ASSESSMENT OF IRON ORE MINING GANGUES IN ITAKPE FOR
SECONDARY RECOVERY OF OTHER METAL VALUES

By

WEHLEEKEMA SIAPLAY, BSc.

A Thesis Submitted to the School of Graduate Studies
African University of Science and Technology, Abuja, Nigeria
in Fulfillment of the Requirements for the Degree of

MASTER OF SCIENCE IN MATERIALS SCIENCE AND ENGINEERING

Department of Materials Science and Engineering

© January 2017
DECLARATION

I hereby declare that except for reference to other people’s work, which I have dully cited, this thesis submitted to the School of Graduate Studies, African University of Science and Technology, Abuja, Nigeria is the result of my own investigation, and has not been presented for any other degree elsewhere.
ASSESSMENT OF IRON ORE MINING GANGUES IN ITAKPE FOR SECONDARY RECOVERY OF OTHER METAL VALUES

By

WEHLEEKEMA SIAPLAY, BSc.

RECOMMENDED:  ----------------------------------------------

Supervisor, Prof Peter A. Onwualu

Dr Adelane R. Adetunji

APPROVED:  ----------------------------------------------

Head, Department of Material Science and Engineering

Prof Peter A. Onwualu

Chief Academic Officer, Prof Charles Chidume

Date
ABSTRACT

Due to rapid industrialization, the demand for heavy metals is ever increasing, but the reserves of high-grade ores are diminishing. Therefore there is a need to explore alternative sources of heavy metals. Ore mine gangues which are industrial wastes can act as potential sources of industrial materials. Typical iron ore minerals may contain antimony, arsenic, beryllium, cadmium, chromium, copper, lead, nickel, as well as silver and zinc [6]. The economic viability of these metals would depend on their various amounts in the iron ore. The analysis of the Itakpe iron ore in Nigeria would determine whether the associated metals are commercially feasible for secondary recovery. Due to these afore mentioned problems, the research was carried out using EDX - XRF and XRD, analyzing five different samples which include coarse iron ore, middle fine iron ore, fine iron ore, gangue iron ore and concentrate iron ore. The sample analysis shows that Itakpe iron ore is hematite-rich with atomic % of Fe to be 40.00 and weight % to be 69.94.

The EDX results showed the presence of iron peak of varying intensity which was observed to have the concentrate with the highest peak of 37500 and the gangue with the least peak of 11200. Also, the presence of silicon peak of varying intensity was observed with the gangue having the highest intensity of 7000 while the concentrated iron ore and coarse iron ore has a negligible intensity. The varying intensity of cobalt in the 5 sample cannot be neglected as the concentrate iron ore has the highest intensity of 5000 and the gangue has the lowest intensity of 1500.

The XRF was used for the elemental analyses which showed high percentage of iron element ranging from 34.60% for GIO, 51.59% for MfIO, 53.80% for FIO, 56.34% for CIO, and 64.51% for CoIO. Also, the samples contain some percentage of Silica ranging from 0.36% for CIO, 1.63% for CoIO,
11.60% for FIO, 12.53% for M\textsubscript{4}IO and 22.97% for GIO. This really shows that the iron and the silicon can still be recovered from the gangue.

Also, the XRD was used for the structural analysis of the unprocessed iron ore, middle fine iron ore, concentrate iron ore and the gangue iron ore samples in which the spectral of the unprocessed iron ore was observed to have many peaks when compared with the spectrum of other samples, this is attributed to high concentration of impurities.

**Keywords:** Heavy metals, EDX-XRF, XRD, Hematite, gangue, CIO, M\textsubscript{4}IO, FIO, CoIO
DEDICATION

This work is dedicated to my father Lawrence W. Siaplay Sr. You are the reason why this work must be and was done.
ACKNOWLEDGEMENT

I wish to acknowledge God Almighty for blessing and protection on my life in carrying through the course of study and successful completion of this research work. I am pleased to acknowledge the following persons for their contributions towards the successful completion of this research work.

1. Dr. Adelana R. Adetunji, Lecturer at the Department of Materials Science and Engineering, African University of Science and Technology, Abuja, Nigeria for his good supervisory skills, dedication, encouragement and timely comments. During the period of this thesis he provided input, guidance and feedback of immense value. He has also been exemplary, as an academic role model to me, upholding the true spirit of science and academic research. He has also assisted me in becoming knowledgeable as it relates to the relevant engineering background. Also to my HOD, Prof. Peter Azikiwe Onwualu, really appreciate your effort towards my academic success.

2. Also appreciate the effort of the Liberia Government through the Interministerial Scholarship Committee for the scholarship provided. I would like to appreciate the Liberian Ambassador to Nigeria Prof. Dr. Alhassan Conteh for his mentorship and fatherly role during my study.

3. Both academic and Non- academic staff of the Material Science and Engineering Department, AUST and Mrs. Theresa Ezenwafor PhD student in African University of Science and Technology, Abuja, Nigeria for her comments, reviews and suggestions.

3. My sincere gratitude and appreciation goes to The Pan African Material Institute (PAMI) for the financial assistance rendered in carrying out this research work. Also to the authority of the Itakpe Iron Ore Mining Company for granting me access to their site and allowing me to collect the samples.
My brothers Prince Dennis, Arthur Collins, Lawrence W. Siaplay Jr, Leekpealay Siaplay, Alakamenlee Siaplay, Gwehfehkpeh Siaplay and my Sister Diapuma Siaplay also my mother Oretha G. Siaplay my Kids Mamenlee Siaplay and Noomennee Siaplay for their supports and prayers during my research.

**TABLE OF CONTENTS**

<table>
<thead>
<tr>
<th></th>
<th>PAGES</th>
</tr>
</thead>
<tbody>
<tr>
<td>TITLE PAGE</td>
<td>i</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>ii</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>DEDICATION</td>
<td>v</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENT</td>
<td>vi</td>
</tr>
<tr>
<td>TABLE OF CONTENTS</td>
<td>vii</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>x</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>xi</td>
</tr>
<tr>
<td>LIST OF ABBREVIATIONS</td>
<td>xii</td>
</tr>
</tbody>
</table>

**CHAPTER ONE**

**1.0 INTRODUCTION**

1.1. Overview

1.2. Problem statement

1.3. Objective of study

**CHAPTER TWO**

**2.0 LITERATURE REVIEW**

2.1 Formation of iron ore deposit

2.1.1. Processes of iron ore formation
## LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1</td>
<td>Properties of Major Iron Minerals</td>
<td>8</td>
</tr>
<tr>
<td>2.2</td>
<td>Iron Ore Production</td>
<td>11</td>
</tr>
<tr>
<td>2.3</td>
<td>Nigeria Iron Ore Reserves (%) and Extent of Development</td>
<td>16</td>
</tr>
<tr>
<td>2.4</td>
<td>Nigeria Iron Ore Deposits under Investigation</td>
<td>17</td>
</tr>
<tr>
<td>4.1</td>
<td>The XRF Results of Elemental Composition of CIO, FIO, MfIO, GfO, and CoIO</td>
<td>33</td>
</tr>
</tbody>
</table>
LIST OF FIGURES

Figure 2.1: The world top 20 steel consuming countries 2013 in Million tones 12

Figure 3.1: Location of the Study Area, Access roads and Major Geological Features 26

Figure 3.2: Map of Nigeria Showing the Location of NIOMCO Concession Area 27

Figure 3.3: Geological Map of Itakpe Iron Ore Deposit 29

Figure 4.1: EDX of CIO 35
Figure 4.2: EDX of FIO 35
Figure 4.3: EDX of M₄IO 36
Figure 4.4: EDX of GIO 36
Figure 4.5: EDX of CoIO 36
Figure 4.6: XRD of UPIO 38
Figure 4.7: XRD of M₄IO 38
Figure 4.8: XRD of CoIO 39
Figure 4.9: XRD of GIO 39
LIST OF ABBREVIATION

EDX.................. Energy Disperse X-ray
XRF.................. X-ray Fluorescence
XRD.................. X-ray Diffraction
SAG.................... Semi-Autogenous Grinding
USEPA................. United State Environmental Protection Agency
BIF..................... Bounded Iron Formation
USGS................. United State Geological Survey
AMSL.................. Above Mean Sea Level
ISO...................... International Standard Organization
GDP.................... Gross Domestic Product
NASENI................. National Agency for Science and Engineering Infrastructure
ICDD................... International Centre for Diffraction Data
MfIO.................... Middle fine Iron Ore
CIO...................... Coarse Iron Ore
FIO...................... Fine Iron Ore
GIO...................... Gangue of Iron Ore
CoIO..................... Concentration of Iron Ore
UPIO.................... Unprocessed Iron Ore
CHAPTER ONE

1.0 INTRODUCTION

1.1 Overview

Iron constitutes about 5% of the Earth’s crust, making it the fourth most abundant element. Iron oxides and hydroxides form the principal iron ore minerals, due to their high iron content and occurrence as large tonnage surface deposits [1]. The presence of the amount of iron varies from an average of 2-3% in sedimentary rocks to 8.5% in the basalt and gabbro [5]. Iron is, however, 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. Typically, a deposit must contain at least 25% iron to be considered economically recoverable. Exploitation of large, low grade iron ore deposits can be achieved through concentration of the iron.

The iron itself is usually found in the minerals of magnetite (Fe$_3$O$_4$), hematite (Fe$_2$O$_3$), goethite (FeO(OH)) or limonite (FeO(OH)$_n$(H$_2$O)), siderite (FeCO$_3$), chamosite (Mg,Fe,Al)$_6$(Si,Al)$_{41/4}$(OH)$_8$, ilmenite (FeTiO$_3$) and pyrite (FeS). Despite the existence of iron in many minerals, five of them are the primary sources of iron: magnetite (Fe$_3$O$_4$), hematite (Fe$_2$O$_3$), goethite/limonite (FeO(OH)), siderite (FeCO$_3$), and pyrite (FeS) [5].

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 characteristic that will allow it to be a source of iron either immediately or potentially [2]. Iron ore is the raw material used to make pig iron, which is one of the main raw materials to make steel. Nearly 98% of the mined iron ore is used to make steel [3]. There are four main types of iron ore deposits worked currently, depending on the mineralogy and geology of the ore deposits. These are magnetite, titanomagnetite, massive hematite, and pisolitic ironstone deposits [2].
The Itakpe iron ore deposit in Nigeria which has a total estimated reserve of about 182.5 million metric tonnes consists mainly of quartzite with magnetite and hematite [6].

The deposit has been developed to supply iron ore concentrates to Ajaokuta steel plant and the Delta steel plant, Aladja, in Nigeria.

The Itakpe Iron deposit is important for the successful development of iron and steel industry in Nigeria though on the national scale there are other silico-ferruginous formations which are interesting from commercial and economic viewpoint especially the deposits of Ajabonoko Hill and Choko-Choko which are said to be similar to those at Itakpe. To be taken seriously also is the revenue loss due to the uncovered minerals in Itakpe iron ore gangue.

The extraction of specific value minerals from their naturally occurring ores is variously termed “ore dressing,” “mineral dressing,” and “mineral beneficiation.” For most metalliferous ores produced by mining operations, this extraction process is an important intermediate step in the transformation of natural ore to pure metal. Although a few mined ores contain sufficient metal concentrations to require no beneficiation (e.g. some iron ores), most contain relatively small amount of the valuable metal, from perhaps a few percent in the case of base metals which are commonly intergrown with economically important and unimportant (gangue) minerals on a microscopic scale in the iron ore gangue.

1.2 Problem Statement

In mining, gangue is the commercially worthless material that surrounds, or is closely mixed with a wanted mineral in an ore deposit. It is thus distinct from overburden, which is the waste rock or materials overlying an ore or mineral body that are displaced during mining without being processed. For any particular ore deposit, and at any particular point in time, the concentration of the wanted mineral(s) in the gangue material will determine whether it is commercially viable to mine that
deposit. The ease with which the ore can be separated also plays an important part. Early mining ventures, with their relatively unsophisticated methods, often could not achieve a high degree of separation, so significant quantities of minerals found their way into the tailing dumps of mines. The success of secondary metals markets depends on the cost of retrieving and processing metals embedded in abandoned structures, discarded product wastes arising from the processing of primary mineral values, and other waste streams and its relation to primary metal price [4].

There are several technologies which may be used to recover metals from industrial waste; examples are pyro metallurgy, hydrometallurgy, and bio-hydrometallurgy [4,7].

Data relating to other constituent apart from iron in the Itakpe iron ore is scarce. There is the possibility of the iron ore containing some other valuable minerals. Therefore, this work focussed on the assessment of the iron ore and the mining gangues for the presence of other valuable minerals suitable for economic recovery

1.3 Objective of Study

The objective of this research is to determine the total useful minerals found in a gangue from the Itakpe Iron ore mining sites, Kogi State (Nigeria).
2.1 Formation of Iron Ore Deposits

An iron ore deposit is a mineral body of sufficient size, iron content, and chemical composition with both physical and economic characteristics that will allow it to be a source of iron either immediately or potentially [2].

Nearly all of the Earth’s major iron ore deposits are in rocks that formed over 1.8 billion years ago. At that time Earth’s oceans contained abundant dissolved iron and almost no dissolved oxygen. The iron ore deposits began forming when the first organisms capable of photosynthesis began releasing oxygen into the waters. This oxygen immediately combined with the abundant dissolved iron to produce hematite or magnetite. These minerals deposited on the sea floor in great abundance, forming what are now known as the “banded iron formation.” The rocks are “banded” because the iron minerals deposited in alternating bands with silica and sometimes shale. The banding might have resulted from seasonal changes in organism activity [8].

Iron ore deposits are formed by three geological processes: direct sedimentation, forming bedded sedimentary deposits; igneous activity, forming segregation or replacement deposit; and enrichment due to surface and near surface weathering [5].

2.1.1 Processes of Iron Ore Formation

**Chemical and physical processes** are the most widespread and common process of metal or mineral concentration is the dissolution of chemical component of rocks, their transport in solution and the precipitation of those components as ore minerals at some specific sites of deposition, usually controlled by chemical contrast or change in chemical environment.
In the process of crystallization from solution, the heavier iron bearing minerals segregate by gravity to form iron-rich deposit. When surface water descends or when underground water ascends through rock strata, iron-bearing minerals may be deposited or gangue minerals such as silica may be leached out [9].

**Mechanical processes** involve erosion, transportation and deposition of generally dense and insoluble rich iron weathered rock material. The higher specific gravity of the iron bearing minerals causes them to be deposited earlier than other lighter materials. This method of formation is responsible for certain sedimentary hematite deposits and magnetite beach sands [10].

2.1.2 **Classification of Iron Ore Deposit**

Iron ore deposits are widespread and have formed in a range of geological environments throughout geological time. These deposits can be divided into four major categories based on their mode of origin [11]. Using aspects of previous classifications by Gross, (1970) and Klemic et al., (1973).

- Sedimentary (banded iron formation, oolitic, placer, swamp),
- Igneous (magmatic segregation and skarn),
- Hydrothermal (proximal and distal), and
- Surficial enrichment (laterite and supergene).

**Sedimentary deposits**, particularly those in banded iron formation (BIF), contain the bulk of the world’s iron resources. Sedimentary deposits include a family of layered silica-rich and iron-rich sediment and meta-sedimentary rocks predominantly composed of cherts or fine –to medium to coarse-grained quartz iron minerals as oxides, carbonates, or silicates [2].

Sedimentary deposits have a common occurrence in Precambrian terranes in rocks of Archean and Proterozoic age older than 1.8 billion years ago with some jaspilite type deposits found in rocks with ages as young as late Precambrian or Cambrian. BIFs are metamorphosed sedimentary rocks composed predominantly of thinly bedded iron minerals and silica (as quartz). The iron minerals
present may be the carbonate siderite, but those used as iron ores contain oxides magnetite or hematite [14].

Banded iron formation (BIF) is commonly associated with sequences containing mafic volcanic rocks, shales and dolomites, consistent with a shallow marine shelf depositional environment e.g. Hamersley Basin, WA [14].

Oolitic sedimentary deposits are massive stratiform oolitic ironstones within marine terrigenous clastic sediments. They are Proterozoic to Cretaceous in age and were an important source of iron before 1970. They are lower in iron content (30-50%) relative to BIF-hosted deposits (55-65%) [1]. Two types have been identified: (1) the Clinton-type which consists of deep red to purple ores composed of hematite, chamosite and siderite and (2) the Minette-type consisting of brownish to dark greenish-brown ores composed mainly of siderite and iron silicates (berthierine and chamosite).

These deposits were formed in shallow marine environments and accumulated along passive continental margins during times of quiescence, extension and global sea level change [15].

**Igneous** deposits are formed either by magmatic segregation of an immiscible magnetite-rich melt in association with layered mafic-ultramafic intrusion or by injection of magnetic-rich fluids into surrounding rocks (e.g. iron sharns). The former occur as massive cumulative-textured seams and are often mined for their economic concentrations of titanium and or vanadium (e.g. Bushveld Complex, South Africa).

Iron skarns (or pyrometasomatic deposits) are mainly derived from granitic to mafic intrusive and can be hosted in a variety of rock types. These deposits are massive, irregularly shaped to tabular bodies that continue to be a source of iron in some countries (e.g. Peru and Russia) [1]. The mineral concentrations in igneous rocks occur as distinctive flows in volcanic successions, or as layers within magmas that cooled at deep crustal levels [9].
Hydrothermal deposits are the source of most of the world’s metals. Hydrothermal deposits provide almost 100% of our Pb, Zn, Mo, and Ag, 60-90% of our Cu, Au and U, as well as gemstones and industrial materials such as clay minerals and quartz. Hydrothermal deposits are diverse, being present in a wide range of geological setting and tectonic environment: some are closely associated with granitic intrusion, others form on the ocean floor and still others are in sedimentary basin [16]. Proximal hydrothermal deposits (also known as volcanic hosted magnetic deposits) are essentially magnetite-hematite bodies that have replaced non-ferruginous host rocks (e.g. Kiruna iron ores). These deposits usually have obvious magmatic signature and adjacent wall rocks are generally intensely altered [17].

Surficial enrichment of iron ore deposits results from sub-aerial weathering processes of generally low-grade ferruginous protore, commonly BIF. Surface and near-surface weathering causes certain dense or hard and chemically inert minerals, which occur in rocks, to survive chemical weathering and be transported in hydrological systems and re-concentrate in different environments. Mature laterites develop under a wet tropical climate and can form extensive duricrust horizontal ferricrete, rich in iron oxyhydroxide.

Supergene enrichment involves weathering and leaching of the upper parts of a mineral deposit (zone of oxidation) and re-deposition of the ore-minerals at lower levels (zone of secondary or supergene enrichment). Fine earthly hematite and iron oxyhydroxides such as goethite, limonite and lepidocrocite are the principal iron minerals produced from surface and near surface enrichment processes [1]. Chemical and physical weathering by soil forming processes of pre-existing iron-bearing minerals resulted in progressive concentration of iron oxides to form iron-rich deposits [5].

2.2 Types of Minerals in Iron Ore

There are almost 300 minerals containing some iron, but only a few of them are considered to be important iron ore minerals. The important iron ore minerals are presented in Table 2.1
Table 2.1: Properties of major iron minerals

<table>
<thead>
<tr>
<th>Minerals</th>
<th>Chemical formula</th>
<th>Theoretical iron content, %</th>
<th>Specific gravity</th>
<th>Mohs Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hematite</td>
<td>Fe$_2$O$_3$</td>
<td>70</td>
<td>5.1</td>
<td>5-6</td>
</tr>
<tr>
<td>Magnetite</td>
<td>Fe$_3$O$_4$</td>
<td>72</td>
<td>5.2</td>
<td>5.5-6</td>
</tr>
<tr>
<td>Martite</td>
<td>α-Fe$_2$O$_3$</td>
<td>70</td>
<td>5.3</td>
<td>5.5-6.5</td>
</tr>
<tr>
<td>Goethite</td>
<td>FeO(OH)</td>
<td>63</td>
<td>3.3-4.3</td>
<td>5-5.5</td>
</tr>
<tr>
<td>Siderite</td>
<td>FeCO$_3$</td>
<td>48</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>Chamosite</td>
<td>(Mg,Fe,Al)$_6$(Si,Al)$<em>4$O$</em>{14}$(OH)$_8$</td>
<td>45</td>
<td>3.1</td>
<td>3</td>
</tr>
<tr>
<td>Pyrite</td>
<td>FeS$_2$</td>
<td>47</td>
<td>4.9</td>
<td>6-6.5</td>
</tr>
<tr>
<td>Limonite</td>
<td>FeO(OH)$_n$(H$_2$O)</td>
<td>63</td>
<td>3-4</td>
<td>5-5.5</td>
</tr>
<tr>
<td>Lepidocrocite</td>
<td>γ-Fe$_2$O$_3$.H$_2$O</td>
<td>60</td>
<td>4.1</td>
<td>5</td>
</tr>
<tr>
<td>Greenalite</td>
<td>Fe$_2$Si$_2$O$_5$(OH)$_4$</td>
<td>45</td>
<td>2.9</td>
<td>3</td>
</tr>
<tr>
<td>Ilmenite</td>
<td>FeTiO$_3$</td>
<td>37</td>
<td>4.7-4.79</td>
<td>5-6</td>
</tr>
</tbody>
</table>

(Source: Ferenzi, 2011)

2.2.1 Types of Iron Minerals

**Hematite** (Fe$_2$O$_3$) is one of the most abundant minerals on Earth’s surface and in the shallow crust. It is an iron oxide with a chemical composition of Fe$_2$O$_3$. It is a common rock forming mineral found in sedimentary, metamorphic, and igneous rocks at locations throughout the world. Hematite ores containing up to 66% iron could be fed directly into iron-making blast furnaces [3]. Their formation is not fully from shallow seas about 1.8-2.6 billion years ago, during the Precambrian period [14].

**Magnetite** (Fe$_3$O$_4$) has higher iron content than hematite ore, but often occurs in lower concentrations. That means it has to be concentrated before it can be used to produce steel. However, the ore’s magnetic properties help separate magnetite ore from rock during processing. Magnetite contains both iron (II) oxide (FeO) and iron (III) oxide (Fe$_2$O$_3$) [18]. High grade magnetite ore normally contains more than 60% iron with some impurities such as silica, alumina and phosphorus.
Martite ($\text{Fe}_2\text{O}_3$) is a secondary hematite formed by chemical replacement of magnetite produced at depth and pressure. The replacement precedes from the outer edges towards the center of the magnetite grains commonly along crystallographic planes [19].

**Goethite and Limonite ($\text{FeO(OH)}$)** are secondary minerals formed by oxidation of Fe-carbonates, hematite, martite, and magnetite. Goethite masses are present in quartz where the goethite has replaced hematite. Goethite that replaced hematite does not contain manganese, whereas goethite that replaced carbonates and martite contains significant amount of Mn (up to 27%wt. Mn) [19]. They are hydrated iron oxide containing up to 60-63% iron. They can occur as primary minerals and always formed relatively near to surface as a result of weathering of the exposed ore.

**Siderite ($\text{FeCO}_3$)** is a valuable iron mineral, since it has 48% iron and contains no sulfur or phosphorus. It constitutes only a small proportion of the total world iron ore reserves.

### 2.2.2 Gangue Minerals

In mining, gangue is the commercially worthless material that surrounds or is closely associated with wanted minerals in an ore deposit. The presence of small amounts of some elements can have either bad or good effect on the characteristic of an iron ore or on the operation of a steel plant. Ideally, iron ore contains only iron and oxygen, but typically, iron ore contains a lot of elements which are often unwanted in modern steel.

There are four main slag forming constituents in iron ores, these include the acidic oxide silica ($\text{SiO}_2$) and the basic oxides lime ($\text{CaO}$) and magnesia ($\text{MgO}$) as well as the neutral alumina ($\text{Al}_2\text{O}_3$). Most iron ores have an excess of $\text{SiO}_2$ and the ash of the coke used for fuel is mainly composed of this oxide, so a basic flux, e.g. limestone must be added [20].

Iron ore contains silica, which is normally removed in the form of slag during the smelting process. Silica is reduced to silicon above 1300°C, which forms an alloy with iron. Major effect of silicon is to
promote the formation of gray iron, which is less brittle and easier to finish than white iron and thus preferred for casting purposes. Silica presence has been reported to reduce shrinkage and the formation of blowholes, lowering the extent of bad casting [21].

Alumina in the form of clays and magnesium aluminum carbonate hydroxide (Mg₆Al₂CO₃(OH)₁₆.4H₂O)) are contained in most iron. Alumina presence increases the viscosity of slag. This will result in slow descent of charge due to thickening of slag and consequently prolonged processes in the furnace. Alumina with higher content will also make slag recovery more difficult, which could lead to a frozen furnace. Increasing the ratio of lime flux will decrease the viscosity [22].

2.3 Global iron ore production and consumption

The world resources of iron ore are estimated to exceed 800 billion tons of crude ore containing more than 230 billion tons of iron [24]. Annual world production of iron ore is usually about one billion tons and, although iron ore is produced in about fifty countries, the 10 with the largest of these countries produce more than 80% of the world total. Table 2.2 present the iron ore production distribution of the world.

Table 2.2: Iron ore production

<table>
<thead>
<tr>
<th>Rank</th>
<th>Country</th>
<th>Iron ore production (thousand tonnes)</th>
<th>Year</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>China</td>
<td>1,380,000</td>
<td>2015</td>
</tr>
<tr>
<td>2</td>
<td>Australia</td>
<td>824,000</td>
<td>2015</td>
</tr>
<tr>
<td>3</td>
<td>Brazil</td>
<td>428,000</td>
<td>2015</td>
</tr>
<tr>
<td>4</td>
<td>India</td>
<td>129,000</td>
<td>2015</td>
</tr>
<tr>
<td>5</td>
<td>Russia</td>
<td>112,000</td>
<td>2015</td>
</tr>
<tr>
<td>6</td>
<td>Ukraine</td>
<td>68,000</td>
<td>2015</td>
</tr>
<tr>
<td>7</td>
<td>South Africa</td>
<td>60,000</td>
<td>2015</td>
</tr>
<tr>
<td>8</td>
<td>United States</td>
<td>43,000</td>
<td>2015</td>
</tr>
<tr>
<td>9</td>
<td>Canada</td>
<td>39,000</td>
<td>2015</td>
</tr>
<tr>
<td>10</td>
<td>Sweden</td>
<td>37,000</td>
<td>2015</td>
</tr>
<tr>
<td></td>
<td>Other countries</td>
<td>&lt;33,000 or 0</td>
<td></td>
</tr>
</tbody>
</table>
Sources: (USGS, 2016)

The largest importer of iron ore is China which is 628 Mt, accounting for two-thirds of world imports in 2009 and produced about 60% of the world’s pigs iron [24]. The same year 2009, Australia is the largest exporting country and in 2009 sent 363 Mt of iron ore overseas. In the same year, exports from Brazil decreased by 3% to 266 Mt and India exported 116Mt [25].

The world most commonly used metal primarily in structural engineering works, in maritime purpose, automobiles, and general industrial application (machinery) is steel. About 89% of iron ore is used to produce pig iron, which is, therefore, the best indicator of iron ore consumption worldwide. The world top 20 steel-consuming countries 2013 in millions tones is shown in figure 2.3.

![Figure 2.1: The world top 20 steel-consuming countries 2013 in millions tonnes (Source: vale, 2015)](image)

The major constraint regarding the iron ore deposits economics is not more often the grade or size of the deposits, reasons are that it is not particularly hard to geologically prove enough tonnage of the
rocks existence. The main constraint is the position of the iron ore relative to market, the cost of rail infrastructure to get it to market and the energy cost required to do so [26].

The Nigerian Steel Development authority Report (1976) describes the relief of the area as a very hilly plateau dipping gently in the northeastern and eastern direction down to the Niger River valley.

At the southwestern part of the plateau, ground elevations range between 230m and 250m above mean sea level (a.m.s.l) while those places closer to the River Niger are no more than 140m-150m (a.m.s.l). Many hills overlooking the plateau are made up of Precambrian gneisses and granites and long ridges with gentle to steep slopes.

The Itakpe Hill deposit strikes E-W for 3Km. Its absolute elevations range from 310m to 410m a.m.s.l i.e. 80m-180m higher than the surrounding valleys.

2.4 Iron Ore Mining

The two main mining techniques used to extract iron ores from their deposits are surface and underground methods. The decision to employ underground or surface mining techniques depends on the proximity of the ore body to the surface [5].

Extraction of mineral or energy resources by operations exclusively involving personnel working on the surface without provision of manned underground operations is referred to as surface mining. While an opening may sometimes be constructed below the surface and limited underground development may occasionally be required, this type of mining is essentially surface-based. Surface mining can be classified into two groups on the basis of the method of extraction; mechanical extraction, or aqueous extraction.

Mechanical extraction methods employ mechanical processes in a dry environment to recover minerals, encompassing the specific mining methods of:

- Open pit mining
- Open cast mining
- Quarrying of dimension stone
- Highwall/auger mining

Open pit and open cast method employ a conventional mining cycle of operations to extract minerals: rock breakage is usually accomplished by drilling and blasting for consolidated materials and by ripping or direct removal by excavator for unconsolidated soil and/or decomposed rocks, followed by materials handling and transportation.

Open pit and open cast mining account for about 96% of non-metal minerals, 87% of metallic ores and 60% of coal production in the world. Surface mining is the most common exploitation method producing nearly 85% of all minerals, excluding petroleum and natural gas. Surface mines of iron ore extract 0.5-30Mt/year.

Dimension stone quarrying is quite similar to open pit mining, but rock breakage without blasting is almost exclusively employed to cut prismatic blocks or tabular slabs of rock. The high labor intensity and cost associated with cutting stone makes quarrying the most expensive surface mining method.

Quarrying of dimension stone and highwall mining are specialized and less frequently used methods. Aqueous extraction in most cases involves the use of water or a liquid solvent to flush minerals from underground deposits, either by hydraulic disintegration or physicochemical dissolution.

Aqueous extraction includes:
- Placer mining
- Solution mining

Placer mining is intended for the recovery of heavy minerals from alluvial or placer deposits, using water to excavate, transport, and/or concentrate minerals. Solution mining is employed for extracting soluble or fusible minerals using water or a lixiviant.
Underground methods are employed when the depth of the deposit, the stripping ratio overburden to one or both become excessive for surface exploitation. Underground mining of iron ore is still being carried out in few large, well established deposit in some parts of the world, for example in the Kiruna Mine in Sweden [1] and Kumba’s Thabazimbi Mine in South Africa [9].

2.5 Iron Ore Mining History and Exploration in Nigeria

The mining of minerals in Nigeria account for only 0.3% of its GDP, due to the influence of its vast oil resources. The domestic mining industry is underdeveloped, leading to Nigeria having to import minerals that it could produced domestically, such as iron ore. Right to ownership of mineral resources is held by the Nigerian government, which grants titles to organizations to explore, mine and sell mineral resources. Organized mining began in 1903 when the Mineral Survey of the Nothern Protectorates was created by the British colonial government. A year later, the Mineral Survey of the Southern Protectorates was founded. By the 1940s, Nigeria was a major producer of some minerals such as tin, columbite, and coal. The discovery of oil in the 1956 hurt the mineral extraction industries, as government and industry both began to focus on this new resource. The Nigerian civil war in the late 1960s led many expatriate mining experts to leave the country [31].

Exploration for iron ore was discovered in Nigeria in 1963 [39] and by 1972/1773 commercial quantity of iron ore was discovered at Itakpe, Kogi State. By 1977, iron ore discoveries at Itakpe had reached 200 million tonnes [40]. More exploration efforts yielded other discoveries in Agbaja, Ajabanoko, Chokochoko, Agbade-Okudu and Nsude Hills. Inventory of proven iron ore reserves in Nigeria is presented in table 3, while iron ore under investigation is presented in table 4. Agbaja has the largest iron ore deposit of 2 billion tonnes.
Table 2.3: Nigeria iron ore reserves (%) and extent of development

<table>
<thead>
<tr>
<th>Location</th>
<th>Fe</th>
<th>Fe₂O₃</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>CaO</th>
<th>MgO</th>
<th>P₂O₅</th>
<th>MnO</th>
<th>TiO₂</th>
<th>Reserved (tonnes)</th>
<th>Extent of dev. as at 2012</th>
</tr>
</thead>
<tbody>
<tr>
<td>Agbaja</td>
<td>45 -54</td>
<td>62.64</td>
<td>8.55</td>
<td>9.06</td>
<td>0.72</td>
<td>0.38</td>
<td>4.16</td>
<td>0.14</td>
<td>0.37</td>
<td>2 billion</td>
<td>Exploration &amp; dev. ongoing</td>
</tr>
<tr>
<td>Itakpe</td>
<td>38 – 45</td>
<td>53.10</td>
<td>44.80</td>
<td>1.00</td>
<td>0.30</td>
<td>0.20</td>
<td>0.05</td>
<td>0.05</td>
<td>0.10</td>
<td>200 – 300 million</td>
<td>Operational but moribund</td>
</tr>
<tr>
<td>Ajabanoko</td>
<td>35.61</td>
<td>47.74</td>
<td>0.41</td>
<td></td>
<td></td>
<td></td>
<td>0.11</td>
<td>0.05</td>
<td>0.06</td>
<td>30 million</td>
<td>Exploration &amp; dev. ongoing</td>
</tr>
<tr>
<td>Chokochoko</td>
<td>37.43</td>
<td>47.65</td>
<td>4.30</td>
<td></td>
<td></td>
<td></td>
<td>0.05</td>
<td>0.52</td>
<td></td>
<td>70 million</td>
<td></td>
</tr>
<tr>
<td>Agbade Okudu</td>
<td>37.43</td>
<td>29.41</td>
<td>0.62</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>70 million</td>
<td></td>
</tr>
<tr>
<td>Nsude Hills</td>
<td>37.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>60 million</td>
<td>Yet to be developed</td>
</tr>
</tbody>
</table>

### Table 2.4: Nigeria iron ore deposits under investigation

<table>
<thead>
<tr>
<th>Location</th>
<th>State</th>
<th>Iron content, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Muro Hills</td>
<td>Nasarawa</td>
<td>25-35</td>
</tr>
<tr>
<td>Dakingari</td>
<td>Kabbi</td>
<td>22-52</td>
</tr>
<tr>
<td>Tajimi</td>
<td>Kaduna</td>
<td>22-52</td>
</tr>
<tr>
<td>Ayaba</td>
<td>Kaduna</td>
<td>27.5</td>
</tr>
<tr>
<td>Rishi</td>
<td>Bauchi</td>
<td>14-19</td>
</tr>
<tr>
<td>Gamawa</td>
<td>Bauchi</td>
<td>40-45</td>
</tr>
<tr>
<td>Karfa</td>
<td>Borno</td>
<td>34-45</td>
</tr>
<tr>
<td>Eginija (Egenerga)</td>
<td>Benue</td>
<td>34-35</td>
</tr>
<tr>
<td>Oko</td>
<td>Anambra</td>
<td>34.4</td>
</tr>
<tr>
<td>Gbege</td>
<td></td>
<td>42.7</td>
</tr>
<tr>
<td>Ajase</td>
<td></td>
<td>39.0</td>
</tr>
</tbody>
</table>


### 2.6 Iron Ore Processing

Iron ore is converted into iron through processes of beneficiation and extraction. Most high grade iron ores can be sent directly to iron extraction plants without beneficiation activities other than crushing and washing, but low grade ores must be beneficiated to upgrade the iron content.

Methods of ore mineral treatment generally involve mechanical and chemical processes. Mechanical methods include hand-picking, wet concentration, dry concentration, amalgamation, magnetic separation, electrostatic separation and flotation [1]. Chemical methods include smelting and the use of solution of chemical agents to dissolve valuable minerals from ore material. Beneficiation improves both physical and chemical characteristics of final iron concentration.

The ore excavated from the mine site is usually crushed using different types of crushers, followed by screening to obtain three basic products as follow: (1) 1-4 cm size fraction, which is used as lump ore in blast furnaces, (2) an intermediate fraction (<1 cm) for sintering, and (3) fines below 150 µm size,
that is either ejected or used to make pellets, often after beneficiation to increase the iron content to over 65%, since the gangue in iron ore gets concentrated in the ‘fines’ fraction [27].

High-grade iron ore is usually crushed and screened to provide direct lump feed of 6-30 mm in size and sometimes fines feed of less than 6 mm in size. The fines can be processed to produce either high-grade sinter (agglomerated fines) or pellets. Low-grade iron ore (e.g. magnetite BIFs, some iron skarns, proximal hydrothermal and oolitic deposits) is usually beneficiated and upgraded by gravity separation, magnetic separation, and/or floatation of the ore minerals, followed by sintering or pelletising [1].

Iron ore is being beneficiated all around the world to meet the quality requirements of iron for the steel industries. However, each source of iron has its own peculiar mineralogical characteristics and requires the specific beneficiation and metallurgical treatment to get the best product out of it. The choice of the beneficiation treatment depends on the nature of the gangue present and its association with the ore structure [28].

2.6.1 Crushing and Grinding

The 1-1.25m lumps excavated at the mine are reduced to a maximum size of 2-4cm for rich ores and to a size which may be as small as 45µm for certain low grade ores, to enable high grade concentrate to be obtained. The first stage of crushing produces particles of 15-25cm in size. Both gyratory and jaw crushers can be used as primary crusher. Factors considered in determining the degree of ore crushing include the concentration of the iron in the ore, its mineralogy, hardness, and moisture content [28].

The second stage of crushing is usually carried out using cone or gyratory crushers, may be carried out in a single operation if the aim is to produce a material in the size range of 3-5cm. If the ore must be ground fine for concentration, it is usually done by rod or ball mills or combination of both down to particle size of approximately 1-2.5cm. Rod mills grind the larger particles preferentially and so
produce a smaller proportion of very fine particles, followed by an initial concentration, after which further grinding in ball mill is carried out.

Grinding system employed in most operation include, autogenous or semi-autogenous (SAG) grinding system. Autogenous grinding uses coarse pieces of the ore itself as the grinding media in mill. Semi-autogenous operations use metallic balls and/or rods to supplement the grinding action of the ore pieces. Autogenous grinding is best suited to weakly cemented ores containing some hard material [28].

2.6.2 Concentration Methods

Separation of iron oxide from gangue minerals involve many methods such as gravity method, magnetic separation, and reduction roasting followed by magnetic separation, floatation and electrostatic separation. In addition to these methods, some degree of concentration can be achieved by washing [30]. Concentration of valuable minerals from gangue involves exploitation of the differences in the mineral properties of the ore after effective comminution [29].

2.6.2.1 Magnetic separation

Magnetic separators exploit the difference in magnetic properties between the ore minerals and are used to separate the magnetic minerals (magnetite, in some application hematite) from non-magnetic gangue such as quartz. Magnetic separation can be classified into low-and high-intensity machines, which may be further classified into dry-feed and wet-feed separation [31].

When the minerals is magnetic, low-intensity (500-1200G) separation is normally practiced because it is relatively cheap and effective. If the particles are of comparatively large size (greater than 6mm), dry magnetic separations is used. When the particles are less than 100µm in size, wet magnetic separation is used. If the size of the ore is intermediate, it is possible to use either method. High-
intensity (1200-22000G) separators can be used to separate weakly magnetic materials, such as hematite and hydrated hematite from gangue materials, for both wet and dry iron ores [31]. Wet high-intensity magnetic separation has its greatest use in the concentration of low grade iron ores containing hematite, which frequently replaces flotation methods, although the trend towards magnetic separation has been slow in North America, mainly due to the very high capital cost of such separators [30]. Magnetic separation techniques are used to beneficiate over 90% of all iron ores in the world, but 20-35% of all the iron ores being beneficiated today is lost to tailings because hematite is only weakly magnetic [5].

2.6.2.2 Washing

Sometimes ores can be enriched by the simple process of washing such ores that consist of coarse and fine particles of clean ore minerals mixed with either barren sand or clay. Generally, clay minerals increase the alumina in iron ore which is easily reduced by washing. Separation of clay minerals consists of a simple scrubbing operation in a long washer or classifier followed by a screening operation to remove the coarser iron ore particles, whereas the overflow usually is the waste product.

2.6.2.3 Froth flotation

This is a selective process and can be used to achieve specific separation from complex ores. This process utilizes the differences in physicochemical surface properties of particles of various minerals. After treatment with reagent, such differences in surface properties between the minerals within the flotation pulp become apparent and, for flotation to take place, an air-bubble must be able to attach itself to a particle, and lift it to the surface.

Froth flotation can effectively be used to upgrade low grade iron ores to high iron ore concentrates. For flotation to take place the particle size should be at least 250µm. Fatty acids or petroleum
sulfonates can be used as collector in anionic flotation to float fine iron oxide, such as magnetite, hematite, or siderite away from waste minerals such as quartz or chert. Cationic flotation may be used to upgrade fine concentrate by floating the gangue minerals away from the iron minerals [32]. The efficiency of froth flotation process decreases when slim is apparent. Magnetic separation and flotation are the most widely accepted technologies for concentrating iron ore particles, however, these processes result in iron concentrate with high amounts of very fine and/or interlocked silica particles [32].

2.6.2.4 Electrostatic separation

Electrostatic separation is limited to relatively few iron ores. The major process makes use of the differences in electrical conductivity between iron oxides and gangue minerals. It works best on crystalline, nonmagnetic iron oxides finer than 1.7mm and coarser than 75µm. The minerals surfaces should be free from slim or dust coating [30].

2.6.3 Fine Iron Ore Processing

Fine iron ores are not preferable as direct feed to that of blast furnace due to the tendency of packing into a non-permeable bed or their fine particles are likely to be carried away as dust by the high gas flow rates. Fine iron ore must therefore be agglomerated into larger particles that will improve permeability of the furnace burden, increase throughput, and reduce the amount of material blown out of the furnace as dust.

Agglomeration is a size enlargement process which involves combining small size particles to create products with new larger particle sizes. Product can come in many different forms such as granules, tablets, briquettes, pellets, sinters, bricks, or compacts. The resulting entity is only apparently a new unit but the original solid particles are still present in the structure, often with completely unaltered
shape and size, and are held together by binding mechanism. Agglomeration processes may involve the application of pressure or thermal method to iron ore fines with materials such as bentonite, limestone and dolomite [33].

2.7 Iron ore characterization

The raw materials for steel plants were statistically and empirically determined not until the 1970s. Control on iron ore blends was exercised only by chemical composition, especially SiO$_2$, Al$_2$O$_3$ and alkali contents. Other properties of iron ore, like porosity and pore size distribution, pseudo-particle formation characteristics, fusibility or assimilation characteristics and several abnormalities in plant operation necessitated detailed examination of iron ores.

The characterization of iron ore is very important method in quantitative classification of mineral deposits and all other steps in process flow sheet development. Without proper understanding of the ore characteristic of the deposit, may be impossible to develop a successful process flow sheet. Iron ore characterization provides information for clearer understanding of the mineralogical as well as the chemical nature of the ore [34].

2.7.1 Chemical Characterization

In order to determining the quantitative chemical composition of samples, it is subjected to chemical analyses [35]. Wet chemical methods of analysis are destructive methods used in chemical mineral assay. Some of the wet chemical methods include classical volumetric, gravimetric, and colorimetric analysis. Classical volumetric and gravimetric methods of analysis are used principally for the determinations of the higher concentrations of various elements in ores and concentrates. Colorimetric methods of analysis are used for trace element analysis.

21
X-ray fluorescence (XRF) analysis is a nondestructive analytical technique used for the determination of elemental concentration of samples. It provides rapid elemental identification and quantification of solid, liquids and loose powders. XRF spectrometers are capable of measuring elements from beryllium (Be) to uranium (U) and beyond at trace levels, often from below 1 ppm up to 100%. XRF spectrometers measure the characteristic wavelength of the fluorescent emission produced by a sample when irradiated with X-rays. ISO 9516 is an international standard method that describes the calibration and elemental contents of a full range of major, minor and trace elements. Some of the elements, which are usually determined in iron ores include iron (Fe), silicon (Si), aluminum (Al), magnesium (Mg), calcium (Ca), sodium (Na), potassium (K), titanium (Ti), manganese (Mn), phosphorus (P), chromium (Cr) and Sulphur (S) [36].

2.7.2 Physical and Mineralogical Characterization

Characterization of iron ore by physical and mineralogical studies identifies major minerals, gangue minerals and their association as grain size in the different ore phases. The mineralogy of iron ore varies from quite simple hematite and quartz with little of aluminum mineral to hydrated hematite along with complex gangue minerals like hydrated aluminum silicate or Kaolinite \(\text{Al}_2\text{Si}_2\text{O}_5\text{(OH)}_4\) and trihydrated aluminum oxide or gibbsite \(\text{Al(OH)}_3\).

The hematite can be partially hydrated as in goethite \(\text{FeO(OH)}\) or deoxidized as in magnetite \(\text{Fe}_3\text{O}_4\). In some ore bodies the quartz is the predominant gangue mineral and for all practical purposes aluminum minerals are absent. Some ore bodies, on the other hand, may contain mainly magnetite and only very little hematite. The gangue minerals may vary from simple to complex types as above. The extend of hematite or magnetite, total iron content, and any of the impurities vary from source to source [34].

Study with unaided eye, with hand lens and/or with stereomicroscopy to gather information on the ore deposit is done by macroscopy method. Physical characteristics, which can be obtained from
macroscopic study include crystalline or amorphous nature of the ore, whether it is soft, hard and flaky, or dust ore material. The magnetic characteristics of an iron ore deposit can also be determined using a magnetic pen.

Microscopic study identifies the major minerals present in the ore body and grain size of each component, which is a useful metal liberation study.

Thermal analysis involves the study of the changes in the weight of the samples as a function of time and temperature. Thermal analysis of iron ore gives an indication of its thermal stability, composition of intermediate compounds that may be formed, and the final residue that is left behind after exposure to any given temperature for a given duration. Thermal methods are used for characterizing a system (element, compound or mixture) by measuring changes in physico-chemical properties at elevated temperature as a function of increasing time.

Two main thermal analysis technique, differential thermal analysis and thermogravimetric analysis, provide information about physical phenomena, such as second-order phase transition, including vaporization, sublimation, absorption, adsorption and desertion. Likewise, thermal analysis can provide information about chemical phenomena including chemisorptions, desolvation (especially dehydration), decomposition, and solid-gas reaction (for example, oxidation or reduction) thermogravimetric analysis is commonly used to determine selected characteristics of materials that exhibit either mass loss or gain due to decomposition, oxidation or loss of volatile matter (such as moisture) [37].

2.7.3 X-Ray Diffractometric Analysis

X-ray diffraction (XRD) is a powerful technique for the analysis of iron ore because it can tell you what phases contain iron, e.g. Hematite Fe$_2$O$_3$, Magnetite Fe$_3$O$_4$, or Goethite FeO(OH); it can identify other minerals phases present, especially silica, and can provide quantitative analysis of all phases.
Also, knowledge of the phases present can predict processing required to beneficiate the ore (flotation, magnetic separation, etc.).

X-ray diffraction (XRD) analysis is used to identify the crystalline phases present in geological specimens and processed materials. About 95% of solid materials are crystalline, which means that they have a regular three-dimensional distribution (lattice) of atom.

An XRD pattern of a sample is the summation of diffraction pattern from each phase in that sample. This allows the identification of phases in the sample from their diffraction patterns. The amount of each phase in a mixture will relate to the strength of its signal in the final pattern and this allows the quantification of phase in mixtures. XRD is an important technique in mineral processing because it is the mineralogy rather than the chemistry that generally controls the mineral processing [38].
CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Location and Study Area

The study area, Itakpe and environs is a rural area in Okene Local Government area of Kogi State in central Nigeria. It lies within latitudes 7º36’N to 7º39’N and longitudes 6º17’ to 6º22’E. Itakpe is northeast of Okene and is about 10Km along the Okene Lokoja road. Fig 3.1 shows the location of the study area, access roads and major geographic features. The National Iron Ore Mining Company (NIOMCO) mining area is located to South of Abuja, in Itakpe, Kogi State. Its local boundaries are with Abuja to the North, Nassarawa State to the North-East, Benue State to the East and Enugu State to the South-East as it is shown in fig 3.2

![Figure 3.1: location of the study area, access roads and major geographical features](image-url)
3.2 Climate and Vegetation

The climate condition in Itakpe, Kogi State is similar to that of other parts of the Middle Belt of Nigeria with rainfall stretching for about seven (7) months, from April to October and dry season lasting for five months from November to March (N.S.D.A 1976). The mean annual precipitation at Itakpe is 1,200 mm. The peak period of rain is between July and September with a short break experienced in August. Rainfall decreases to 10 mm-15 mm in December and January. Harmattarn or cool Northeastern winds blow in the area for most part of the dry season. This is specifically intense in January and February. This is characterized by dust from the Sahara. Relative air humidity varies from 80% between July and August to 60% between January and February. The monthly average temperature ranges from 28°-29°C in July and August to 34°-36°C in February and March. The vegetation of the area is characteristic of the forest Savanna with bush scattered low trees, grasses and shrubs.

Figure 3.2: Map of Nigeria showing the location of the National Iron Ore Mining Company (NIOMCO) concession area (Source: WCNF, 1997).
3.3 Geology of the Study Areas

The Itakpe iron-ore deposit is localized within the gneiss-migmatite quartzite unit of the Nigeria basement complex [39]. The general geology of Itakpe noted that the dominant lithologic unit in the area is the granodiorite-tonalite gneiss, overlain by a sequence of low grade metasediments and intruded by granodioritic and granitic rocks. The main rock types identified include granite gneiss, amphibolites, quartzites, schists, granite and pegmatimatites [42].

There are two types of quartzites in the area: the ferruginous and non-ferruginous. The ferruginous quartzites occur as magnetite-rich and hematite-rich bands and lenses about 10-60m wide in alternation with gneiss. The non-ferruginous quartzites are rare on the Itakpe, but constitute the bulk of the rock on its southern edge [42].

Three main orebodies have been delineated which comprise a group of ferruginous quartzite bands or lenses. All orebodies crop out at the surface or are capped by thin overburden. The northern orebody occur at the northern flank of the ridge. It has a strike length of 400m and a length of 200m along dip direction with thicknesses ranging from 60m to 30m. The central orebody extends along strike for 200m, and it increases in thickness from west to east. Two small ore lenses 35 and 15m thick coalesce to form the main orebody which is approximately 40m wide. The southern orebody dips steeply to the south and extends along strike for about 500m with a thickness of approximately 15m. Its width is about 40m. The deposit has been folded and faulted and it has been affected by regional metamorphism.
3.4 Experimental Procedure

So as to achieve the objective of this research work, the experimental procedure are based on sampling of the iron ore and the mining gangue, characterization of the iron ore and the mining gangue, and quantification of associated valuable minerals in the ore at Itakpe.

3.4.1 Sample Collection

The methods specified in the ISO 3082, 2009 are applicable to both the loading and discharging of a lot by means of belt conveyors and other ore-handling equipment to which a mechanical sampler may be installed or where manual sampling may safely be conducted (ISO 3082, 2009).

The iron ore was crushed after which samples of iron ore gangue and iron ore concentrate were collected from three locations for 5 different samples within the Itakpe iron ore mining deposit.
Three samples of iron ore after crushing from the blending yard were collected which include the coarse, middle fine and fine grains sizes. Location (1) 3.5 kg of each were collected at NO 7º 36’ 53.1” and EO 06º 19’ 39.6”.

Samples of iron ore concentrate were collected from the sinter feed. Location (2) 3.2 kg of the concentrate were collected NO 7º 37’ 04.2” and EO 06º 19’ 05.7”.
Iron Ore gangue sample were also collected from the disposal area. Location (3) 4.3kg at NO 7º 36’ 44.8” and EO 06º 19’ 51.1”.

3.4.2 Sample Preparation
Samples were collected, dried, blended, divided, crushed, and ground as required. Sample analysis was performed in the laboratory of NASENI Centre of Excellence in Nanotechnology and Advanced Materials, Akure, Ondo State, Nigeria.

3.4.3 Laboratory Analysis
The X-ray machine used for the analysis was EMMA 0141 GBC. The samples of iron ore: coarse, middle fine and fine grains sizes, concentrate and gangues samples of the processed type was grinded to a powdered sample of each using an agate mortar for uniformity. The grounded samples was compacted in the corvette and placed inside the sample holder of the machine.

The samples were exposed to the radiation of the machine at an average wavelength of 1.541178 angstrom for 120seconds. The spectrum and the parameters was obtained after the running of the samples and the result of each samples further analyzed using the software TRACE and PDF 2 containing the data base supplied by the International Centre for Diffraction Data (ICDD) which is installed in the PC attached to the machine.
Also the iron ore gangue and concentrate was carried out using SKYRAY Energy Dispersed X-Ray Fluorimeter (EDX3600BXRF). The spectrometer applies XRF technology to conduct fast and accurate analysis of complex composition. The system detect elements between Sodium (Na, Z=11) and Uranium (U, Z=92) with high resolution and fast analysis.

The non-homogeneous samples was pulverized to fine homogeneous size and then pelletized. The equipment was calibrated using pure silver sample to confine the efficient of the equipment before testing the unknown samples. Each of the samples were loaded and tested on the equipment and the data was saved before unloading the sample from the equipment after which the result was exported to the Excel sheet.
CHAPTER FOUR

4.0 RESULT AND DISCUSSION

4.1 Sample analysis

Iron ores should preferably have high Fe content and low impurity element content, in order to justify the investment during exploration. The generalized content for the most important elements in the iron ore sample from the Itakpe Iron ore Mining Company is hematite ($\text{Fe}_2\text{O}_3$) or Iron oxide with atomic % of Fe 40.00 and weight % of Fe 69.94. Also, there are some minerals of values which are associated with iron ore that are disposing as waste in the gangues. In the world practice, no minimum standards have been set for iron, silica, alumina, calcium, and magnesium percentages in commercial iron ores, although certain generalizations can be made [5].

4.2 Elemental analysis

The complete elemental analysis of the five samples of the ore is shown in Table 4.1. The result of the samples showed high percentage of iron element ranging from 34.60% for GIO, 51.59% for $M_\text{i}\text{IO}$, 53.80% for FIO, 56.34% for CIO, and 64.51% for CoIO (i.e. $\text{GIO}<M_\text{i}\text{IO}<\text{FIO}<\text{CIO}<\text{CoIO}$). Since the amount of iron present in the gangue is as high as approximately 35%, this can still be recovered. Also, all the sample contains some percentage of Silica ranging from 0.36% for CIO, 1.63% for CoIO, 11.60% for FIO, 12.53% for $M_\text{i}\text{IO}$ and 22.97% for GIO (i.e $\text{GIO}>M_\text{i}\text{IO}>\text{FIO}>\text{CoIO}>\text{CIO}$); this amount of silicon present in the gangue can be recovered. The gangue contains the lowest percentage of iron but the highest percentage of silicon. The reason may be attributed to the fact that the $C_o\text{IO}$ is fully processed for industrial application. From the analysis, it was also observed that sulfur and potassium can only be found in the fine iron ore, middle fine iron ore and gangue iron ore while there are no traces of magnesium, titanium, arsenic, gold, cadmium and niobium in the 5 analyzed samples.
Although, there are a little traces of Cd in the middle fine iron ore, 0.0001%; this can be neglected.

In the gangue, there is 2.43% of Aluminium. This is not economically viable. A higher percentage of Aluminium can be gotten from Bauxite and it’s going to be economically feasible if it contains more than 50% of aluminium oxide. Tungsten can only be found in the gangue of iron ore.

Table 4.1: The XRF Result of Elemental composition of CIO, FIO, M₄IO, GIO and C₆IO [wt (%)]

<table>
<thead>
<tr>
<th>ELEMENT</th>
<th>CIO</th>
<th>FIO</th>
<th>M₄IO</th>
<th>GIO</th>
<th>CoIO</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mg</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
</tr>
<tr>
<td>Al</td>
<td>0.1257</td>
<td>0.6702</td>
<td>0.8090</td>
<td>2.4328</td>
<td>0.4462</td>
</tr>
<tr>
<td>Si</td>
<td>0.3577</td>
<td>11.6003</td>
<td>12.5277</td>
<td>22.9689</td>
<td>1.6303</td>
</tr>
<tr>
<td>P</td>
<td>0.0066</td>
<td>0.2046</td>
<td>0.2306</td>
<td>0.1547</td>
<td>0.0278</td>
</tr>
<tr>
<td>S</td>
<td>0.0000</td>
<td>0.0072</td>
<td>0.0229</td>
<td>0.0725</td>
<td>0.0000</td>
</tr>
<tr>
<td>K</td>
<td>0.0000</td>
<td>0.0017</td>
<td>0.0007</td>
<td>0.4017</td>
<td>0.0000</td>
</tr>
<tr>
<td>Ca</td>
<td>0.0293</td>
<td>0.1124</td>
<td>0.1513</td>
<td>0.0813</td>
<td>0.0787</td>
</tr>
<tr>
<td>Ti</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
</tr>
<tr>
<td>V</td>
<td>0.0223</td>
<td>0.0156</td>
<td>0.0115</td>
<td>0.0120</td>
<td>0.0174</td>
</tr>
<tr>
<td>Cr</td>
<td>0.0696</td>
<td>0.0477</td>
<td>0.0430</td>
<td>0.0312</td>
<td>0.0589</td>
</tr>
<tr>
<td>Mn</td>
<td>0.0869</td>
<td>0.0819</td>
<td>0.0678</td>
<td>0.0615</td>
<td>0.1102</td>
</tr>
<tr>
<td>Co</td>
<td>0.6473</td>
<td>0.7989</td>
<td>0.9918</td>
<td>0.6779</td>
<td>0.8996</td>
</tr>
<tr>
<td>Fe</td>
<td>55.3439</td>
<td>53.7953</td>
<td>51.5900</td>
<td>34.6072</td>
<td>64.5093</td>
</tr>
<tr>
<td>Ni</td>
<td>0.0570</td>
<td>0.0403</td>
<td>0.0280</td>
<td>0.0383</td>
<td>0.0442</td>
</tr>
<tr>
<td>Cu</td>
<td>0.0185</td>
<td>0.0181</td>
<td>0.0168</td>
<td>0.0296</td>
<td>0.0157</td>
</tr>
<tr>
<td>Zn</td>
<td>0.0308</td>
<td>0.0285</td>
<td>0.0301</td>
<td>0.0583</td>
<td>0.0292</td>
</tr>
<tr>
<td>As</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
</tr>
<tr>
<td>Pb</td>
<td>0.1573</td>
<td>0.0903</td>
<td>0.0670</td>
<td>0.0285</td>
<td>0.1151</td>
</tr>
<tr>
<td>W</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0248</td>
<td>0.0000</td>
</tr>
<tr>
<td></td>
<td>Au</td>
<td>Ag</td>
<td>Rb</td>
<td>Nb</td>
<td>Mo</td>
</tr>
<tr>
<td>---</td>
<td>------</td>
<td>------</td>
<td>------</td>
<td>------</td>
<td>------</td>
</tr>
<tr>
<td></td>
<td>0.0000</td>
<td>0.0003</td>
<td>0.0046</td>
<td>0.0000</td>
<td>0.0798</td>
</tr>
<tr>
<td></td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0024</td>
<td>0.0000</td>
<td>0.1217</td>
</tr>
<tr>
<td></td>
<td>0.0000</td>
<td>0.0017</td>
<td>0.0023</td>
<td>0.0000</td>
<td>0.1946</td>
</tr>
<tr>
<td></td>
<td>0.0000</td>
<td>0.0029</td>
<td>0.0022</td>
<td>0.0000</td>
<td>0.2141</td>
</tr>
<tr>
<td></td>
<td>0.0000</td>
<td>0.0000</td>
<td>0.0042</td>
<td>0.0000</td>
<td>0.0994</td>
</tr>
</tbody>
</table>


### 4.3 Spectral Analysis

The spectral result of CIO, FIO, MfIO, CIO and GIO using EDX attached to the XRF machine is shown in figure 4.1 to 4.5. The results indicate the presence of iron peak of varying intensity. The intensity was observed of the increasing order of CoIO > FIO > CIO > MfIO > GIO (i.e. 37500 > 27500 > 23000 > 20000 > 11200). The obtained result affirmed the fact that concentrate iron ore sample has the highest content of iron while the gangue contains the lowest iron content (since the value of the intensity is proportional to the concentration) as indicated in the XRF result. Also, the presence of silicon peak of varying intensity was observed with the gangue having the highest intensity of 7000 while the concentrated iron ore and coarse iron ore has a negligible intensity. The varying intensity of cobalt in the 5 sample cannot be neglected as it varies in the order GIO < MfIO < CIO < FIO < CoIO (i.e. 1500 < 2850 < 3000 < 4000 < 5000).
Fig. 4.1: EDX of CIO

Fig. 4.2: EDX of FIO
Fig. 4.3: EDX of M₁IO

Fig. 4.4: EDX of GIO

Fig. 4.5: EDX of C₃IO
4.4 Structural Analysis

Figure 4.6 to 4.9 shows the result of structural analysis of (UPIO), M,IO, C,IO and GIO using XRD machine. The spectral of the UPIO contains so many peaks when compare with the spectrum of other samples, this is attributed to high concentration of impurities. Analysis carried out on the analyzed samples using International Centre for Diffraction Data (ICDD) software attached to the machine indicated that the type of iron ore deposit at Itakpe is Hematite (Fe₂O₃) with the crystal structure of Centrosymmetric (Joint Committee on Powder Diffraction Standard card number; 00-006-0502). Moreover the observation from the spectrum of GIO show seven (7) prominent peaks against three (3) peaks on the C,IO spectrum indicating that the gangue contains high impurity especially silica.

4.5 Presence of Silicon the Gangue

From Table 4.1, the gangue contains about 23% Si by weight while the concentrate contains 1.63%. The presence of Si in the iron ores is usually in the form of silica (SiO₂). Thus, it may be said that the concentrate and the gangue contain 2.56% SiO₂ and 36% SiO₂ respectively. The SiO₂ content in the concentrate is extremely useful as a slag forming flux in the subsequent smelting operation on the concentrate. The production of Si is mainly in the form of alloys, either as silicomanganese or ferrosilicon. Again, since the gangue still contains a some iron, the silicon in the gangue may be recovered as ferrosilicon.
Fig. 4.6: XRD of Unprocessed Iron Ore

Fig. 4.7: XRD of $\text{M}_3\text{IO}$
Fig. 4.8: XRD of CoIO

Fig. 4.9: XRD of GIO

NB: UPIO = Unprocessed Iron Ore, MfIO = Middle Fine Iron Ore, GIO = Gangue Iron Ore, CoIO = Concentrate Iron Ore.
CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusions

In this study, the iron ore mining gangues was investigated for the recovery of metal which can be processed in making different useful materials.

The sample analysis using the XRD indicates that the iron ore deposit at Itakpe is hematite ($\text{Fe}_2\text{O}_3$) or Iron oxide-rich with atomic % of Fe to be 40.00 and weights % of Fe to be 69.94. The main impurity in the ore is silica.

The elemental analysis on the coarse, middle fine, fine, concentrate and gangues using the XRF shows that the gangue still consist of 34.6% of iron and 22.97% of silicon which can be recovered as ferrosilicon while the concentrate iron ore has 64.51% -the highest percent of iron and 1.63% of silicon in the five samples examined.

From the structural analysis, the sample of the unprocessed iron ore has more content of impurities indicated by several peaks and noise when compared with the middle fine, concentrate, and gangue iron ore. As been expected, the concentrate has fewer peaks and noise since it contains little amount of impurities.

The spectral analysis result shows that the concentrate iron ore has the highest content of iron since it has the highest intensity of 37500. The gangue iron ore has the lowest iron content of 34.6% with the lowest intensity of 11200 when compared to the other sample. This is also evidence that the gangue iron ore consist of metal that can still be recovered.
5.2 Recommendations

The high percentage of iron concentration (34.60%) in the gangue which we term as waste has indicated loss of iron mineral during processing. Therefore there is need for further work on the beneficiation method or research into developing a new method which will not only be more economical but also increase the recovery of more iron from the gangue leading to its reduction to at most between 5 % - 10% instead of 34.60% as observed in the analyzed gangue. Also, the gangue can be recycled into the system for recovery of metal and this will decrease the amount of iron present in the gangue and the same increase the amount of iron produced.


45

43. Annor and Freeth (1985), wrote on the thermo tectonic evolution of the basement complex around Okene Nigeria with special reference to deformation mechanism.


