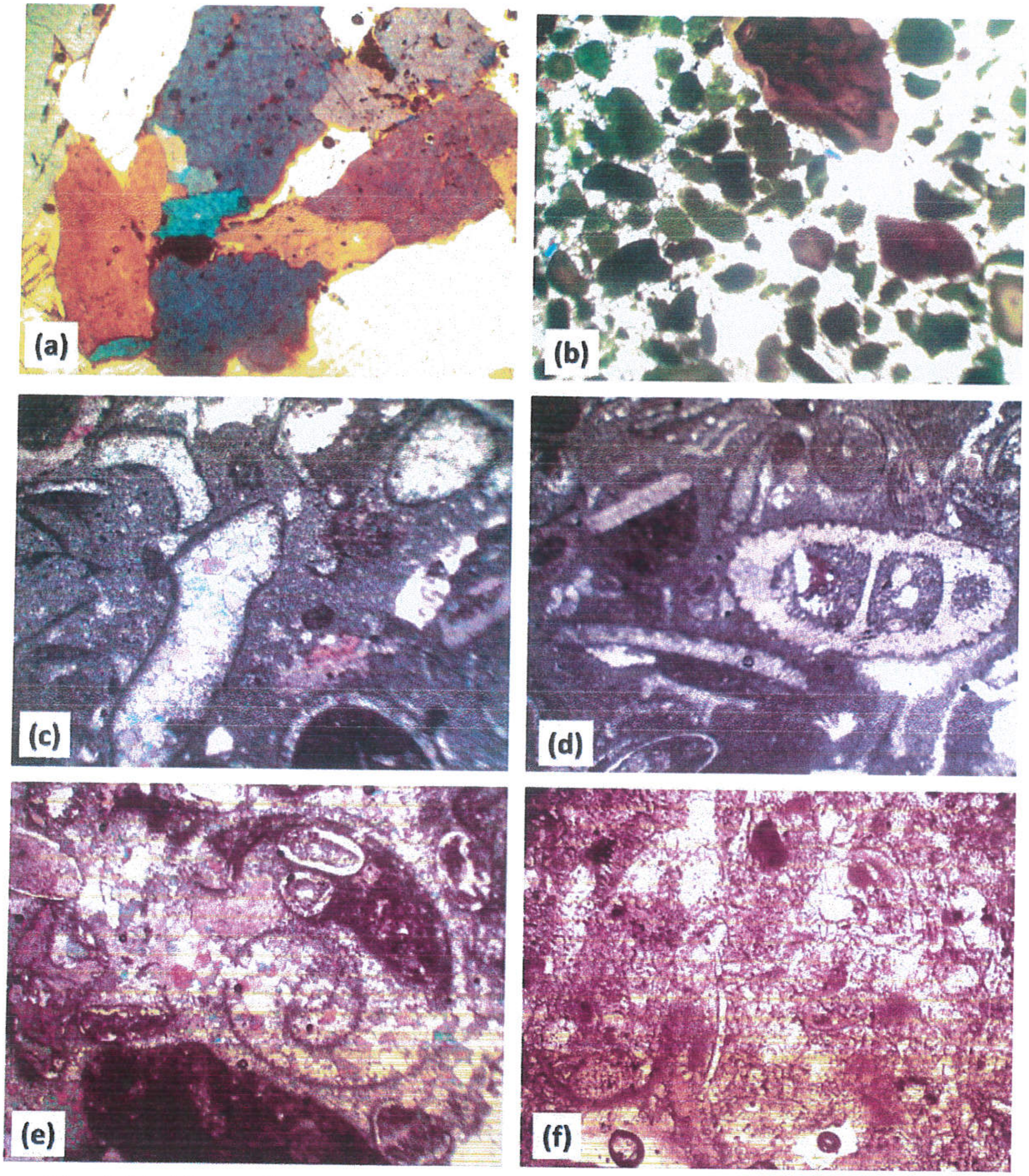

Fig. 4.13: Photomicrograph showing the different features of the limestone samples (a) SWM-03 (XPL) - cementation observed, (b) SWM-05a (XPL): Micritization features are showing, (c) SWM-06a (XPL): Neomorphic features observed, (d) SWM-06 (PPL): Evidences of Compaction, (e) SWM-07a (XPL): micritization features on bioclasts, (f) SWM-08a (PPL): Neomorphism is evident.

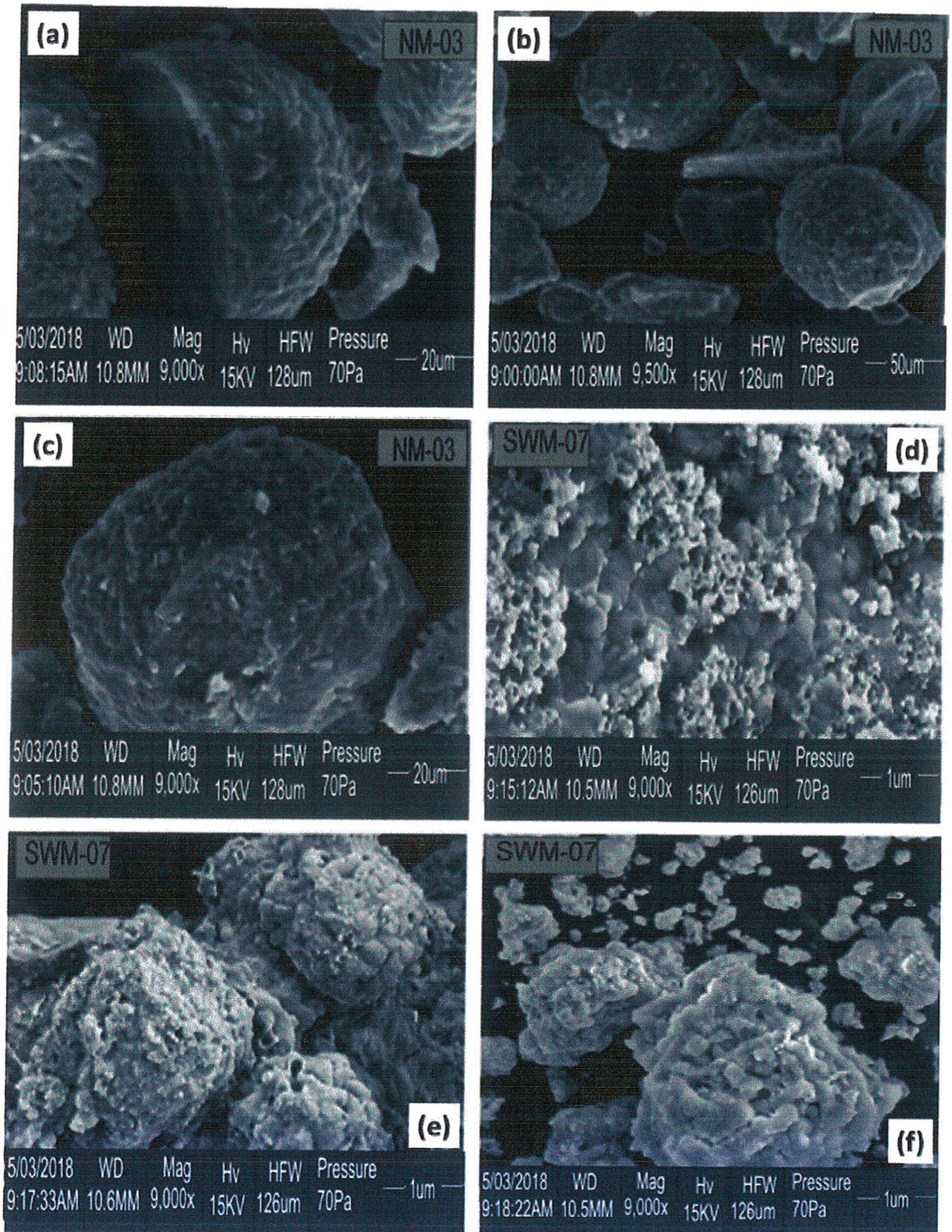

Fig. 4.14: SEM images of NM-03 and SWM-07 samples.

CHAPTER FIVE

SUMMARY, CONCLUSION AND RECOMMENDATION

5.1 Summary

Stratigraphically, the Ewekoro Formation being dominantly Limestones, is overlain by the Akinbo Formation, which constitutes the thin ferruginized sandstone, shales and Glauconite. The entire sequence is of Paleocene to Eocene in age.

Geochemically, the magnesium content of the carbonate rock is low when compared to calcium, while the average percentage composition ranges between 0.24%. This low composition is marked by the increased presence of advanced phyla than simpler organisms like algae, based on this premise, a shallow marine environment of deposition is suggested. The presence of SiO_2, K_2O and Al_2O_3 is an indication of detrital input and degree of diagenesis on the lithofacies.

Petrographic studies of Ibese limestone deposit have shown that the rock is highly fossiliferous with the identified fossils indicating deposition in an open shelf environment. Moreover, the limestone deposit was equally observed to be principally mud supported which is indicative of rocks deposited in quiet water and a low energy environment. From the aforementioned textural characteristics, Ibese limestone deposits can be classified as mainly wackestone according to Dunham, (1962).

The industrial potential of the area include sandstones, shales and limestones which constitute essential building and construction materials. Limestones is used in the manufacturing of cement and also used as filler in industrial products such as asphalt, rubber, paint, plastics and fertilizer

5.2 Conclusion

Detailed mapping and logging of the study area showed the total thickness of the limestone ranged from 18.0 m to 21.0 m. The limestone consists of dirty white to white fossiliferous units. The major element percentages revealed an increase in CaO with depth and an average of 56% and also a corresponding increase in MgO with average of 0.4% which show the calcitic to dolomicitic nature of the deposit. The significant occurrence of Al_2O_3 and Fe_2O_3 in the sequence is an indication of continental influence in the diagenetic history of the sedimentation. Four different Facies were recognized based on Folks classification, they are sparse biomicrite, Packed biomicrite, Unsorted biosparite and Fossiliferous micrite. The predominance of micrite as the cementing matrix revealed that the rock was deposited in a quiet shallow inner shelf environment occasioned by storm waves (Boboye and Nwosu, 2013). The elemental composition of the rock units suggested the suitability of deposit for cement production.

5.3 Recommendation

A more detailed study should be carried out with drill core samples across the Ewekoro and Akinbo Formations in order to validate the findings from this research. Further studies could look into the sequence stratigraphy of the same study area to understand certain deposition process better.

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