GEOLOGY, MINERALOGY AND GEOCHEMISTRY OF IRONSTONE EXPOSED AROUND MANIGI LOCALITY, NORTHERN BIDA BASIN, NIGERIA

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MTech/SPS/2017/7398

DEPARTMENT OF GEOLOGY

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

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A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES, FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA, NIGERIA IN PARTIAL FULFILMENT OF THE REQUUIREMENTS FOR THE AWARD OF THE DEGREE OF MASTERS OF TECHNOLOGY IN GEOLOGY (MINERAL EXPLORATION)

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ABSTRACT

The geology, mineralogy and geochemistry of Ironstone around Manigi locality was investigated in order to provide a comprehensive data on the geological, geochemical and mineralogical composition. Field work, sedimentological logging, and sample collection were carried out at a road cut near Manigi in the northern part of Bida basin. Sixteen representative ironstone samples were subjected to geochemical and mineralogical studies using X-ray fluorescence spectrometry, X-ray diffraction techniques and transmitted light microscopy. The sedimentological logging revealed the geology of the area to comprise of two main formations, the Enagi overlain by the Batati Formation. The result of the chemical analysis revealed that the concentrations of Fe_2O_3 ranges from 28.73 - 85.60 w% with an average of 52.65w% and averages of SiO₂, Al₂O₃, and TiO₂ are 33.5w%, 4.7w% and 0.9wt% respectively. Other components such as BaO₂, CuO, CrO₃, MnO, CaO, K₂O, ZnO, Br, MgO, Rb₂O, ZrO₂, CdO, TaO₅, PbO, and HfO₂ exist in a negligible amount. Calculation of the grade shows that the ironstone grade is 36.823% suggestive of low grade ore. Similarly low concentration of MgO ranging from 0.001wt% to 0.11wt% with an average of 0.025wt% and absence of sulfur in the ironstone shows that the sediment were deposited in a non marine or shallow marine environment. The mineralogical analysis revealed the presence of goethite and hematite as the major iron bearing minerals and quartz as major gangue materials. Thin section petrography reveals quartz as the predominant framework grain and iron cement as the cementing material. The floating contact displayed by the framework grains suggests that the iron cements were eodiagenetic in origin. It can be more useful as cast iron, although adequate beneficiation (to remove excess silica) and transformation of goethite to hematite can make it useful in the production of iron and steel. Further studies to reveal the tonnage, extent and reserve estimation should be undertaken.

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ABBREVIATIONS

RMRDC	Raw Material Research Development Council
US EPA	United State Environmental Protection Agency
MII	Mineral Information Instittute
BIF	Banded Iron Formation
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
NGSA	Nigeria Geological Survey Agency
LOI	Loss on Ignition
SW	South West
NW	North West
SE	South East
NE	North East

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

The level of industrialization of a country is measured by their iron and steel development because they are the most widely used engineering materials for production, fabrication, construction and manufacture of most items including ships, automobile, domestic appliances and military hardware. This shows the reason why per capital consumption of steel is an index for measuring development in the economy of every country. The accessibility and development of the iron and steel sector is of principal significance for industrial growth, increased engineering capacity and enhancement of technical skills (Raw Materials Research and Development Council, 2010).

This thesis explores the geology, geochemistry and mineralogy of ironstones most especially those occurring in Phanerozoic sedimentary rocks in Bida Basin. The research is confined to the Ironstone exposed around Manigi locality in the extreme part of Northern Bida basin, located in Mashegu local government area of Niger State, Nigeria. It is the least investigated ironstone in the basin and the basin also is the least investigated among the major inland sedimentary basins in Nigeria. Ironstone occurrences in this area are hosted in the Campanian to Maestrichtian rocks associated with sandstone, siltstone, and claystones. The ironstone was correlated with host rock by using mineralogical, geochemical and sedimentological evidences. The elemental abundance of iron varies between about 5% of the Earth's crust and as much as 80% of the planet's core (Morgan and Anders, 1980). This makes iron the fourth most abundant element on the earth crust. Iron oxide and hydroxide make up the primary iron ore minerals, because of their large amount of iron content and occurrence as large tonnage surface deposit (Frenczi, 2001). However, iron is present in low concentration in most parts of the earth, thus a deposit must have a high percentage of the metal to be considered ore grade for economic purposes. Ideally, a deposit must contain at least 25% iron to be considered economically recoverable (Whiten and Brooks, 1972). The presence of the amount of iron in rocks varies from an average of 2-3% in sedimentary rocks to 8.5% in igneous rocks (United State Environmental Protection Agency, 1994).

Iron ores are rocks and minerals from which metallic iron can be extracted. An iron ore deposit is a mineral body of sufficient size, iron content, and chemical composition with physical and economic characteristics that will allow it to be a source of iron either immediately or potentially (Kennedy, 1990). Iron ore is the raw material used to make pig iron, which is one of the main raw materials to make steel. Approximately 98% of the mined Iron ore is used to make steel (Mineral Information Institute, 2006). Ironstone is any rock that contains great percentage of iron minerals such as Haematite, Goethite or Limonite, Magnetite, Siderite, Ilmenite, Chamosite, and Pyrite. However for Ironstone to be useful in iron and steel industry, it must contain 25-35% Iron (Fe), < 0.3% Sulfur and < 0.4% Phosphorus (Whitens and Brooks, 1972). Even though iron exist in many minerals, five primary sources of Iron are the Magnetite (Fe₃O₄), Hematite (Fe₂O₃), Goethite/Limonite (FeO(OH)), Siderite (FeCO₃), and Pyrite (FeS) (US EPA, 1994).

1.1.1 Types of iron ore deposits

Iron ores deposits are grouped in to different types. Evans, 1993; Robb, 2004; Boggs, 2006 classified iron ore deposits in terms of morphology, texture and mineralogy in to three main types, these are; Banded Iron Formation, Phanerozoic Iron, and Bog Iron deposits.

1.1.1.1 Banded iron formation (BIF)

These contain the bulk of the world's Iron ore resources. It has more reserve and total production value than the bog and Phanerozoic iron deposits. The banded iron formation is a stratigraphic unit composed of ironstone that may be cherty or non-cherty but with banded appearance. They were formed during substantially three periods throughout the Archean and Proterozoic earth history, namely 3500–3000 Ma, 2500–2000 Ma, and 1000– 500 Ma (Kimberley, 1989; Klein and Beukes, 1993). These periods are equivalent to different tectonic settings that are referred to as Algoma, Lake Superior and Rapitan types (James and Trendall, 1982; James, 1983; Maynard, 1991; Klein and Beukes, 1993; Bekker et al., 2010, 2014). Algoma types are associated with volcanic arcs and examples are found in the greenstone belts of Ontario Canada. The Lake Superior types are mostly located on the stable continental platforms, example the Harmesely basin of Western Australia, the Transvaal basin of South Africa etc. While the Rapitan types are found associated with the glaciogenic sediments formed during the major Neoproterozoic ice ages and the examples are found in the Mckenzie mountain of Northwest Canada (Holland, 1984; Maynard, 1991; Robb, 2004; Boggs, 2006; Bekker et al., 2014). Some of the main iron minerals in the banded iron formation are hematite (Fe₂O₃), magnetite (Fe₃O₄), greenalite (Fe₃Si₂O₅ (OH) 4), stilpnomelane (2(Fe, Mg)O (Al, Fe)₂·O₃·5SiO₂·3H₂O), minnesotaite ((Fe, Mg)₃ Si₄O₁₀ (OH)₂) and pyrite (FeS₂)(Deer *et al.*, 1992)

1.1.1.2 Phanerozoic ironstones

These ironstones were formed during Phanerozoic period and are usually referred to as the Phanerozoic ironstone deposits. The deposits are of widespread occurrence representing an important source of iron, before 1970s. They are Proterozoic to Cretaceous in age and are also referred to as the Oolitic or Pisolitic ironstone, occurring within marine terrigeneous sediment. They have low iron content (30-50% Fe) when compare with BIF-hosted deposits (55-65% Fe) (Ferenczi, 2001). The term iron-rich is restricted to sedimentary rocks that contain at least 15% of total iron (Kimberley, 1994).Boggs (2006) used the term ironstone for non-banded, noncherty, commonly Oolitic, Iron-rich sedimentary rocks while the term iron formation was allocated to the cherty, well-banded Iron-rich sedimentary rocks. The sedimentary ironstone deposits are thin sequences that formed in shallow marine or non-marine environments (Young and Taylor, 1989). Two types have been identified; ironstone deposits occurring in the Jurassic sediments of England and the Alsace-Lorraine region of France and Germany were referred to as Minette or Lorraine-type iron ores. Other types recorded in North America were known as Silurian Clinton type iron ores of Kentucky and Alabama that are analogues of the younger European deposits (Bottke, 1981; Barnes, 1989; Evans, 1993; Mücke and Farshad, 2005).

The ironstone deposits in the Phanerozoic were formed in different ways, the most common being the oolite type. This is mainly present in the Ordovician, Silurian, Jurassic, Cretaceous and Cenozoic periods but shows a wide stratigraphic range from Precambrian to Holocene (Petránek and Van Houten, 1997). Most Oolite ores are economically exploited and major development took place during the Ordovician and Jurassic periods. Ordovician Iron oolites are found in North Africa, Spain, Portugal, France, Germany, England, Poland, the Czech Republic (James, 1966). The Ordovician Oolitic Ironstones occur widely in marine shelf sequences of South west Europe (the Western European Platform), the Avalonian Terranes and in North Africa where they form the most important group of deposits of the two major periods of Phanerozoic ooidal ironstone generation (Ordovician-Devonian and Jurassic - Paleogene; (Young and Taylor, 1989). Van Hutton (1992) stressed that Cenozoic Ironstone is also of wide occurrence in the north central Africa and South west Europe in Paleocene, Eocene, Oligocene, Miocene and Pliocene episodes and mostly with ooid fabrics. Van Hutton, (1992), Petránek and Van Houten, (1997) revealed that the Cenozoic Ironstone occurs in Northern Pakistan, western Siberia, southern Germany, Northwestern Venezuela, Northeastern Colombia, Northwestern Romania, south-central United State of America (USA) and central North Africa. Some of the main iron bearing minerals recognized in the Phanerozoic ironstone include; hematite (Fe₂O₃), goethite (FeO·OH), siderite (FeCO₃), ankerite ((Ca, Mg, Fe)(CO₃)₂), ferroan dolomite (Ca Mg, Fe (CO₃)₂), ferroan calcite ((Ca, Fe)CO₃), pyrite (FeS₂), jarosite (KFe₃³⁺(SO₄)2(OH)₆), chamosite $(3(Fe^{2+},Mg)_5Al (AlSi_3O_{10})(OH)_8)$, berthierine $(Fe^{2+},Fe^{3+},Al, Mg)_2 \cdot 3(Si,$ Al)2O₅(OH)4), nontronite (NaO.3Fe₂ ((Si, Al)₄O₁₀) (OH)₂·nH₂O) and glauconite (K Mg (Fe, Al) (SiO₃)₆·3H₂O) (Deer *et al.*, 1992) in (Afify, 2016)

1.1.1.3 Bog iron deposits

The third type of iron ore deposits is the bog iron. They are mostly form in the swamp and lakes of glaciated tundra areas of the northern hemisphere examples occur in the northern Canada and Scandinavia (Boggs, 2006). Stanton (1972), Robb (2004) and Boggs, (2006) identified that the deposits are typically small and thin, and comprises concentrations of goethite and limonite associated with organic rich shale. They formed in recent geological periods in areas where Iron-bearing groundwater typically emerges as a spring. The iron is oxidized to ferric hydroxide upon encountering the oxidizing environment of the surface. Bog ore often combines goethite, magnetite, and vugs or stained quartz. Oxidation may occur through enzyme catalysis by iron bacteria. It is not clear whether the magnetite precipitates upon first contact with oxygen, and then oxidizes to ferric compounds, or whether the ferric compounds are reduced when exposed to anoxic conditions upon burial beneath the sediment surface and re-oxidized upon exhumation at the surface (http://www.encyclopedia.org). Akande *et al.* (1998) reported the occurrence of bog iron in the Agbaja formation of southern Bida basin.

1.1.2 Mechanisms of formation of iron ore deposits

Many literatures have so far been documented on the origin of iron ore and lots of contrasting hypothesis dealing with the iron ore deposits. Young and Taylor (1989) submitted that sedimentary ironstone deposits are thin sequences that formed in shallow marine or non-marine environments. James (1966) postulated that the formation of iron could involve the mobilization and transport of the ore forming metals by seawards flowing groundwater of continental origin. According to (Van Houten and Authur, 1989) the

formation of Ironstone could be related to a pattern of global tectonic cyclicity and specifically to times of continental dispersal and sea-level high stand, as well as periods of warmer climate and increased rates of chemical sedimentation. (Mücke, 2000; Mücke and Farshad, 2005) reveal that weathering of a variety of rocks under lateritic conditions and further transportation of iron through fluvial drainage systems into marine basins was pointed out as a model for ironstone formation. Also (Siehl and Thein, 1989) proposed that mechanical reworking, in-situ weathering and re-deposition from basement rocks in nonmarine basins via river systems are the main processes leading to the accumulation of Iron under pedogenic conditions. Their model simply shows the transfer of the pedogenic pisoids to a marginal marine setting with further mechanical abrasion and reworking/concentration of pisoids.

Sturesson *et al.*, 1999 stressed that hydrothermal solutions produce iron oolites in certain areas today and erosion of volcanic rocks can locally cause enrichment of the elements needed for ooid formation. According to (Dreesen, 1989; Sturesson, 1992; Sturesson *et al.*, 2000) Volcanism was a major source of iron leading to ironstone formation from volcanic ash with hydrothermal fluids enriching seawater in iron (Fe), Aluminium (Al) and Silicon (Si). Meanwhile, Kimberley (1989, 1994) also documented precipitation of iron from exhalative fluids allied with active faults. Other process that lead to the formation of different types of iron include; replacement of carbonates (Kimberley, 1979; Loope *et al.*, 2011), mechanical accretion of chamositic clay particles (Bhattacharya and Kakimoto, 1982; Van Houten and Burucker, 1984), precipitation in marine environment linked to

sedimentary exhalative hydrothermal processes in tectonically active areas (Hein *et al.*, 2016), and crystallization from precursor iron oxyhydroxide gels (Harder, 1989).

1.1.3 Iron ore deposits in Nigeria

It has been proven that Nigeria is blessed with abundant mineral resources (Bamali *et al.*, 2011). The country is endowed with numerous iron ore deposit which are known to occur in two forms; these are the banded iron formation which occurs in folded bands and lenses associated with the Precambrian metasedimentary schist belts and the Phanerazoic ironstone deposit (oolitic and pisolitic) deposits.

1.1.3.1 Banded iron formation (BIF)

Banded Iron Formation (BIF) occur in different location within the Precambrian Basement Complex of Nigeria associated with schist belts such as Lokoja-Okene-Kabba, Maru, Muro and Birnin Gwari schist belts (Figure.1.1).These rocks are commonly associated with the metasedimentary and metavolcanic rocks of late Proterozoic age. Three main facies of the BIFs in Nigeria are recognized; these include the oxide, silicate and sulphide facies. The most widespread been the oxide facies, represented by the banded silica-iron oxide assemblage. The silicate facies consists of the quartz-garnet grunerite assemblage, while the sulphide facies includes the pyrite-bearing carbonaceous schist or phyllite intercalated with iron-rich layers (Bolarinwa, 2017). Nigeria BIF has been investigated by various researchers such as (Olade, 1978; Adekoya, 1998; Mucke and Neumann 1986; Okonkwo (1980) Adekoya *et al.*, 2012). Itakpe iron ore deposit the Itakpe ore deposit around Okene are classified into a massive magnetite, a banded to granular hematite-magnetite, and a homogenous hematite-magnetite ores and they are regarded as a product of high grade amphibolite facies metamorphism of iron-rich sediments (Olade, 1978). Mucke and Neuman (1980) deduced from mineralogical studies, that a combine magmatic and contact metamorphic processes lead in to the formation of Itakpe iron ore because of the association of certain primary minerals such as magnetite which is rich in exsolution bodies such as ilmenite occurring with Kaolinite plus relics of feldspar in the same paragenesis correspond to typical intramagmatic titanomagnetite deposits. Okonkwo (1980) also reported the occurrence of a banded iron formation BIF in the phyllite and quartz micaschist of the Kushaka schist belt. Mucke and Neuman (1986) recorded magnetite rich deposit at Kukan, which was recorded to be igneous progenitor. They also reported dark iron-rich bands alternating with lighter quartz bands in the rocks of the Ajabonoko area. Adekoya (1998) and Adekoya *et al.* (2012) reported the occurrence of BIF within the pelitic to semi-pelitic phyllites of the Maru and Muro areas.

1.1.3.2 Phanerozoic ironstones

The second type of iron ore occurrence in Nigeria are the Phanerozoic ironstone also referred to as the Cretaceous sedimentary (Oolitic) Ironstone deposits. These can be found in Bida, Lokoja, Nsude and Taranji areas.

Records have shown the presence of sedimentary Ironstone in the Middle Niger basin also known as Bida or Nupe Basin in the central part of Nigeria. These Ironstones occur within the Upper Cretaceous alluvial and shallow marine facies sedimentary sequence of Bida basin (Ladipo *et al., 1994*). The basin host several Iron ore bodies, these include Agbaja, Lokoja, Patti, Bassa Nge, Ate, Sakpe, and Batati Ironstone. Cretaceous oolitic Ironstones deposits of appreciable reserves estimate variedly reported extensively occur over the basin. These ironstones were first recognized by earlier workers such as Falconer (1911), Du preez (1952), Jones (1955, 1958), Adeleye and Dessauvagie (1972), Adeleye (1973).

Three contrasting models were proposed on the origin of ironstone in the Bida basin. These are; the sedimentary formation, lateritic and post- diagenetic genesis. (Falconer, 1911; Adeleye and Dessauvagie, 1972; Adeleye, 1973 and Oresajo, 1979) proposed sedimentary origin for the ironstone because of the depositional characteristics and field relationship of the ironstone to other sedimentary rocks in the basin. However Du preez (1956) suggested lateritic origin because of the presence of fossils in the ironstone beds. Also lateritic origin was suggested due to the presence of the iron oxide and hydroxide in the ooids (Jones 1955, 1956, 1958; Kogbe 1978). Lastly a post- diagenetic iron enrichment model was suggested by (Hasse, 1993; Mucke, 1994; Lapido *et al.*, 1994; Abimbola, 1994).



Figure1.1 The location of BIFs in the geological map of Nigeria (Adapted from Bolarinwa, 2018)

	Percentage Occurrence (%)										
Location	Fe	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	CaO	MgO	P ₂ O ₅	MnO	TiO ₂	Reserved (tones)	Extent of dev. as at 2012
Agbaja	45-54	62.64	8.55	9.06	0.72	0.38	4.16	0.14	0.37	2billion	Exploration and development on going.
Itakpe	38-45	53.10	44.80	1.00	0.30	0.20	0.05	0.05	0.10	200-300 million	Operational but moribund.
Ajabanoko	35.61	47.74	0.41	-	-	-	0.11	0.05	0.06	30 million	Exploration and development on going
Chokochoko	37.43	47.65	4.30	-	-	-	0.05	0.52		70 million	-
Agbade Okudu	37.43	29.41	0.62	-	-		-	-		70 million	-
Nsude Hills	37.43	-	-	-	-		-	-		60 million	-

Table1.1 Nigeria Ire	on ore reserves (%)) and extent of dev	elopment

Bamali et al., (2011)

1.3 Statement of the Research Problem

The global industrial revolution has generated the problem of high demand of iron and steel in most developed and developing nations like Nigeria because of its usefulness in engineering construction (building of bridges, sky scrapers, automobile, ship building, vehicles, pipes and other domestic appliances), production, fabrication and manufacture of most items including military hardware. In the world today, iron and steel are recognized as the keystones or bases to any country's industrial development and a means of accelerating socio-economic development (Amigun and Ako, 2009).

Nigeria is blessed with abundant deposits of iron ore (Banded iron formation and Cretaceous oolithic and pisolithic ironstones) cutting across several states of the Federation (Niger, Kwara, Kogi, Benue, Nasarawa, Plateuau, Bauchi, Oyo, Kebbi, Kaduna, Borno, and Anambra State) with an estimated reserve reported to be about 2.3 billion tones (Bamalli et al., 2011; Ohimain, 2013). Works of (Adeleye and Dessauvagie, 1972; Adeleye, 1973) indicate the presence of sedimentary ironstones (Oolithic and Pisolithic) around Sakpe and Batati locality in the Northern Bida basin, and are wide spread throughout the sub-basin. Adeleye (1973) categories Ironstones in the Northern Bida basin in to upper and lower ironstones, referred to as Batati and Sakpe Ironstones separated by the argillaceous Enagi Siltstone. Adeleye and Dessauvagie (1972) identified the type locality of Batati Ironstone at a small cliff, 7km South of Batati where it exposed approximately 5meters. They observed that the Ironstone consist of brown, yellow to white oolites in a yellow limonitic silty matrix, And are dominated by argilacious goethitic, oolitic ironstone and a subsidiary sideritic, chloritic and kaolinitic ironstone. These ironstone occurrences were not well assessed; their total reserves and chemical qualities have not been studied in detail. Hence their availability as sources of iron ores for Nigerian steel industry was not emphasized. Not much have been reported on the upper ironstone of the northern Bida basin especially those cropping out around Manigi locality in the northern extreme of the basin.

However researchers such as Umeroah (1987), Abimbola (1997), Akande *et al.* (1999), Agunleti and Salau (2015) and Imrana and Haruna (2017) have investigated the Agbaja ironstone which is the lateral equivalent of Batati Ironstone in the southern Bida basin.

1.4 Justification for the Research

Very little literatures have so far been documented on the upper Ironstone of Cretaceous sedimentary sequence of northern Bida basin. Must work carry out in the basin such as (Adeleye and Dessauvagie, 1972; Adeleye, 1974; Braide, 1992a; Olaniyan and Olubaniyi, 1996; Okosun et al., 2007 and Goro et al., 2014) focused on the stratigraphy, sedimentological aspect and depositional environment of the sediments. Although (Adeleye, 1973) observed the source rock of the ironstone through their combined petrology and facies association in space and time in the central part of the basin near Bida town. He did not extend to the extreme north of the basin. His use of petrographic thin section analysis failed to coin out the geochemistry of the Ironstone in the central part of the basin. Hence there is need for detailed study of the Ironstone using an integrated method of Geochemical and petrographic analyses. More recently Olurunfemi and Waziri (2018) investigated the geology, mineralogy and geochemistry of Sakpe Ironstone at Jima, near Bida. They reported that the ironstone is of high grade, low magnesium content and lack sulphur, which is an indication that the sediments were deposited in a non marine to shallow marine environment. The present work focuses on the geology, mineralogy and the

geochemistry of Batati Ironstone exposed around Manigi locality in the extreme northern Bida basin. The research will provide information the petrology and geochemistry of part of Batati Ironstone cropping out around Manigi, Kawo-Mashegu -Makera area in the extreme northern Bida basin.

1.5 Aim and Objectives of the Research

The aim of this research is to investigate the geology, mineralogy and geochemistry of Ironstone exposed at a road cut near Manigi along Jebba-Kaduna road, Northern Bida basin, Northwestern Nigeria. With the intention of assessing the ore grade of the Ironstone.

The objectives are to:

- Documents sedimentological features of the exposed part of the Batati Ironstone near Manigi locality
- 2. Determine the textural and mineralogical composition of the Ironstone
- 3. Determine the geochemistry of the Ironstone.
- 4. Assess the ore grade of the Ironstone using the percentage of elements of major interest.

1.6 Scope of the Research

The present work is restricted to field geological study which involved sedimentological logging of well expose outcrop section in the area to identify rocks lithologic units, geological mapping and collection of representative samples. Laboratory studies involving petrographic thin-section, mineralogy and geochemical analysis (trace and major elements

characteristics) of the representative samples collected from the field in order to achieve the above stated objective.



1.7 Study area

Figure1.2: Approximate location (red arrow) of the study area in a simplified geological map of Bida basin (Adapted from Okosun *et al.*, 2007)

1.7.1 Location extent and accessibility of the study area

The study was conducted within and around area between Manigi and Kawo-Mashegu localities in Mashegu local government area of Niger state in the North-central, Nigeria. It coordinates lies within latitude 9°45′20″ to 9°47′30″N and longitude 5°30′40″ to 5°33′20″E covering approximately 16 kilometer square in the extreme Northern Bida basin (Figure1.2). The area is traverse by major road (Kaduna- Jebba road), number of minor

roads and footpaths are used in order to access the area especially where good exposures are found. It is also drained by seasonal rivers and streams.

1.7.2 Relief and drainage of the study area

The topography of the area is an undulating terrain characterized by limited lowland and spotted outcrops forming mesas, consisting of different layers and varying litholofacies which include the basal Bida sandstone, Enagi Siststone/mudstone and the Batati Ironstone at the top. The ironstones occur as a cap on top of the NE-SW trending ridges (Mesas) that characterized the area.

The geomorphological features in the northern Bida basin consist of river Niger, its flood plain and tributaries characterized by belt of mesas and plains. The area is being drained by Rivers Niger and Kaduna. Other minor rivers and streams flow in to the main rivers, the streams are intermittent and thus dry off at the peak of dry season. Generally, the drainage pattern is simply dendritic, where many tributaries join the major river.

1.7.3 Climate and vegetation

The studied area has a climate that is related to its neighboring city (Kontagora) which lies within a tropical climate characterized by the alternating wet and dry season. In winter (Harmmatan), there is much less rainfall than in summer (raining season). This climate is considered to be Aw according to the Koppen Geiger climate classification. The average temperature here is 26.4°C in a year and the average rainfall is 1162 mm. The least amount of rainfall occurs in December and the highest occur in August-September with an average of 268mm. The temperatures are highest on average in April at around 30.2°C. In August,

the average temperature is 24.3°C this is the lowest average temperature of the whole year (<u>http://en</u>. climate.data.org/AM OP/)

Vegetation in the area is typical of Guinea savannah like in other parts of the country where this biome occurs, it is characterized by woodlands, where grasses occur either totally or mixed with other herbaceous or shrubby plants. The grasses are green in the wet season with fresh leaves, but become dry during the dry season when bush burning, fire wood cutting and other human activities are triggered.

1.7.4 Human geography and Land use

The people that settle in the minor and major settlement around the area are Nupe, Cambari and some Hausa-Fulani. The districts are mainly inhabited by peasant farmers that grow maize, guinea corn, and groundnuts during the rainy season. In the dry season, most of the inhabitants graze their animals, while others turn to other business activities like trading between nearby town and villages.

CHAPTER TWO

2.0 LITERETURE REVIEW

2.1 Preamble

Basically the geology of Nigeria is made up of three components, these are; the Precambrian Basement Complex (which consist of granite, gneiss, migmatite, migmatitegneiss, and schist), the Jurassic younger granite and the Cretaceous-Recent sedimentary inland basins which consist of different litholofacies ranging from sandstone, siltstone, clay stone, shale and conglomerate (Figure 2.1). There are six inland sedimentary basins in Nigeria and these include; The Anambra Basin, Dahomey Basin, Lower Middle and Upper Benue Trough, Chad Basin, Bida Basin and Sokoto Basin.



Figure 2.1: Geological sketch map of Nigeria showing different geological components (after Obaje, 2009)

2.2 The Bida Basin

The Mid Niger basin also known as Bida basin or Nupe basin is a NW-SE trending intracratonic sedimentary basin which extends from Kontagora in Niger state to the north to areas slightly beyond Lokoja in Kogi state to the south, covering a distance of approximately 400km (Figure 2.2). The Campanian to Maastritchian Bida basin is bounded in the Northeast and Southwest by the Basement Complex while it merges with Anambra and Sokoto Basins in sedimentary fill comprising post orogenic molasse facies and a few thin unfolded marine sediments (Adeleye, 1974).



Figure 2.2: Geology and location of the Bida Basin and Environs (After Obaje, 2011)

The basin has being investigated by several earlier workers, resulted in to a variety of contrasted hypotheses dealing with both the origin of the basin and the ironstones in the basin (Falconer, 1911; Jones, 1958; Russ, 1930; Dup reez,1952; Adeleye, 1971, 1973,

1974; Adeleye and Dessauvagie, 1972; Jan Du chene *et al.*, 1978; Allen, 1982; Whiteman, 1982; Mebradu *et al.*, 1986; Ladipo, 1988; Ojo and Ajakaiye,1989; Braide,1992a; Abimbola, 1994; Ladipo *et al.*, 1994; Vibkal 1999) and more recently (Udensi and Usazuwa, 2004; Akande *et al.*, 2005; Okosun *et al.*, 2007; Obaje,2009; Obaje *et al.*, 2011; Goro *et al.*, 2014).

Falconer (1911) was the first worker that investigated and described the upper Cretaceous rocks in the basin; he referred to them as the Lokoja series. (Russ, 1930) referred to it as the Nupe sandstone. It is subdivided in to two sub-basins known as the Northern and Southern Bida basins (Lokoja and Bida sub-basins) (Jones, 1958). Adeleye and Dessauvagie (1972) proposed the term Nupe group for the stratigraphic succession in both Lokoja and Bida subbasin. Three formations were recognized near Lokoja at the Niger-Benue confluence these are; the basal Lokoja sandstone, Dekina clay-shales and Lokoja carbonaceous and shally sandstone (Jones, 1951) these formations correspond to the Lokoja series of Falconer (1911). In the same Lokaja (Jones, 1951) while he was investigating on the ironstone of the area identify two formations; the oldest Lokoja sandstone overlain by the younger Patti Formation. According to Adeleye and Dessauvagie (1972) who described the general stratigraphy of the basin. The southern sub-basin otherwise known as the Lokoja sub-basin is divided in to the basal Lokoja sandstone followed successively upward by the Patti Formation and capped by the Agbaja formation all of which are laterally equivalent to their counterparts in the Northern sub-basin. Adeleye (1972) acknowledged four lithostratigraphic successions in the Northern Bida basin also known as the central Bida basin, these are: The oldest Bida Sandstone overlain by the Sakpe Ironstone followed successively upward by the Enagi Siltstone and the youngest Batati Ironstone at the top.

Jan Du chane *et al.* (1979) and Whiteman (1982) investigated on the micropaleontolgy of the Lokoja and Patti formation and documented the palynomorph-foraminiferal associations including the interpretation of the paleoenviroments. Jones (1958); Adeleye (1971, 1973, 1974) and Jan Du Chene *et al.* (1978) revealed that palynomorphs of Late Cretaceous occur in the basin. On the basis of palynofossils recovered from road side exposures north of Lokoja, a Maastrichtian age was assigned Jan Du Chene *et al.* (1978). Meanwhile, Mebradu *et al* (1986) suggested a Maastrichtian age for the same or nearby exposures, using palynofossils. Akande *et al.* (2005) work on the paleoenvironments of the sedimentary successions in the southern Bida Basin and interpreted it as ranging from continental to marginal marine and marsh environments for the Cretaceous lithofacies.

Braide, 1992a; Olaniyan and Olubaniyi, 1996; Olugbemiso and Nwajide, 1997 believed that Bida and LokoJa sandstones are continental deposits. Meanwhile, Okoson *et al.* (2007) carried out outcrop and sub-surface analysis of Bida formation based on inferred environment of deposition and recognized five different facies. (i) alluvial fan facies which dorminate in the northern part of the basin and consist of conglomerates and sandstones of debris flow and flood plains (ii) lacustrine facies characterized by siltstone, sandy mudstones, silty mudstone and mudstones (iii) fluvial facies recognized by their basal conglomerates, coarse- grained channel sandstones and fine to medium grained bar top sandstone organized in to series of fining upward succession. (iv) Floodplain facies with horizontally laminated fine-grained channel sandstones, siltstone, and mudstone and (v) mire facies characterized by peat/lignite beds.

Also the origin of the Cretaceous Oolitic Ironstones in the Bida basin has been discussed by several workers (Adeleye, 1973; Ladipo *et al.*, 1994; Abimbola, 1997; Akande *et al.*, 1998).

Recent study of the basin on the stratigraphy, paleography and hydrocarbon resource potential by (Obaje *et al.*, 2011) revealed that the potential source rocks in the basin are overwhelmingly gaseous. They also concluded base on the field studies that there is indication of petroleum system elements such as source rocks, reservoir facies and trapping mechanism exist to some limited extent.

2.3 Origin of Bida Basin

Several researches have been conducted in order to unveil the origin of the basin and this lead to contrasting models proposed regarding the genesis of the NW-SE inland Bida basin. It was described as a rift bounded tensional structure produced by faulting associated with the Benue Trough system and drifting apart of African and Brazilian Plates King (1950) and Kennedy (1990).

The genesis of Bida Basin is closely associated with the Santonian oregenic movements of south eastern Nigeria and the Benue valley making it a down warped trough. The basin is NW-SE trending embayment adjacent and approximately perpendicular to the main axis of the Benue Trough and Niger Delta Basin. It is usually regarded as the north-western extension of the Anambra Basin, both were major depocentres during the third major transgressive cycle of southern Nigeria in Upper Cretaceous time (Murat, 1972).The origin of the basin as proposed by Adeleye (1976) the basin may have originated as simple sag structure subsequent to Santonian folding in the Benue Trough; a failed arm of the tripartite division. Whiteman (1982) proposed that the origin of the basin is simple cratonic sag

based on field relationship and lack of fault scarp. Although he did not rule out the possibility of a rift origin based on the gravity and aeromagnetic data. Adeniyi (1984) suggested a rift origin (Okosun *et al.*, 2007). According to (Kogbe *et al.*, 1983) the interpretations of Landsat images, borehole logs, as well as geophysical data across the entire Mid-Niger Basin suggest that the basin is bounded by a system of linear faults trending NW–SE. Although Ojo and Ajakaiye (1989) recognized that the origin of the basin has been associated with isostatatic readjustment and gentle down warping of the basement which resulted from the removal of earth materials during the emplacement of younger granite in the Jurassic time. Ojo and Akande (2012) considered the basin to be a graben developed at an angle to the strike-slip principal fault movement along the Benue Trough.

Although Likkason (1995) suggested that Bida basin was initiated during the Campano-Maestrichtian, but related its origin to a postulated mantle plume occurring close to the confluence of the river Niger and Benue. Zaborski (1998) viewed that it is possible that the Bida Basin originated earlier than it is generally assumed, perhaps by translation of strikeslip movement along the Benue Trough into extensional stresses that favours post-Santonian origin due to apparent absence of mid-Cretaceous marine beds and the relatively undeformed nature of the sediments. However (Udensi and Osazuwa, 2004) used spectral determination of depth to buried magnetic rocks in Bida basin to estimate the thickness of the basin shows the thickness to be approximately 4.7km. Recent study carried out in some part of the basin using spectral depth analysis shows two prominent magnetic zone of shallow and deeper depth with an average depth value of 0.968km for shallow magnetic zones and 3.063km for deeper magnetic zones (Tsepav and Mallam, 2017).

2.4 Stratigraphy of Bida Basin

Many investigations have been carried out on the stratigraphy and sedimentation of Bida basin among which is the work of Russ (1930); Adeleye (1973, 1974), Ladipo (1988), Abimbola (1997), Braide (1992b), Obaje (2009) and Obaje *et al.* (2011).

The stratigraphic succession of Middle Niger embayment otherwise known as Bida basin comprises a two-fold subdivision, the Northern and Southern sub-basin collectively referred to as the Nupe group (Adeleye and Dessauvagie, 1972). Russ (1930) referred to sediments around Lokoja as the Nupe sandstone. The southern Bida basin consist of three formations the Campanian Lokoja Sandstone (made up of pebbly and clayey sandstones), the Maestrichtian Patti formation (which consist of shales, claystone and sandstone) and the Agbaja Ironstone (Jones, 1958). Adeleye and Dessauvagie (1972) identified four stratigraphic units in the northern Bida basin comprises of the basal Bida Sandstone (mainly sandstone and conglomerate) the Sakpe Ironstone formation (made up of the Pisolithic and Oolithic Ironstones), the Enagi formation (consist of siltstone, claystone, and fine grain sandstone) and the younger Batati Formation which consist of goetithic and Oolithic Ironstone. Recently (Obaje, 2009) modified the stratigraphy of the basin. He also recognized the two sub-basins the northern and southern Bida basin.

2.4.1 The Southern Bida Basin

The southern Bida basin popularly known as the Lokaja basin is made up of three different formations (Figure 3 .2) all of which are Campanian to Maestrichtian in age (Adeleye,
1971; Adeleye and Dessauvagie, 1972; Agyingi, 1991). They are the basal Lokoja Sandstone, succeeded upward by the Patti Formation and the uppermost Agbaja Ironstone.

2.4.1.1 The Lokoja Sandstone

The name Lokoja sandstone was proposed by Adeleye and Dessauvagie (1972). The sandstone unconformably overlies the Precambrian basement rock (Figure 2.3). It was referred to as the Lokoja series by (Falconer, 1911). The Lokoja formation is the lateral equivalent of Bida Sandstone and it is comprise of basal conglomerates and sandstone finely interbedded with siltstone and claystone. They are generally poorly sorted, feldspathic sandstones with interbedded siltstone and claystone.



Figure 2.3: Stratigraphic succession in the Bida Basin (After Akande, 2005; Obaje, 2009)

According to (Akande *et al.*, 2005) the basal conglomerate consists of sub-rounded to wellrounded pebbles and cobbles of quartz and feldspars embedded in a whitish clay matrix. The sandstones show fining upward sequence. They interpreted sandstones and conglomerates to have been formed in continental environment dominated by alluvial and braided stream process.

2.4.1.2 The Patti Formation

The formation directly overlies the Lokoja Sandstone and consists of fine to mediumgrained, grey and white sandstones, clays and carbonaceous silts and shales, oolitic ironstone and thin impure coal seams. The name Patti Formation was proposed by Adeleye and Dessauvagie (1972). According to Jones (1958) the maximum exposed thickness is about 70m and contain Oolitic ironstone ranges from 7-16m thick. Agyingi (1993) recorded a thickness of up to 220m for these sediments and inferred a meandering river depositional environment. It is the lateral equivalent of the Enagi Siltstone in the central Bida basin. It consists of argillaceous rocks (shale, siltstone and claystone) which are well exposed around areas between Kotonkarfe and Abaji locality (Obaje *et al.* 2011).

Mineralogically the sandstone of Patti Formation is more matured than those of the underlying Lokoja formation (Akande *et al.* 2005). Jan Du Chene *et al.* (1978) inferred fluviatile to shallow lacustrine environments of deposition for the formation. While (Braide, 1992b) interpreted the predominance of argillaceous rocks, (Siltstone, Shale, and claystone) in the Patti Formation to be a low energy environments probably restricted body of water.

2.4.1.3. The Agbaja Ironstone

This formed the uppermost part of the southern Bida basin. It exists in form of a persistent cap for the Campanian - Maastrichtian sediments in the southern Bida basin and is the lateral equivalent of the Batati Ironstone in the northern sub-basin. The name Agbaja Ironstone was proposed by Adeleye (1973). The formation comprises of sandstones and claystones interbedded with Oolitic, concretionary and massive Ironstone beds. The sandstones and claystones were interpreted as abandoned channel sands and over bank deposits that were subjected to marine reworking forming the massive concretionary and oolitic ironstones (Ladipo *et al.*, 1994). According to (Braide, 1992b; Olaniyan and Olobaniyi, 1996) minor marine influences have affected the dominantly continental environment of the upper parts of the Lokoja Sandstone and the Patti Formation and this appear to have continued throughout the period of deposition of Agbaja Ironstones.

However researchers such as (Adeleye, 1973), Umeorah (1987), Abimbola (1997), Akande *et al.* (1999), (Agunleti and Salau, 2015) and (Imrana and Haruna, 2017) have investigated the the genesis, and ore grade of Agbaja Ironstone using petrography and geochemical analysis. Adeleye (1973) suggested that the ooidal and pisolitic ironstones were similar to Minette-Type deposits, having formed within high-energy, subtidal shore line environments encroaching into fluviatile and swamp area. Base on the depositional environment and facies relationship Umeorah (1987) recognized four major facies;

- i. Goethite +Kaolinite
- ii. Hematite+Goethite+Kaolinite
- iii. Magnetite+Goethite+Kaolinite
- iv. Siderite+Magnetite+Kaolinite

Abimbola (1997) recognized three facies within the Agbaja Ironstone. These are:

 (i) At the top, a breccia mud- ironstone comprising kaolinitic matrix with angular to sub-angular detrital grains and angular clasts mostly made up of kaolinitic false ooids with faint replacement goethite rings. (ii) Pisoidal pack-ironstone consisting of poorly sorted, loosely packed pisolites made up of goethite, minor haematite and relict kaolinite with core of goethite with an outer rim of haematite. The matrix is composed kaolinite.

(iii) At the bottom, ooidal pack-ironstone comprising spherical ooids, made up of kaolinite, kaolinite goethite or haematite within kaolinite matrix.

Imrana and Haruna (2017) carried out geochemical investigation on Koton karfe Oolitic Ironstone. They concluded that it is Iron (Fe %) rich but it is of low grade when compared with the world standard.

2.4.2 The Northern Bida Basin

Four mappable stratigraphic successions are recognized in the Northern sector these are; the basal Bida Sandstone, Sakpe Ironstone, Enagi Siltstone and the uppermost Batati Ironstone (the youngest) Adeyeye and Dessauvagie (1972).

2.4.2.1 The Bida Sandstone

The formation unconformably overlies the Basement Complex and it is the lateral equivalent of the Lokoja Sandstone in the southern sector of the basin. The type section is at Doko 16 km south of Bida. It consist of approximately 80 metres of massive and flat bedded arkoses and coarse to medium sandstones with breccia horizons. But higher up in the section the sandstone are cross bedded, medium to fine grained with intercalations of shale and mudstone (Adeleye and Dessauvagie, 1972). The formation is further divided in to Doko and Jima member on the bases of lithofacies, texture and structural elements. The basal Doko Member was estimated to be at least 183 m thick and of probable braided river origin and consists mainly of very poorly sorted, pebbly arkoses, sub-arkoses and quartzose

sandstones. It is thought to have been deposited in alluvial fan setting. The Jima,upper member is estimated to be at least 90 m thick and of meandering river origin. It is dominated by cross-stratified quartzose sandstones, siltstones and claystones. It is believed that the source area of the sediments may be from the crystalline Basement complex of south-western Nigeria (Adeleye and Dessuvagie, 1972). It is believed that Bida sandstone was deposited from fluvial processes which were interpreted and inferred to be mainly meandering and braided river system (lapido *et al.*, 1994; Okosun *et al.*, 2007; Ojo and Akande, 2011). Goro *et al.* (2014) investigated the formation around Doko and identifies four lithofacies which allowed the definition of two architectural elements; the channel and overbank Architectural elements.

2.4.2.2 The Sakpe Ironstone

The Sakpe ironstone lies between Bida Sandstone and the Enagi Siltstone. The name Sakpe Ironstone was proposed by (Adeleye and Dessauvagie, 1972) The type locality is near Jima 14km SW of Bida where it is found to be up to 3metres thick of dark brown to dark yellow coarse Oolitic and Pisolitic. It consists of brown, yellow or white clay or fine sand matrix (Adeleye and Dessauvagie, 1972). Marine mollusks are found poorly preserved around Doko suggesting a Massstrichtian age. Most common structures found in the ironstone formation of Sakpe are lamination and thin to thick flat beddings (Adeleye, 1973). The Sakpe Ironstone sits on fluviatile sediments, and comprises mainly goethitic, pisolithic and Ooolitic ironstone with subsidiary Kaolinitic Oolites and Pisolites. The distribution of facies within this level, the fauna and flora, oolitic and pisolitic texture, and sedimentary structures suggest that the ironstones represent subtidal shoreline deposits laid down possibly under two freely high energy environmental conditions (Adeleye, 1973).

Olurumfemi and Waziri (2018) carried out geochemical and mineralogical characteristic of Sakpe ironstone near Jima. They concluded that the ironstone is of high grade, contain large amount of Iron (goethite and hematite) and silica and it lack deleterious materials such as Sulphur and Phosphorus.

2.4.2.3 The Enagi Siltstone

This formation directly overlies the Sakpe Ironstone and it is composed of laminated Siltstones with subordinate fine grained and Kaolinitic claystones. Adeleye and Dessauvagie (1972) proposed the name Enagi Siltstone for the formation. The pale yellow to purplish brown bedded siltstone has a thickness that varies between 10 and 79 metres and minor horizon of mudstone and sandstone that occur within the basin (Adeleye and Dessauvagie, 1972). It was inferred to be deposited in a fan, floodplain and lacustrine environment.

2.4.2.4 The Batati Ironstone

Adeleye and Dessauvagie (1972) classified the Oolitic Ironstone which consists of brown to white goethitic oolites in a yellow limonitic matrix that overlies the Enagi Formation as the "Batati Ironstone". Stratigraphically this formation is the uppermost lithological unit in the central Bida basin and its lateral equivalent in the Southern Bida basin is the Agbaja Ironstone (Figure 2.3). In most areas the thickness varies from 0.3metres up to 15 metres this may be as a result of erosion. (Adeleye, 1973) recognized two principal subfacies of the Oolitic Ironstone, these are; the coarse- grained Oolitic subfacies and the fine-grained Oolitic subfacies. Where the two sub-facies occur together the fine grain subfacies rests uncomformably on the Kutigi (coarse grain) subfacies (Adeleye, 1973). The formation is of

Maestrichtian base on a marine fauna of mollusks and worms recovered from it (Adeleye and Halstead, 1972). The Batati ironstone is dominated by goethitic, Ooltic Ironstone. Subsidiary sideritic, chloritic and kaolinitic Ironstones are present. The distribution of the Oolitic sub-facies, fauna and flora, and sedimentary structures also suggest that the deposits document subtidal shoreline sediments possibly deposited under two closely related, high energy, environmental conditions (Adeleye, 1973).

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials used

The main materials used for the study are Ironstone samples collected from the exposure at a road cut near Manigi. A total of sixteen ironstone samples were collected during the field work.



Plate I: A hand specimen of Ironstone obtained from the study area

3.2 Methods

The method employed in the study was divided in to four phases these include; Desk studies (literature review of relevant published and unpublished reports), fieldwork which involve sedimentological logging and sample collection, laboratory studies (Mineralogical and geochemical analysis) and the result interpretation.

After intensive desk study, the exploration exercise begun with geological field work during which fresh samples were obtained from the well exposed mesas where the sedimentological logging was carried out. Parts of the obtained samples were sent to the Nigeria Geological Survey Agency (NGSA) Laboratory, for the geochemical analysis using the principles of X-ray fluorescence (X-RF) techniques and mineralogical analysis using X-ray diffraction (X-RD) technique, while the Petrographic thin-sections of representative Ironstone samples were prepared and studied in the Department of Geology, Federal University of Technology, Minna. Geochemical data interpretations and writing of report were carried out using Microsoft office suite. Descriptive statistic was used to show statistical relationship between the predictor and response variables. Results of the whole rock geochemical analysis were used to evaluate the ore grade and established the viability of the Ironstone in the area.

3.2.1 Field work

A traverse was taken with the aid of compass in the extreme northern Bida basin from Kawo-Mashegu down to areas slightly before the Manigi locality where a very good exposures were found at a road cut along Kaduna-Jebba road. A well exposed section was logged in detailed and described with more emphasis and attention paid to the lithofacies and sedimentary structures. The accessibility and collection of representative rock samples were made by traversing the roads and footpaths in the area. Observation and description of the exposed outcrops was done with the view to understand the color, thickness, texture, grain size, structure, and extents of the ironstone in the area.

3.2.2 Lithological logging

The field logging of a well exposed outcrop at a road cut near Manigi at a point with coordinate location Latitude 9° 46′ 35″ N and longitude 5° 32′ 22″ E was achieved using a measuring tape for delineating the thickness of beds between the formations, Global Positioning System (GPS) for taking the coordinate of the points. Representative samples of Ironstone from six different beds were taken using geological hammer and chisel and were convey with the sample bag/sack. Field notebook book was used for recording of the field observations. Hand lens and grain size charts were use for the detailed study of the textural characteristics of the rocks. Also a digital camera was used to take photographs. The altitude of the outcrop and beds were measured using compass clinometers.

3.3 Laboratory studies

The laboratory analysis involved three different methods the mineralogical (X-RD), geochemical (X-RF) as well as the Petrographic thin-section studies in order to obtained accurate results. The main geochemical method of analyses used in the study is the X-Ray Fluorescence spectrometry. Others (X-ray diffraction techniques and petrographic thin-sections studies) are done to better confirm the minerals present in the representative ironstone samples. A total of ten representative samples of ironstones collected from different beds during lithological logging in the study area were sent to Nigeria Geological Survey Agency (NGSA) laboratory in Kaduna. Six out of ten samples undergoes X-ray fluorescence (X-RF) analysis while the remaining four undergoes X-ray diffraction (X-RD) analysis. Thin-section of six representative rock samples were also prepared and examined

under transmitted light petrograhic microscope in the Department of Geology, Federal University of Technology, Minna.

3.3.1 Petrographic Thin-section Analysis

Thin sections of six representative Ironstone samples collected from the study area were prepared and examined under the transmitted light petrological microscope in the microscope room in the Department of Geology, Federal University of Technology, Minna. The petrographic studies involved the identification of the textural materials (such as matrix, cement, determination of grain size, sorting, roundedness, nature of contact of the framework elements and preserved porosities).

3.3.1.1 Procedure for Thin-section Preparation

A rock chip was cut out from each representative samples and trimmed to a platy small size using a diamond saw machine. There after it was reduce to a fine size by rubbing it on a metal plate against different sizes of Carborundum abrasive (coarse, medium and fine sizes). The procedure lasted about 10-15 minutes. The filed sample was then washed to prevent it from mixing with the abrasive. The glass slide was smeared with an adhesive (Aradite) and the filed rock chip was mounted on it. The glass slide containing the chip sample was placed on top of a hot plate in order to achieve proper gluing of the rock sample to the glass slide. Pressure was then applied carefully to the glass slide to remove the air bubbles. It was then trimmed to reduce the thickness after which a thin transparent glass was placed on it as a cover.



Plate II: Photograph of a petrological microscope used for thin-section studies

3.3.2 X-ray Diffraction (XRD) Analysis

The powder X-ray diffraction is one of the most powerful techniques used for mineral identification. It is preferred to other methods here because of its ability to carry out whole rock mineral identification, clay speciation and identification of forms of silica.

3.3.2.1 Sample Preparation for XRD Analysis

The grinded sample (sample powders) less than 150 microns were smeared uniformly on the sample holder made of aluminum material and analyzed by a Schimadzu Model 6000 X-ray diffraction spectrometer. The analyses were carried out between 2 to 60 degrees 2θ as the sample scanning range and the scanning rate was set at 6 degree per minute.

3.3.3 X-Ray Fluorescence (XRF) Analysis

This method is chosen because it is a non-destructive analytical technique used to identify and determine the concentrations of elements (both major and trace element) present in solid, powdered and liquid samples. This is done in this study in order to confirm what we suspected in the X-ray diffraction analysis. X-RF spectrometry is capable of measuring elements from Beryllium (Be) to Uranium (U) at trace levels and up to100%. It is the most widely used analytical technique in the grit of the major and trace element chemistry of rock samples (Rollinson, 1993). It is based on the development of fluorescence as a result of the quantum energy released when an atom is de-exited after irradiation with X-rays. The X-ray photons are a type of electromagnetic radiations (10 -100 ev range of energy).

They are produced when lofty speed electrons decelerate or when electron transition occur involving inner orbit energy state of atoms. When any sample is bombarded with electron radiation (irradiation), an inner shell electron is ejected, which leaves the atom in an excited state. A de-excitation process occurs to return it to a stable ground state. The vacancy created by the emission of an inner shell electron is filled by an electron previously residing at a higher energy level.

The excess energy resulting from the transition is often dissipated as electromagnetic radiation called x-ray photon. These photons have a very slight energy band-width and are specific for particular electron transition that occurred and are characteristic of the elements from which they are emitted. The x-ray that is emitted by the sample are absorbed by the detector which act as diode in converting these incident x-rays to electronic pulses whose amplitudes are proportional to the energies of the corresponding x-rays. The pulses are processed and sorted according to the amplitude. The entire range of pulses amplitude is divided into 1024 intervals called channels and those pulses falling within each interval are counted in a multi-channel analyzer. The channel number is usually converted into units by

calibrations. The intensity or number of counts in a peak is a direct result of the number of fluorescing atoms of that element in the sample. Thus, the area under a peak is proportional to the concentration of that element in the sample.

3.3.3.1 Sample preparation for X-Ray Fluorescence (XRF) Analysis

The samples were crushed and pulverized to reduce the grains to smaller sizes to pass through 150 micron sieve. It was then parked in to a sample holder in order to provide for a random orientation of the sample materials. About 5g of the pulverized sample was weighed into a beaker with the addition of 1 g of starch binder. The meticulously mixed material was hard-pressed under high pressure (6 tones) to make a pellet. Approximately 0.4 g of sample was fused with 7.6g flux to generate a glass beads. The samples were analysed for major and trace element concentration using an Energy Dispersive X-ray fluorescence (EDXRF) spectrometer (model "Minipal 4"). Loss on ignition (LOI) was determined gravimetrically by heating 1g of the powdered sample at 1000°C.

3.4 **Result interpreta**tion

Writing of report and interpretation of results was achieved using Microsoft office word, excel, and power point. The Ironstone grade was computed by calculating the proportion of molecular weight percent of the Iron from its oxide. The average percentage of the iron oxide was then multiply by the conversion factor to give us the grade. The obtained grade was compared with the world set standard (Natural resources Canada, 2012 and Dobbins and Burnet, 1982). The results were also compared with other results of ironstones investigate elsewhere in Nigeria and around the world. Regression analysis and fitted line

plot was done to show statistical relationship between the predictor and response variables (the main elemental oxide of interest and other oxides)

CHAPTER FOUR

4.0 **RESULTS AND DISCUSION**

4.1 Field Aspect

Field observation revealed that the area around Manigi is characterized by steep slope mesas trending in a NE-SW direction. The formations present are the basal Enagi Siltstone and the uppermost Batati Ironstone which is the interval of study (plate,V). The lithologies encountered include Siltstone, mudstone, ferruginous claystone, and a subsidiary Sandstone (Figure 4.1). Presence of localized trace fossils (burrows). Observable structures include; horizontal lamination, Planer beddings and Ironstone concretions. Two different contact boundaries were also observed; the transitional boundary between ferruginous Sandstone and Ironstone and the abrupt contact between Siltstone and clay stone in the studied mesa at a road cut along Kaduna-Jebba road where representative samples of Ironstones were collected. Petrographic Thin-section studies revealed two major minerals and other accessory minerals. The frame work grains are made up of quartz supported by a matrix cement of Iron (Fe). Mineralogical analysis shows goethite and haematite as the iron bearing minerals while the whole rock geochemical analysis revealed fairly rich iron (Fe₂O₃) and high amount of gangue material (SiO₂).

4.1.1 Field Outcrop Description

Field description of outcrops was carried out by detailed traversing of the area, critically observing different formations, exposures and their lithologies. Offsets were also made from mesa to mesa in the study area in order to get good exposures of Ironstones. The observable physical properties used to describe the outcrops include color, thickness,

texture, grain size, and structures. Generally the outcrops are massive consisting of different layers and capped with the resistant Batati Ironstone formation.



Figure 4.1 Geological map of part of Akerre Sheet 162 NW Nigeria

4.1.1.1 The Enagi Formation

At the studied mesa, the formation is at the base overlain by the weathered Batati Ironstone. It comprises of intercalation of siltstone and mudstone ranging from 5-40cm thick with overall thickness of about 3 metres. Occurrence of sandstone pinch outs was also observed on the formation. The mudstone is whitish to grayish and is parallel bedded. The thickness of the Mudstone increases towards the top of the mesa. Massive clay about 1.2meters overlay the intercalation of Mudstone and Siltstone which is followed by the Occurrence of laminated clay and silt streak approximately 0.6meters. Ferruginous claystone of about 0.7meters consisting of parallel laminations was observed between the Ironstone and massive claystone Enagi Formation.



Plate III: Intercalation of siltstone, mudstone unit with parallel beddings

4.1.1.2 The Batati Ironstone

This formation constitutes the uppermost units in the studied mesa and according to Adeleye (1973) it constitutes the uppermost unit in the sedimentary sequence of the Bida

basin. The Ironstone is dark brownish and consist of spotted yellowish color. They are more weathered at the top of the mesa. It is fine grain and moderate to well sorted. The ironstone in the area can be divided in to two; Ironstone with liesegang structures interbedded with claystone followed successively upward with the weathered ironstone at the top most part of the mesa forming the capping.



Plate IV: Weathered Ironstone at top surface of the mesa

The thickness of the Ironstone formation in the logged section is about 3metres and is classified in to different beds, namely B1,B2,B3,B4,B5 B6, with thickness 0.5m, 0.7m, 1.2m, 0.6m, 0.5m and 0.7 respectively.



Plate V: Exposed part of Enagi clay stone overlain by the Batati Ironstone

4.1.1.3 Sedimentary Structures

The structures observed in the area include: planar beddings, parallel laminations, ironstone concretions, clay partings and localized burrows are well developed. In some beddings, color bands which are referred to as liesegang structures are well developed.



Plate VI: Interbedded ironstone claystone with planar beddings

The beds are generally massive, presence of thin to thick planar beddings which consist of bands of colors (Liesegang structures). Horizontal laminations are well developed. The beds show irregular lithological changes from claystone to ferruginous claystone and then ironstone. There is presence of sandstone pinch outs within the mudstones. The vertical burrows are rarely present. Ironstone concretion superimposes most of the lithologies including the siltstones and clay stone. They enclose the fresh looking whitish clay commonly found at the base of the outcrops.



Plate VII: Thin to thick parallel laminations superimposed by the Ironstone concretions

4.2 Lithological Logging

The sedimentological logging was done at a well exposed outcrop at a road cut near Manigi at a point with coordinate location Latitude 9° 46′ 35″ N and longitude 5° 32′ 22″ E in the extreme northern Bida basn. Below is the summary of the lithological section logged (Figure 4.2).



Figure.4.2: Sedimentological log of road-cut exposure near Manigi, Niger state, Nigeria $(9^{0}46'35''N \quad 5^{0}32'22'' \text{ E})$

4.3 Petrography

The petrographic thin-section analysis revealed the occurrence of two main minerals; quartz and iron (goethite) with other minor or accessory minerals suspected to be clay. Oolite and Pisolite grains were not observed. The framework grains (quartz) are colorless, absence of twinning, with no cleavage and they display floating contact. They are generally the most predominant minerals. While the cementing materials are dark brown non pleochroic with sported reddish brown color and some minor amount of yellowish materials which are suspected to be goethite Figure 4.3 A and B. The studied samples have fabric that is matrix supported (consisting of framework grains and cementing materials), well sorted and sandy in nature with quartz forming the predominant framework grains, Iron bearing minerals forming the cementing materials Figure 4.3. The cementing material filled the interstitial spaces between the frame work grains. This implies that the irons are early formed (eodiagenetic) before the compaction of the sediments. The predominant amount of quartz implies that the ironstone contain high amount of impurities which may affects the quality and grade of the iron.



Figure.4.3: Photomicrographs of Ironstone under plane and cross polarized light: the ferruginized fine grain and non oolithic nature of the ironstone

4.4 Mineralogy

The results of XRD analysis are presented in the Table 4.1 with quartz being the most predominant mineral identified. Other minerals include hematite, goethite and rutile. The representative diffraction patterns are presented at Figure 4.5 and the pie chart showing the mineral abundance Figure 4.4.The results of X-ray diffraction analysis identified two minerals in sample B2 namely quartz and goethite. Quartz is the dominant mineral within the sample, accounting for up to 85%, and goethite 15% (Table.4.1). The petrographic study of this sample confirms the predominance of quartz over iron minerals (Figure 4.3 G and H).

Sample B1 contains 61% quartz and 39% goethite and when compared with the petrographic studies (figure 4.4 C&D) shows that quartz are still the dominant minerals held within reddish brown cement. The platy nature of the grains of iron in the thin-section is a characteristic of goethite (Mohapatra, 2008).Sample B5 consist of hematite and quarts as the essential minerals with hematite accounting for about 54% and quartz 46% (Table 4.1). Petrographic studies of this sample truly confirm the results of the XRD analysis. The grains are matrix supported and with quartz forming the frame work grains and iron material yellow to grayish in color filling the interstitial spaces between the framework grains. Sample B6 comprise of rutile and goethite as the dominant minerals. Although petrographic study of this sample indicates the presence of quartz as the framework grains. (Figure 4.3 A and B). The controversial occurrence of amount of rutile in this sample may be as a result of defect in sample preparation. The iron minerals are dark brown in color and consist of spotted reddish brown within. The shape of the grains is platy in nature, which are said to be the characteristic of goethite and hematite (Mohapatra, 2008)

Minerals	SB1	SB2	SB5	SB6
Goethite	39	15	54	33
Hematite	0	0	0	0
Rutile	0	0	0	67
Quartz	61	85	46	0
Total	100	100	100	100
Rutile Quartz Total	0 61 100	0 85 100	0 46 100	67 0 100

Table 4.1: Mineral abundance in Ironstone around Manigi



Figure 4.4: Mineral abundance in Ironstone around Manigi



Figure 4.5(a): Powder diffraction pattern for sample B1



Figure 4.5(b): Powder diffraction pattern for sample B2



Figure 4.5(c): Powder diffraction pattern for sample B5



Figure 4.5(d): Powder diffraction pattern for sample B6

The result of X-ray fluorescence analysis of Ironstone exposed around Manigi locality is shown in the Table 4.2. The concentration of Fe₂O₃ ranges from 28.73-85.60wt% with an average of 52.65wt% and SiO₂ ranges from 1.32-43.90wt% with an average of 33.50wt%. Al₂O₃ and TiO₂ ranges from 3.36-6.83wt% and 0.48-1.65wt% with averages 4.79wt% and 0.95wt% respectively. Certain impurities such as BaO₂, CuO, Cr₂O₃, MnO, CaO, Cl, K₂O, ZnO, Br, MgO, Rb₂O, ZrO₂, CdO, TaO₅, PbO, and HfO₂ exist in the iron ore in a considerable amount. The loss-on-ignition ranges from 4.20 - 9.40wt% with an average of 5.933wt%. However SO₃ and P₂O₅ are not found in any of the samples or they are below detection limits.

Comparison of the average chemical composition of the analyzed samples of the ironstone around Manigi with other Nigerian ironstone and the other ironstones around the world shows that Batati Ironstone around Manigi has less iron (Fe₂O₃) and alumina (Al₂O₃) content. However it has higher silica (SiO₂) and Titanium (TiO₂) content and contains more impurities when compare with other ironstones found elsewhere in Nigeria (Table 4.3). However when compare with that of Pedrada in Brazil and Reboredo in Moncorva, their iron oxide, silica and Alumina are within the same level and they all lack sulfur oxide (SO₃) in other words they are similar. Although Pedrada and Reboredo iron ore contain Phosphorus oxide (P₂O₅), it was not detected in this study, Table 4.4.

The distribution pattern of the analyzed samples and the average abundance of major oxides are presented in Figures 4.5 and 4.6. It is observed that concentration of iron oxide is

generally with the highest peak followed by the concentration of silica. This is also confirmed by the petrography and X-ray diffraction analysis.



Figure 4.6: Distribution pattern of geochemical elements in Ironstone around Manigi



Figure 4.7: Average abundance of major oxides of the Ironstones at Manigi

Oxide coposition %	SB1	SB2	SB3	SB4	SB5	SB6	Average
SiO ₂	29.1	56	43.9	29.9	40.8	40.8	33.5
TiO ₂	0.694	1.62	1.65	0.53	0.48	0.48	0.95
Al ₂ O ₃	6.83	4.99	3.94	4.11	3.36	3.36	4.79
Fe ₂ O ₃	56.81	28.73	37.45	59.09	48.22	48.22	52.65
Cl	0.74	0.001	1.03	0.81	0.77	0.77	0.742
CaO	0.02	0.055	0.001	0.001	0.02	0.02	0.046
MgO	0.008	0.021	0.001	0.001	0.01	0.01	0.025
Na ₂ O	0.017	0.27	0.001	0.001	0.014	0.014	0.098
K ₂ O	0.011	0.12	0.001	0.001	0.011	0.011	0.035
MnO	0.12	0.001	0.001	0.001	0.001	0.001	0.021
V_2O_5	0.001	0.001	0.001	0.001	0.001	0.001	0.001
Cr ₂ O ₃	0.01	0.041	0.085	0.041	0.037	0.037	0.042
CuO	0.032	0.038	0.062	0.038	0.028	0.028	0.044
ZnO	0.001	0.001	0.001	0.001	0.001	0.001	0.003
Br	0.001	0.001	0.016	0.001	0.001	0.001	0.002
Rb ₂ O	0.007	0.01	0.001	0.004	0.008	0.008	0.004
ZrO_2	0.11	0.21	0.3	0.13	0.16	0.16	0.0195
CdO	0.88	0.001	1.42	0.001	0.001	0.001	0.383
BaO	0.16	0.65	0.75	0.36	0.25	0.25	0.442
Ta_2O_5	0.001	0.067	0.001	0.001	0.067	0.06	0.032
PbO	0.001	0.18	0.001	0.001	0.001	0.001	0.03
HfO ₂	0.05	0.001	0.001	0.086	0.066	0.001	0.033
LOI	4.4	7	9.4	4.9	5.7	4.2	5.933

 Table 4.2: Chemical composition of analyzed Ironstone around Manigi

4.5.1 Grade Determination

In determining the grade, the Iron oxide (Fe_2O_3) was converted to its elemental state of iron total (tFe). After which the conversion factor was multiply by the average weight percent of iron oxide to get the grade.

From the result of geochemical analysis in Table 2.4 above, Ironstone around Manigi has Fe_2O_3 ranges from 28.73 - 85.60wt% with an average of 52.65wt% .The conversion is explain below.

Atomic weight of Iron (Fe) = 55.845g

Atomic weight of Oxygen (O) = 15.999g

Therefore the molecular weight of $Fe_2O_3 = 2(55.847) + 3(15.999) = 159.69$ g/mol For the Iron (Fe total) proportion = $Fe_2/Fe_2O_3 = 2(55.847/159.69)$

$$=$$
 111.694/159.69 $=$ 0.69**9**4

Therefore to get the grade, the average wt% of Fe_2O_3 (52.65wt %)is multiply by the conversion factor above. That is 52.65x 0.6994 = 36.823% (Grade).

4.5.2 Quality of ironstone around Manigi

In order to assess the quality of Ironstone at Manigi the geochemical results of the ironstone was compared with the composition of the extracted and analyzed ore of other ironstones in Nigeria and ironstone from other nations around the world. Among the biggest iron ore producing countries in the world include Brazil, China, Australia, and Russia.

Elenent	Drasant	Abimbola 1007	Agunlati & Salau 2015	Imrono & Horuno 2017	Olurunfemi&Waziri,
(Oxide	riesein	Additiona,1997	Aguineti&Salau,2015	IIII ana& Haruna,2017	22018
s %)	Study	(AgbajaFormation)	(Oolitic&pisolitic)	(Kotonkarfe Oolitic)	(Sakpeformation)
SiO ₂	33.5	11.05	3.41	16.98	28.938
Al_2O_3	4.79	10.82	8.98	12.49	12.906
SO_3	-	-	-	0.15	0.02
P_2O_5	-	3.09	2.23	0.98	0.008
Na ₂ O	0.098	0.2	-	-	0.00029
K_2O	0.035	1.0	-	-	0.0027
MgO	0.098	0.2	-	0.2	0.071
CaO	0.046	0,24	0.54	0.178	0.0176
TiO_2	0.95		0.4	0.279	0.1839
MnO	0.021	0.24	0.57	0.125	-
Fe ₂ O ₃	52.65	64.06	83.42	67.81	58.859
V_2O_5	0.001	-	0.12	-	-
BaO	0.442	-	-	-	-
Cr_2O_3	0.042	-	-	-	-
CuO	0.044	-	-	-	-
H_2O^+	-	-	-	-	-

 Table 4.3: Comparison of the average chemical composition of the analyzed Ironstone around Manigi with other Nigeria Ironstones

Iron content, impurities, and deleterious materials such as sulfur, phosphorus are the most important features to be considered in the determination of the quality of the iron ore body (Marden, 1982). Dobbins and Burnet (1982) has submitted that natural iron ore should be low in silica (<6%), Alumina (3-4%), Phosphorus (0.05-0.07%) and sulfur (0.1%) to be acceptable iron ore (Table 4.5).

Element	Present	Pedrada Brazil	Reboredo, Moncorva
(Oxides %)	Study	(Rebelo, 1985)	(Orey, 1980)
SiO ₂	33.5	36.9	36.7
Al ₂ O ₃	4.79	6	7.3
SO ₃	-	-	-
P ₂ O ₅	-	1.7	2.4
Na ₂ O	0.098	0.2	0.2
K ₂ O	0.035	1	1.1
MgO	0.098	0.2	0.2
CaO	0.046	0.1	0.4
TiO_2	0.95	0.3	0.4
MnO	0.021	0.1	0.1
Fe ₂ O ₃	52.65	51.9	48.2
Cr ₂ O ₃	0.042	-	-
CuO	0.044	-	-
H_2O^+	5.933	0.7	1.6

 Table 4.4: Comparison of the average chemical composition of the analyzed Ironstone around Manigi with the other Ironstones around the world

For the classification and evaluation of quality and grade of ironstone, Dobbins and Burnet (1982) divided the raw iron ores in to three basic classes depending on the total iron content: (i) high grade iron ores with a total iron content above 65%, (ii) Medium grade ores with iron content in the range of 62-64% and (iii) low grade ore with iron contents below 58% (Table 5.4). Although according to the Natural Resources Canada (2012) an ore that has > 54 weight percent of iron is a high grade ore.

Components	Total Fe			SiO ₂	Al_2O_3	Р	S
Contents (mass %)	Low	Medium	High	<6%	3-4%	0.05 -0.07%	0.1%
	<58	62-64%	>65%	-			

 Table 4.5: Generalized percentages of elements of major interest in assessing iron ore quality

(Dobbins and Burnet, 1982)

Therefore Ironstone around Manigi is rich in iron ranging from 28.73Wt% to 85.60Wt% with an average of 52.65% and from the calculation of the grade above 36.823% show that it is a low grade using the generalized percentages of elements of major interest by (Dobbins and Burnet, 1982) and Natural Resources Canada (2012) in assessing its iron ore quality. It silica (SiO₂) content ranges from 1.32% to 56.00% with an average of 33.50% is higher than the generalized percentage of < 6% (Dobbins and Burnet, 1982). Alumina (Al₂O₃) in this study ranges from 3.36% to 6.83% with an average of 4.79% this is in considerable level and can be manageable using a generalized percentage of 3-4% by (Dobbins and Burnet, 1982). Phosphorus and Sulfur were found to be absent in the ironstone. This shows that the ironstone has less contaminant.

4.6 **Result Interpretation**

From the results above (Table 4.2) in page 57, Ironstone around Manigi has high silica (33.50%) content which exceeds the generalized percentage by (Dobbins and Burnet, 1982). Base on that it cannot serve well as a good raw material for steel production but rather it will be good for cast iron production since silica is not a problem in the cast iron
production. For this ironstone to be useful in the steel production, it will require proper beneficiation. Aluminium oxide increases the viscosity of the slag (Terkel, 1983). However in this study it is slightly above the permissible level of 3.4%. Hence it can be manageable when properly beneficiated. Phosphorus makes steel brittle, even at concentration of as small as 0.6% (Gordon, 1996). However Batati Ironstone is free from phosphorus or it is below the detection limits. Similarly sulfur which causes cracks and in turn causes iron to fail is also found to be absent in this study. From the above findings we can say that the Batati Ironstone around Manigi can be acceptable metallurgically for high quality steel production.

4.7 Environment of Deposition of the Ironstones

The geochemical composition of the analyzed samples was used to infer the environment of deposition of the ironstone. Results from the chemical analysis revealed that the concentration of Magnesium oxide (MgO) ranges from 0.001wt% to 0.11wt% with an average of 0.025wt%. The average concentration of CaO, Na₂O, K₂O, MnO, V₂O₅, and CuO are 0.046, 0.098, 0.035, 0.021, 0.001and 0.044 respectively. It can be deduce from the low concentration of magnesium in all the analyzed samples that the environment of deposition of the ironstone is non marine or shallow marine environment. Absent of sulfur oxide (SO₃) in all the analyzed samples also support this claim. The reason is that marine ironstone is usually sulfur and magnesium rich. Similarly low concentration of CaO and absent of CO₃ suggest that the environment is an oxidizing environment.

4.8 **Regression Analysis and Fitted Line Plot**

The regression analysis and fitted line plot are use to investigate the statistical relationship between two or more predictors and the response variables. In this study the main elemental oxide of interest, that is Fe_2O_3 and other oxides that are present in the ore (such as SiO_2 , Al_2O_3 , CaO, MgO, Na₂O, MnO, and TiO₂.

Results of regression analysis and fitted line plots are presented in Figure 4.8. It was observed that SiO₂, TiO₂ and K₂O have an inverse relationship with Fe₂O₃ (negative regression) with coefficient of determination (\mathbb{R}^2) 0.981, 0.444 and 0.014 respectively (Figures 8.4 a, b and c). While Al₂O₃, CaO, and MgO has a direct relationship with Fe₂O₃ (positive regression) with coefficient of determination (R^2) 0.114, 0.459 and 0.549 respectively (Figures 4.8 d, e and f). The sign of each coefficient indicate the direction of the relationship. The closer the value of R^2 is to one, the more the closeness of the points to the fitted line. Hence the more the two variables are related. According to (Young and Tailor, 1989) it is usually controversial and difficult to determine the origin of ironstone geochemically, because of complexities in the formation of Oolites and processes of concentration of iron in the supergene environment. Although (Guilford and Mackenzie, 1980) are of the opinion that the positioning of various elements given by Goldschmidth classification (lithophile, siderophle, chalcophile and atmophile) in the periodic table can form the basis for the relationship between the elements and iron. Silicon which is a non metallic lithophile element has an inverse relationship with a metallic siderophile Iron (Figure 4.8a). This may be as a result of the electron affinity between the two elements. Calcium an alkaline earth metal and a lithophile element has a direct relationship with siderophile Iron (Figure 4.8f) although the coefficient of determination is very low.

Aluminum a metalloid lithophile also has direct relationship with siderophile iron (Figure 4.7d) even though the points are not close to the fitted line. Also magnesium a lithophile element has a direct relationship with iron (Figure 4.8e). Titanium a lithophile element has an inverse relationship with siderophile iron (Figure 4.7b). They exist together because they both have similar characteristics of transition elements. Finally Potasium non metal and a lithophile element have a direct relationship with a very low coefficient of determination (Figure 4.8c).



Figure 4.8(a): Regression Analysis with fitted line: SiO₂ versus Fe₂O₃



Figure 4.8(b): Regression Analysis with fitted line: TiO₂ versus Fe₂O₃



Figure 4.8(c): Regression Analysis with fitted line: K₂O versus Fe₂O₃



Figure 4.8(d): Regression Analysis with fitted line: Al₂O₃ versus Fe₂O₃



Figure 4.8(e): Regression Analysis with fitted line: MgO versus Fe₂O₃



Figure 4.8(f): Regression Analysis with fitted line: CaO versus Fe₂O₃

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The results of this study shows that the Ironstone exposed around Manigi locality in the extreme northern Bida basin is fairly rich in iron oxide in the form of goethite and some appreciable amount of hematite which ranges from 28.73 – 85.60wt% with an average of 52.65w%. Calculation of the grade of the iron revealed approximately 36.823% which has been described as a low grade. The study has also shown that the ironstone contain high amount of gangue materials (SiO₂). Its Alumina (Al₂O₃) is within the permissible level and it lacks the deleterious materials such as sulfur and phosphorus. It was deduced that the environment of deposition of the sediments is non-marine or shallow marine on the bases of low magnesium (MgO) concentration in all of the analyzed samples. Also absence of sulfur and phosphorus supported this claim, since marine sediment are mostly Sulfur rich.

Field outcrop studies shows that the contact between Enagi Formation and the Batati Ironstone is transitional. From the petrographic studies, the floating contact displayed by the framework grains suggests that the iron cements were eodiagenetic in origin. The absence of oolitic and pisolitic grains in the studied interval suggests that the lower parts of the Batati Formation are ferruginized sandstones.

The result of XRD analysis revealed the occurrence of quartz as the most dominant mineral. It appears in almost all the sample which was also confirmed by the petrographic analysis. Sample B2 contains 85% quartz followed by 61% in sample B6 and then 46% in sample B5. The predominant iron bearing mineral is goethite identified in sample B1 39%

followed by 33% in sample B6 and then 15% in sample B2. Hematite was identified in sample B5 with 54%. The result from both the petrographic and X-ray diffraction analysis shows and confirmed the occurrence of goethite and hematite as the predominant iron bearing minerals.

It can be concluded from the results obtained from this study that Ironstone exposed around Manigi area in the extreme Northern Bida basin is of low grade. It can be more useful as cast iron, although adequate beneficiation (to remove excess silica) and transformation of goethite to hematite can make it useful in the production of iron and steel.

5.2 **Recommendations**

The Ironstone shoud be adequately beneneficiated before use, or it should be averagely use as cast iron.

Further studies to reveal the tonnage, extent and reserve estimation of the Batati Ironstone around Manigi should be undertaken for the purpose of commercial exploitation to feed the Nigerian steel industries.

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X-raydiffraction analysis result for sample B1

<u>Peak List</u>

Pos.[°2Th.]	Height [cts]	FWHMLeft[°2Th.]	d-spacing [Å]	Rel. Int. [%]
21.0491	1091.82	0.1023	4.22068	20.60
21.3877	216.10	0.2047	4.15462	4.08
26.8185	5300.64	0.1279	3.32436	100.00
33.4567	170.24	0.2047	2.67841	3.21
36.7106	582.90	0.1023	2.44813	11.00
39.6231	487.98	0.0768	2.27464	9.21
40.4447	262.03	0.0768	2.23031	4.94
42.6040	383.44	0.0768	2.12213	7.23
45.9433	197.12	0.0768	1.97537	3.72
50.2769	786.96	0.0768	1.81479	14.85
53.4293	131.18	0.3070	1.71492	2.47
55.0190	437.19	0.0768	1.66907	8.25
55.4933	170.57	0.1535	1.65593	3.22
60.0917	590.22	0.0768	1.53974	11.13
64.1676	314.94	0.1535	1.45142	5.94
67.8726	550.90	0.1023	1.38094	10.39
68.2766	518.30	0.0936	1.37261	9.78
68.4575	669.58	0.0936	1.36942	12.63
73.6443	84.29	0.3070	1.28632	1.59

Visib	le Ref.Code	Score	Compound Name	Displ.[°2Th]	Scale
Fac.	Chem. Formula				
*	96-101-1177	33	Quartz low	0.000	
0.161	Si6.00 06.00				
*	96-900-2159	21	Goethite	0.000	
0.064	Fe4.00 H4.00 O8	.00			

X-ray diffraction analysis result for sample B2

<u>Peak List</u>

Pos.[°2Th.]	Height [cts]	FWHMLeft[°2Th.]	d-spacing [Å]	Rel. Int. [%]
12.5068	46.81	0.3070	7.07763	0.87
21.0377	1008.09	0.1023	4.22294	18.66
25.1242	37.65	0.6140	3.54457	0.70
26.8125	5402.63	0.1279	3.32510	100.00
36.6969	385.25	0.1023	2.44901	7.13
39.6317	598.81	0.1023	2.27416	11.08
40.4485	285.49	0.0768	2.23010	5.28
42.6025	469.41	0.0768	2.12221	8.69
45.9520	269.22	0.0768	1.97501	4.98
50.2743	1064.51	0.1023	1.81488	19.70
53.6115	49.89	0.6140	1.70952	0.92
55.0168	405.21	0.0768	1.66913	7.50
60.0863	838.94	0.0936	1.53859	15.53
60.2578	509.05	0.0936	1.53843	9.42
64.1580	138.40	0.3120	1.45042	2.56
67.8860	627.35	0.1248	1.37955	11.61
68.2675	834.28	0.0936	1.37277	15.44
68.4632	727.48	0.0936	1.36932	13.47
73.5844	222.59	0.0936	1.28616	4.12

Visib	le Ref.Code	Score	Compound Name	Displ.[°2Th]	Scale
Fac.	Chem. Formula				
*	96-500-0036	35	Quartz	0.000	
0.113	Si3.00 06.00				
*	96-900-2160	6	Goethite	0.000	
0.031	Fe4.00 H4.00 O8	.00			

X-ray diffraction analysis result for sample B5

<u>Peak List</u>

Pos.[°2Th.]	Height [cts]	FWHMLeft[°2Th.]	d-spacing [Å]	Rel. Int. [%]
20.9106	148.75	0.1535	4.24834	8.31
24.1830	69.72	0.6140	3.68035	3.89
26.6935	1790.35	0.1023	3.33966	100.00
33.2439	325.89	0.2047	2.69506	18.20
35.6913	334.83	0.2558	2.51567	18.70
36.6054	170.24	0.1535	2.45492	9.51
39.5276	137.78	0.1535	2.27991	7.70
40.8506	88.22	0.8187	2.20908	4.93
49.5752	148.03	0.3070	1.83882	8.27
50.1738	416.18	0.0768	1.81828	23.25
54.1557	264.35	0.2047	1.69362	14.77
59.9912	334.34	0.0768	1.54208	18.67
62.6112	198.71	0.4093	1.48371	11.10
64.1341	211.83	0.4093	1.45210	11.83
68.3601	274.50	0.0936	1.37114	15.33

Visibl	le Ref.Code	Score	Compound Name	Displ.[°2Th]	Scale
Fac.	Chem. Formula				
*	96-900-9783	64	Hematite	0.000	
0.197	Fe12.00 018.00				
*	96-901-2601	55	Quartz	0.000	
0.360	Si3.00 06.00				

X-ray fluorescence analysis result for sample B6

<u>Peak List</u>

Pos.[°2Th.]	Height [cts]	FWHMLeft[°2Th.]	d-spacing [Å]	Rel. Int. [%]
27.4660	334.50	0.1535	3.24745	100.00
33.8108	267.37	0.2047	2.65116	79.93
36.3773	238.67	0.2558	2.46979	71.35
37.2450	105.91	0.3070	2.41422	31.66
40.2161	83.82	0.4093	2.24246	25.06
41.5572	116.31	0.3070	2.17314	34.77
43.1718	79.89	0.3582	2.09553	23.88
46.4934	66.07	0.6140	1.95327	19.75
50.1677	175.36	0.2558	1.81849	52.43
50.9461	215.44	0.2047	1.79252	64.41
54.6939	269.63	0.3070	1.67822	80.61
58.1300	66.33	0.3070	1.58694	19.83
60.6238	140.85	0.3070	1.52749	42.11
63.1893	177.18	0.4093	1.47152	52.97
64.8378	206.36	0.3582	1.43803	61.69
68.9021	254.34	0.2047	1.36280	76.04

Visibl	e Ref.Code	Score	Compound Name	Displ.[°2Th]	Scale
Fac.	Chem. Formula				
*	96-900-4142	16	Rutile	0.000	
0.857	Ti2.00 04.00				
*	96-900-3080	8	Goethite	0.000	
0.745	Fe4.00 08.00				