Reverse Flotation Potential of Bikilal Iron Ore Deposit: A Project Work on Wollega Area Western Ethiopia

Samuel Getaneh Simegn

A project Submitted to

Center for Ethio-Mines Development

Presented in Partial Fulfillment of the Requirements for the Degree of Master of Science (MSc) in (Mineral Engineering)

ADDIS ABABA UNIVERSITY
Addis Ababa, Ethiopia
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Approval page

Addis Ababa University

School of Graduate Studies

This is to certify that the Project prepared by **Samuel Getaneh Simegn** entitled: Reverse Flotation Potential of Bikilal Iron Ore Deposit : A project work on the Floatability of Magnetite Iron Ore Deposit found in Bikilal Area Wollega Western Ethiopia, To be submitted for the fulfillment of the required Degree of Master of Science (MSc) in mineral engineering with the standard document quality according to the rules of the university and accepted by the graduation committee regarding to the originality of the paper.

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Abstract

Iron is one of the most abundant rock forming elements, constituting about 5% of the earth’s crust. It is the fourth most abundant element next to oxygen, silicon and aluminum and after aluminum, the most abundant and widely distributed metal. Iron is extracted primarily from iron ores. The iron ores mainly include oxides and hydroxides such as magnetite (Fe_3O_4), hematite [Fe_2O_3], goethite [FeO(OH)], and limonite [$FeO(OH)\_nH_2O$].The primary gangue mineral in iron ores is quartz. The main problem regarding to beneficiate the Bikilal iron ore deposit are the presence of some deleterious gangue minerals such as Phosphorous, Alumina and silica and lack of advanced separation equipment. The aim of this project work is to indicate that the Bikilal iron ore deposit has naturally low grade iron content which is 41.2%Fe and it can be improved by anionic reverse flotation. The method used to upgrade this ore is first the qualitative and quantitative mineralogical analysis has been determined by atomic absorption spectroscopy (AAS) to know the initial amount of iron in the ore. Based on this information reverse anionic flotation has conducted in a batch flotation cell in two size classes at 75µm and 63µm. Oleic acid has been used as a collector whereas Ethanol used as a Frother. Modifiers such as activators, depressants and pH regulators have also used to promote the efficiency of flotation process. The concentrate of flotation has been analyzed again by (AAS) to evaluate the performance of the selected method. The amount from the concentrate has significantly increased from 41.2%Fe average grade to 62%Fe grade. The recovery of iron have been calculated as 65% for under 63µm and 74.4% for under 75µm.The amount of the concentrate for 63µm was 215g and that of 75µm was 250g. Therefore the recovery of iron from 215g under 63 µm is 65.5%Fe which is better than the recovery from 250g under 75µm that is 74.4%Fe. This project work mainly conducted based on (AAS) analysis results but it is not suffifeint to judge the general mineralogical composition and beneficiation process that should be applied in the Bikilal iron ore deposit. Therefore additional investigations and laboratory analysis such as (XRD), (SEM) and (XRF) are recommended to come up with a more tangible and realistic data. This paper have also discusses in detail about the factors that can affect the flotation performance of the ore and the alternative chemical reagents that can go through the mineralogy of Bikilal iron ore deposit.

Key words: Iron ore beneficiation, Flotation, Direct flotation, Reverse Flotation, Cationic reverse flotation, Anionic reverses flotation.

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CHAPTER ONE - INTRODUCTION

1.1. General Background

Iron is the backbone of the world we have built around us and it is the basic ingredient of steel. It is a very useful metal because it can be mixed with other metals to make a whole variety of alloys which are stronger and don't rust easily, can be shaped into products from cars to pins, household appliances to buildings, bridges to railways, food cans to tools (Australian Government Geo science, 2015).Iron is one of the most abundant rock forming element, constituting about 5% of the earth's crust. It is the fourth most abundant element next to oxygen, silicon and aluminum and after aluminum, the most abundant and widely distributed metal. The iron ores mainly include oxides and hydroxides such as magnetite [Fe3O4], hematite [Fe2O3], goethite [FeO(OH)], and limonite [FeO(OH)_nH2O].The primary gangue mineral in iron ores is quartz. In addition to quartz, iron-bearing silicates (e.g., amphiboles and pyroxenes), carbonates, clays (e.g., kaolinite), and gibbsite are also commonly present in iron ores.

Bikilal iron ore deposit is a Kiruna-type magnetite – ilmenite deposit located in West Wollega Western Ethiopia. This deposit have been proved a total ore reserve of 57 million tons of magmatic origin at average grades of 23.3 % magnetic iron and 41 % total iron with the help of drilling and trenching (Ethio-Korean Iron Exploration Project, 1988). Bikilal iron ore deposit consists of minerals like magnetite, ilmenite, hematite, sulphides, apatite and gold. From these the principal ore minerals are magnetite and ilmenite. Based on the ore microscopic studies, the ore mineral assemblages consists of 5-12% ilmenite, 20-25% of magnetite, >10% of hematite, and small amount of sulfides (pyrite, pyrrhotite, and chalcopyrite, pentlandite) in average (Solomon, R. 2013).The major gangue minerals are quartz, K-feldspar, biotite and chlorite while calcite, sphene, rutile, and goethite are minor gangue minerals.

Research of iron ore flotation dates back to 1931 and judging from processing of iron quartzite, reverse cationic flotation is a very efficient method of beneficiation of oxidized iron ores. The same method can be also applied to reduce (SiO_2) content in magnetite concentrates obtained by low-field magnetic separation. A variety of flotation routes have been developed to remove silica from iron minerals. Recently there is a marked shift from iron ore lumps to fines in iron making. The processes, which permit the use of ore fines, have become commonplace in iron and steel make industry. Further, recovery of values from fines generated during mining, milling and washing wastes is being practiced at all mines. Usual physical separation techniques like Low Intensity Magnetic Separation (LIMS), Medium Intensity Magnetic Separation (MIMS) and High Gradient Magnetic Separation (HGMS) are inadequate to address the problems associated with fines and interlocked particles. Flotation is the most suitable technique for the selective separation of iron ore fines from gangue minerals. Direct flotation of iron ore using oleic acid is relatively expensive in terms of high reagent consumption and entrapment of gangue minerals in the froth.

Typically there are 3 different types of chemicals that are used in the froth flotation process: collector, Frother, and modifier. First the “Collector” is added to the iron ore slurry for selectively coat the iron oxide particles, makes the surface hydrophobic. The slurry then goes to a flotation cell, where air bubbles is generated using an impeller and aerator. At this step, the “Frother” process (for example, fuel oil) is added to the ore slurry to form stable froth and air bubbles. A “Modifier” process is added in some cases in order to enhance the performance of the collector process.

1.2. Statement of the Problem

Iron making is currently shifting from lumps to fines, but traditional physical separation techniques like Low Intensity Magnetic Separation (LIMS), Medium Intensity Magnetic Separation (MIMS), and High Gradient Magnetic Separation (HGMS) are not effective in addressing the challenges associated with fines and interlocked particles. Flotation is the most appropriate method for the selective separation of iron ore fines from gangue minerals. However, direct flotation is not suitable for some gangue materials, including sulfur, phosphorus, fluorine, and carbonate gangue materials, as they behave similarly to iron ore in direct flotation, making it difficult to achieve good separation. The use of reagents that can penetrate both valuable and gangue minerals is another challenge, particularly with commonly used cationic amine collectors that have both collecting and frothing properties, resulting in high viscosity and poor fluidity of the froth during flotation. The dosage of reagents used in cationic reverse flotation and desliming stages is also a challenge and requires additional costs for processing iron concentrate. Furthermore, cationic amine collectors are toxic and pose an environmental hazard if not disposed of properly.

The presence of phosphorus in the form of apatite is a significant issue in the beneficiation of iron ore, as it is challenging to remove and can make the steel brittle. Phosphorus is the main gangue mineral in Bikilal iron ore deposit in the form of apatite, and its high level attracts a penalty and substantially reduces the profit margin. The increased residence time in alkaline leaching of phosphorus decreases its removal due to re-precipitation phenomena. To address these challenges, the proposed project uses anionic reverse flotation instead of cationic reverse flotation, with appropriate chemical reagents compatible with the mineralogy and physical properties of the Bikilal iron oxide deposits. Fatty acids are used as a collector in anionic reverse flotation, and sodium silicate is used as a depressant of iron oxides, enhancing the recovery of iron ore and reducing the problems associated with cationic reverse flotation.

1.3. Objective of the Study

I. General Objective:

The main objective of this research is to improve the iron content of the low grade Bikilal iron ore deposit using anionic reverse flotation techniques to eliminate apatite, silica, alumina and other minor gangue minerals in order to increase the grade and recovery of iron so that it can be the higher concentrate feed for further processing methods.

II. Specific Objectives of the Project:

1. To study the most appropriate way of removing Phosphorus (Apatite) and quartz minerals from the valuable iron ore with a selective flotation process.

2. To identify the most important flotation reagents that can be go through the mineralogy of the iron ore type in terms of economic viability and environmental impact.

3. To determine the optimum size range for flotation process so that adequate results can be obtained with a good recovery and grade.

4. To give an additional insight for the coming researchers who are devoted on the flotation of iron ores to be the input for their researches, and in turn it can help to deliver the more complete and detail outcomes.

1.4. Scope and limitation of the project

This project work have been conducted in the Bikilal iron ore deposit area in Wollega, western Ethiopia .It mainly focuses on the separation of the iron ore by using reverse anionic flotation methods. The project work have included conducting different laboratory analysis (that means quantitative and qualitative) of the sample and selecting chemical reagents for the flotation techniques based on the laboratory results, performing flotation methods, interpreting the results and preparing the report according to the experimental results.

The limitations to perform these activities are: lack of tangible and realistic mineralogical data, lack of well-organized laboratory in the country, non- availability of chemical reagents and instability or social conflict around the deposit area so as to collect primary data. There are also financial limitations to accomplish all the data analysis even outside of the country.

1.5. Significance of the study

The main problem in the beneficiation of Bikilal iron ore is the presence of Phosphorus in the form of apatite and removing this mineral from the target mineral is a very critical issue. The removal of Phosphorus in the flotation process will be successfully done by anionic reverse flotation because in leaching process particularly in alkaline leaching, Phosphorus removing is ineffective due to the re-precipitation behavior of Phosphorus.

The present project tried to minimize the problem of losing valuable minerals during magnetic separation as well as gravity separation, reduce the environmental impacts that come as a result of using toxic and hazardous chemicals in the cationic reverse flotation. The selected processing method can optimize the product cost by removing the de-sliming steps, which are very expensive and complicated in the cationic flotation. Anionic reverse flotation has the advantages of phosphorus removal by reducing the above problems to obtain the product (Concentrate) with the acceptable level of Phosphorus (0.025%wt).

This research has also a great role to give an additional idea about how to produce a better iron concentrate with low amount of alumina, Silica and other gangues. Reverse flotation of silica can reduce the problems of high dosage of reagents in the process this results the optimization of overall product cost.

CHAPTER TWO- LITRATURE REVIEW

2.1. Previous Studies on the Area

Iron ores are deposited under various geological conditions. It occurs in basins of sedimentation with eroded deep seated intrusive and where deep tropical weathering prevails. The criteria for the selection of the most suitable beneficiation method for each application include a series of parameters; the most important among them is related to ore mineralogy. So, the understanding about constituent minerals is the key in evaluating the success of any mineral processing operation. Froth flotation of iron ores is one of the most widely used methods to upgrade iron ore into high grade concentrates. The flotation of iron ore was first investigated in America starting in 1931 (Iwasaki 1983, Uwadiale 1992). Over time, several different flotation routes have been devised. These routes can be divided into 3 categories: direct flotation, reverse anionic flotation, and reverse cationic flotation. Direct flotation refers to processes which float the iron ore into the froth product, whereas reverse flotation processes float the gangue materials into the froth to be discarded. Direct flotation and reverse anionic flotation were initially developed by Hanna Mining and American Cyanamid in the 1930s and 1940s. These methods were implemented in Minnesota and Michigan concentrators by the 1950s (Filippov, Severov and Filippova, 2014). Cationic reverse flotation was simultaneously being developed by the U.S. Bureau of Mines, which eventually became the primary flotation route for the United States and other countries in the west (Filippov, Severov and Filippova, 2014).

2.2. Principles of Flotation

Foam flotation utilizes the differences in the physico-chemical surface properties of minerals to separate them. By treating the minerals with reagents, differences in surface properties between minerals in flotation slurries become apparent, and air bubbles can attach to particles and lift them to the surface for flotation to occur. This method can be used for relatively fine particles, as particles that are too large will not adhere to air bubbles due to the adhesion force being less than the weight of the particles. In the flotation process, a suspension of fine iron ore in water is treated with a small amount of flotation agent, which modifies the surface of iron oxide or the main constituent of gangue (usually silica). These particles adhere to air bubbles and are carried to the surface where they are removed as bubbles. Iron ore flotation can be performed using two methods: the direct method and the reverse method. In the former, iron oxide is floated using anionic reagents such as petroleum sulfonates and fatty acids, which have negatively charged ionic heads attached to a long organic "tail". Conversely, in the reverse method, the basicity is kept high (pH 11-12) and other organic dampening agents such as starch or synthetic organic polymers are added to prevent cationic reagents (positively charged heads) from depleting iron-bearing minerals, and silica is floated instead. Although grinding of the ore is not strictly a part of flotation, it is important for optimum flotation results that the valuable minerals be completely separated from the waste rock (gangue) and from one another in the grinding step. However, this is often not economically feasible, and even when complete separation is attained, other complicating factors may arise. A flotation machine is used to create a rising current of air bubbles by mechanically agitating the ore pulp, introducing air under pressure, or both.

2.3. Direct Flotation of Iron Ore

The first flotation route used in industry was direct flotation in an alkaline medium. Soda ash was used to adjust the pH value to between 9–10, and fatty acids were used as collectors. This process was used starting in the 1950s by the Humbolt concentrator in America and the Donganshan concentrator in China (Houot 1983; Iwasaki 1983). This flotation route is quite simple and can be made effective without de-sliming. At high pH values however, the surfaces of the common gangue minerals and the iron ore behave quite similarly, limiting separation power. A characteristic example of direct flotation in alkaline media can be found in the Republic Mine. Nowadays several flotation media have been used for direct flotation processes with iron ore, including alkaline media, acidic media, dispersing media, selectively flocculating media, and most combinations of the previous either in series or (where possible) simultaneously. Direct flotation is not well-suited for some gangue materials – in particular, sulfur, phosphorous, fluorine, and carbonate gangue materials behave too similarly to iron ore in direct flotation to achieve good separations (Rao 2004; Uwadiale 1992).

2.4. Reverse Flotation of Iron Ore

Reverse flotation is presently the most popular methodology for concentrating iron ore. In reverse flotation the goal is to cause the gangue materials to move up into the froth, which is precisely the opposite of direct flotation (Ma 2012; Filippov, Severov and Filippova 2014).The performance of the reverse flotation of iron ores is largely governed by the interactions between collectors and minerals, which are complex. A common task in the reverse flotation of iron ores is to separate quartz from iron oxides as quartz is often the major gangue mineral in iron ores. In some iron ores, removal of non-quartz gangue minerals is also essential, which has been the subject of several studies (Luo, X.; Wang, Y.; Wen, S.; Ma, M.; Sun, C.; Yin, W.; Ma, Y. 2016). These non-quartz gangue minerals include alumina-containing minerals, phosphorus-containing minerals, iron-bearing carbonates, and iron-bearing silicates. The presence of these gangue minerals not only complicates the flotation system, but also imposes a detrimental effect on downstream steel-making processes. The surface properties of these gangue minerals are different from that of quartz, so they are often treated separately in the flotation process. Furthermore, given that apatite is a sparingly soluble mineral, it has been reported that its dissolution accounts for the floatability of apatite. Finkelstein, N.P. (1989**)**, argued that, immediately after leaving the mineral lattice, Ca^{2+} ions interact with the Oleate molecules. The formed calcium Oleate then precipitates and renders the mineral surface hydrophobic. Horta, et al. (2016), found that the apatite more dissolvable in water could provide more Ca^{2+} ions and exhibit better floatability. It was concluded that, in general igneous apatite (e.g., fluorapatite, $Ca_{10}(PO_4)_6F_2$) bears better solubility than sedimentary apatite (e.g.,carbonate-fluorapatite, $(Ca,Na,Mg)_{10}(PO_4,CO_3)_6(F,OH)_2$) (Horta, et al.2016). Flotation of apatite using cationic collectors was also studied. Reverse flotation is broadly divided into two categories depending on the choice of collector: cationic reverse flotation and anionic reverse flotation.

2.5. Flotation Reagents

For effective flotation, generally three types of flotation reagents are used. They are collectors, Frother, and modifiers. In addition to surfactants, a number of inorganic compounds are included. These are either used as pH regulators (lime, soda ash, sulphuric acid, caustic soda, etc.), activators (metal ions), or depressants (soluble silicates and sulfides and organic polymeric compounds as shown in Table 3 below.

Table 1 Reverse flotation types and reagent schemes applicable for iron ore beneficiation process.

Flotation Reagents	**Flotation Types**	
	Anionic reverse flotation	Cationic reverse flotation
Collectors	Fatty acids, Oleic acid, line oleic acid, Petroleum sulphonate, Carboxylic acid , Lauryl acid, acintol (tall oil C = 6-18), Sodium petroleum sulphonate.	Amine Alcohols, Fatty amines, Ether amines, mono amines di amines, Dodecyl amines (DDA)…
Frother	Pine oil, Aliphatic Alcohols, Propylene glycols, Cresylic acids(C =5-8), Methyl Isobutyl Carbonyl (MIBC).	Ether amines have both collecting and frothing properties, Non-ionic alcohols like: Iso-alcohols, dodecanol alcohols.
Activators	Lime (CaO), Calcium chloride ($CaCl_2$), Magnesium chloride ($MgCl_2$), Calcium oxide, and Alkylsulfonic acid, Hydroxamates, Sulfuric acid and sodium or potassium salts and their soaps.	Iso-alcohols, dodecanol alcohols
PH Regulators	Sodium Hydroxide (NaOH), Sulfuric acid (H_2SO_4).	Lime(CaO),Slaked lime $Ca(OH)_2$, Soda ash, Caustic soda, Sulfuric acid (H_2SO_4)
Depressants	Sodium silicate ($NaSiO_4$), Starch, Lime, Dextrin, Carboxyl Methyl Cellulose (CMC).	Corn starch, Tapioca starch Humic acid, Carboxyl Methyl Cellulose (CMC).

CHAPTER THREE- MATERIALS AND METHODS

3.1. Secondary Data Collection

Different literatures, Journals , articles published and unpublished reports have been collected from different sources and reviewed to have a detail understanding about the mineralogy of the rock that will be examined, the flotation principles and required materials, The factors that affect the process of flotation. The mineralogical data obtained from the Atomic Absorption Spectroscopy (AAS), Atomic Emission Spectroscopy (AES) and Inductively Coupled Plasma -Mass Spectrometry (ICP-MS) or X-Ray Fluorescence (XRF).The concentrate minerals can be determined by (AAS) or (AES),Whereas the elemental identities(qualitative data) can be determined using (ICP-MS) or (XRF) analytical techniques.

3.2. Materials and Resources Needed

The method that has been used to upgrade the Bikilal iron ore is reverse flotation specifically anionic reverse flotation. The low grade iron ore dominantly magnetite (23.3%) followed by ilmenite and hematite ore samples with a total iron grade of 41% was taken from Ethiopian Geological Institute laboratory store which brought from Bikilal iron deposit area in Wollega Western Ethiopia. The grain size of magnetite is 0.1-1.5mm so the recovery of the target mineral will depends on the final size of the concentrate after size reduction. The size of the concentrate should not be greater than 75µm for the effective flotation process. Slimes should not be less than 20µm to obtain a good liberated particle size in the flotation without using very complicated and economically non-viable steps such as de-sliming (agglomeration, flocculation and dispersion).

Calcium or Magnesium ions are used to activate the naturally negatively charged silica (quartz) as an activator. Lime (CaO) can be used as an alternative activator and has an advantage of activating siliceous minerals while allowing for strong depression of the iron-bearing minerals in highly alkaline conditions. The slurry and the reagents mixed for 20 minutes. The concentration of the slurry should be in the range 25-64%wt solids. It is more attractive to use a tall oil composed of a mixture of oleic acid and linoleic acid. Moreover, the froth is controlled by the addition of a Frother. The flotation process has done by mechanical flotation cell, which is one of the most widely used flotation equipment. Anionic reverse flotation is selected for the Bikilal iron ore because of the mineralogy of the ore and grain size. This process does not necessitates the de-sliming stages, uses environmentally friendly and easily available chemical reagents. The primary advantages of anionic reverse flotation are a decreased sensitivity to slimes, and a lower collector cost as the fatty acids are a major waste product from the paper industry for strong depression of the iron-bearing minerals in highly alkaline conditions. Anionic reverse flotation can also be used to float apatite gangue materials, to lower the phosphorous content of the resulting concentrate.

3.3. Methodology of the Experiment

For the present project work the following chemical reagents has been used to get the required output.

- First the sample was crushed by a jaw crusher and a secondary cone crusher then grinded by a ball mill adjusted at 250 rpm for 25-30 minutes so that the specified size reduction have been obtained by sieving with different mesh sizes.
- The main target was to get sufficient amount of particle size classes under 75µm and under 63µm.
- 500g of each particle size samples have prepared for flotation process.
- After preparing the samples with these two size ranges flotation process performed with four rounds for each particle sizes that mean 250g for one test.
- First a sieved sample mixed with water to have 45% solid solution.
- Only one step batch flotation was carried out with mechanical flotation cell adjusted at the agitation speed of 2200rpm.
- The total residence time for a flotation of one feed was 20 minute and chemical reagents added with 3 minute intervals.
- 1ml.ethanol was used as a Frother and 1ml. fatty acids (Oleic acid) used as a collector as it is one of the most commonly used and effective anionic collector.
- Modifiers such as activators, PH regulators and depressants have also used. Calcium ions (Ca^+) from calcium chloride activate the silica and other gangue minerals to float it and skimmed off from the cell. Sodium hydroxide added to control the PH and lime is used as a depressant for iron bearing minerals.

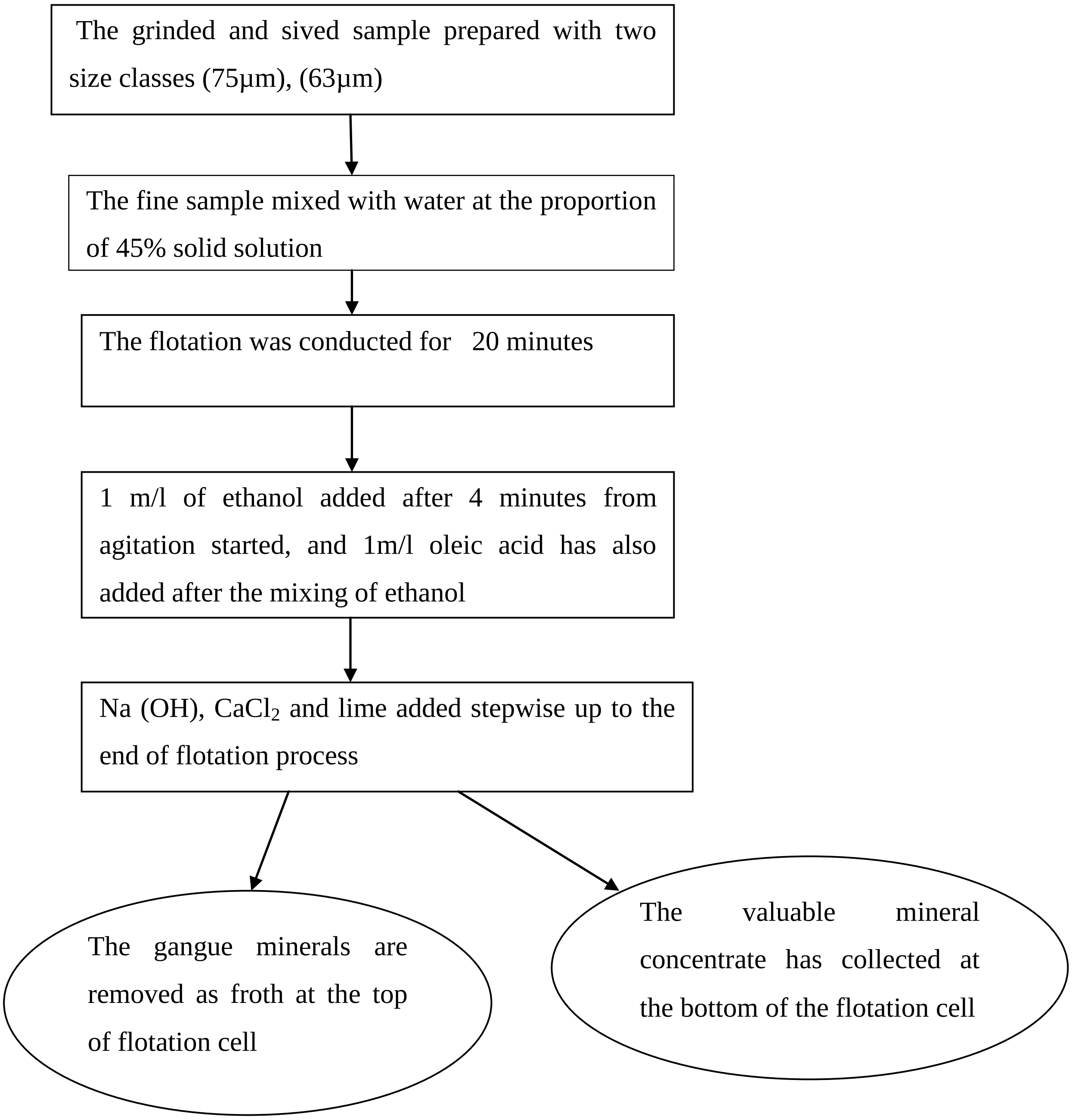

Figure 1 Schematic flotation process activities of the project.

CHAPTER FOUR- THE ORE CHARACTERSTICS

4.1. Mineralogy of Bikilal Iron Ore Deposit

Ultra-mafic rocks are the host for several metallic and industrial minerals including chromites, Fe, Ni, Cu sulphides, and PGM (Scott et al. 2000). Magnetite- ilmenite ore bodies in Bikilal deposit are genetically and spatially related with basic-ultra basic gabbroic intrusive rocks. Bikilal iron ore deposit consists of minerals like magnetite, ilmenite, hematite, sulphides, apatite and gold. From these the principal ore minerals are magnetite and ilmenite. Based on the ore microscopic studies, the ore mineral assemblages consists of 5-12% ilmenite, 20-25% of magnetite, >10% of hematite, and small amount of sulfides (pyrite, pyrrhotite, and chalcopyrite, pentlandite) in average. In this ore body hematization and meritization of both magnetite and ilmenite is very common (Solomon, R.2013).The total tonnage of Bikilal iron ore deposit is calculated to by 57,796,080 ton of iron with 23.29% magnetite from 41 % total iron, 0.36 P_2O_5 , 0.77% Sulfur, 16.72% Titanium oxide and 0.24% Vanadium oxide(Zewdneh, T. 1990). In the Bikilal Ore bodies titanium occurs mainly in ilmenite ($FeTiO_3$).It has also been found in rutile (TiO_2) and as Exsolution lamellae in the magnetite lattice. According to the investigation carried out by the Ethio-Korean iron exploration project the magnetic separation tests shows that most of the ilmenite appears as free grains and only a minor part occurs as thin Exsolution lamellae in the magnetite.

Table 2 Chemical formula and abundance of iron ores in the earth crust.

No.	Iron Ore Types	Chemical Formula	Iron Content (%)
1	Hematite	Fe_2O_3	69.9% Fe
2	Magnetite	Fe_3O_4	72.4%Fe
3	Limonite	$FeO(OH).n(H_2O)$	55.0%Fe
4	Goethite	$(FeO(OH)$	62.9%Fe

4.2. Phosphate occurrences and Removal from Bikilal Iron Ore

The Occurrence of phosphorus in iron ore depends on the type of iron minerals. In magnetite phosphorus is often found in the form of discrete phosphorous containing minerals which are referred to as apatite ($Ca_5(PO_4)_3(Cl,F,OH)$). It is the most frequent and important constituent of the class of phosphate.

This type of phosphorus containing minerals can be removed by physical methods such us flotation. For example the magnetite ore at LKAB (Luossavaara-Kiirunavaara AB) in Kiruna, Sweden contains about 1wt% of Phosphorus, which is removed by magnetic separation in combination with flotation. Besides apatite, various other phosphates may be found in deposits of iron ore. Phosphorus is spread on the edge of mineral particles of iron oxide and embedded in quartz or carbonate minerals and a small amount is present in the iron mineral grid.

The acceptable levels of Phosphorous in hot metal varies from 0.08 to 0.14%, however the Phosphorous content of <0.08 wt% is most desirable (Cheng, 1999).The distribution of Phosphorus in Goethite crystals prevents the effective use of physical beneficiation methods. This type of phosphorus containing minerals can be rejected by chemical methods.

There are many citations in the literature on the reduction of phosphorus in iron ore through anionic flotation, using fatty acids as collector and Sodium silicates as a depressant of iron oxides, when the main phosphorus-bearing mineral is apatite (Ranjibar, 2002; Siirak and Hanock, 1990; Paytt, 1990).

In anionic flotation of apatite, fatty acids (e.g., oleic acids) and their soaps are primarily used as collector, often in conjunction with hydrocarbon supplements (e.g., kerosene and fuel oil), to reduce the collector consumption. Su, et al. (1998), separated apatite from magnetite with a modified fatty acid collector (Atrac-1562) at pH 8.5–9.0 and at a pulp temperature of approximately $20C^0$. Kou, et al. (2010),carried out phosphate flotation using a refined tall oil fatty acid at a dosage of 0.45 kg/t at pH 10 with a 9:8 (by mass) concentration ratio of fatty acid to diesel. Cao, et al. (2015), employed a mixed collector (i.e., 54 wt. % oleic acid, 46 wt. % linoleic) for apatite flotation at pH 9.5 and at a pulp temperature of approximately $23C^0$. The interaction between the carboxyl group of the fatty acids and $Ca(OH)^+$ ions exposed at the mineral surface was considered the mechanism of attaining the flotation of the apatite (Horta, et al. 2016), which is the same as the adsorption mechanism of oleic acid with activated quartz. Furthermore, given that apatite is a sparingly soluble mineral, it has been reported that its dissolution accounts for the floatability of apatite. Finkelstein (1989), argued that, immediately after leaving the mineral lattice, Ca^{2+} ions interact with the Oleate molecules. The formed calcium Oleate then precipitates and renders the mineral surface hydrophobic. Horta, et al. (2016), also found that the apatite more dissolvable in water could provide more Ca^{2+} ions and exhibit better floatability.

Phosphorus is a harmful element in steel-making, causing product defects such as increased hardness and brittleness and decreased ductility (Nunes, A.P.L.; Peres, A.E.C.; De Araujo, A.C.; Valadão, G.E.S.2011).The amount of apatite deposit in the target area, which is the Bikilal iron ore deposit, is 0.36% which is beyond the acceptable grade for iron and steel production.

CHAPTER FIVE- RESULTS AND DISCUSSION

5.1. The Outcome of the Experimental Analysis

The results of the present study of flotation process yield the required amount of slurry that sinks at the bottom of the flotation cell. The amount of froth (Foam) formed at the top of the flotation cell was very stable that can carry the weight of the hydrophobic gangues and the size of bubbles were as sufficient as expected from the experiment with the average diameter of about 2mm (Figure 2).

Figure 2 Shows the froth size and stability during reverse flotation process.

The total mass of feed for the flotation under 75µm was 500g and from these amount 250g dry concentrate obtained under the flotation cell. The amount of slurry in -63µm was less than that of -75µm due to finer particle sizes under 63µm have been removed as slimes with gangues Figure 13 (left the concentrate under -75 µm) and (right the concentrate under-63µm).

Figure 3 Iron concentrate deposited at the bottom of flotation cell after anionic reverse flotation carried out.

Based on these data the material balance of the system can be calculated by the rule of conservation of mass.

Mass balance: $F = C+T$... (Eq.1)

F = mass of feed

C = mass of Concentrate

T = mass of tailing

Component balance: $Ff = Cc + Tt$ f = Feed assay

c = concentrate assay

t = tailing assay

From this mass of tailing also calculated to be

$$500gF = 250gC + T$$

$$T = 500gF - 250gC$$

Therefore, mass of tailing = 250g

The mass of the feed under 63µm has been also 500g and the concentrate was 215g

$$F = C + T$$

Feed = 500g, Concentrate = 215g then,

Tailing = 500gF-215gC = 285g

5.2. The complete silicate (Major oxide) analysis results of the ore sample

Two samples has been selected for Atomic Absorption Analysis (AAS) to determine the composition of the ore sample as well as to know the percentage of the target mineral and the gangue minerals associated with iron oxides.

The average grade of the ore analysis result is 41.2% and that of P_2O_5 is 0.34% that is not much different from the Phosphorous values mentioned in several literatures. Based on previous studies of the Bikilal iron ores taken from the core samples of the borehole the grade of iron is about 41% and the composition of P_2O_5 is 0.6%. The compositional values of Aluminum and silica have also a significant effect on the quality of iron for steel production. According to the result from (AAS) the percentage of Alumina (Al_2O_3) and silica (SiO_2) in the ore sample was 8.5% and 36.8% respectively as indicated figure below. This indicates that the amount of these minerals in the ore imposes the value or quality of the target mineral iron.

Table 3 The chemical analysis results of the natural Bikilal iron ore.

Mineral types (%)	**Composition**								
	Fe	TiO_2	P_2O_5	SiO_2	Al_2O_3	MgO	CaO	Na_2O	K_2O
BIO/01	41.8	9.7	0.62	35.79	6.31	3.09	1.3	0.41	0.04
BI0/02	40.6	5.3	0.85	37.8	10.7	2.3	0.9	0.15	1.05
Average	41.2	7.5	0.73	36.79	8.5	2.69	1.1	0.28	0.54

Note: BIO Bikilal Iron Ore

5.3. The complete silicate (Major oxide) analysis results of the concentrate

The final product of flotation process or the concentrate has been collected with small plastic bags in two main size ranges of under 75µm and under 63µm so as to determine that which size range can yield a good result in terms of grade and recovery as revealed in table 9.These concentrate analyzed with (AAS) method and the result shows that there is a high increment of the valuable mineral which is Fe_2O_3. The result of flotation concentrate shows a significant reduction of gangues especially the detrimental elements such as Phosphorous, Aluminum and Silica, Which are the main focuses of the project work. Although the two size classes of the result indicates the enrichment of iron ore there is a small difference of outputs based on size differences. In summary the iron grade less than 63micron is a little bit better than that of less than 75 micron. Furthermore the percentage of major gangue minerals is reduced in -63 micron than in -75 micron. Therefore the optimum liberation size or grinding size for a specified ore under investigation is less than 63µm to obtain a good result.

Table 4 The chemical analysis results of the concentrate for Bikilal iron ore after flotation.

Concentrate sample types (%)	Composition												
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI	Total
BIC/75/1	4.3	2.34	86.59	0.52	1.75	0.16	0.08	0.70	0.18	2.94	0.04	0.65	100
BIC/75/2	5.20	1.9	85.22	0.6	1.69	0.07	0.10	0.46	0.30	4.32	0.08	0.44	100
BIC/75/3	4.10	2.06	87.08	0.47	1.82	0.31	0.09	0.52	0.24	3.17	0.06	0.53	100
BIC/63/1	3.03	2.05	88.65	0.64	1.71	0.14	0.33	0.42	0.20	2.46	0.03	0.67	100
BIC/63/2	3.41	1.44	89.58	0.50	1.18	0.15	0.04	0.75	0.16	2.50	0.01	0.82	100
BIC/63/3	3.7	1.82	87.85	0.37	1.41	0.12	0.05	0.66	0.13	3.27	0.02	0.95	100

Note: BIC Bikilal Iron Concentrate

LOI Loss on ignition

From the above table it is possible to calculate the chemical composition (%) of Fe from Fe_2O_3 by the formula,

$$\%\text{of Fe by mass} = \frac{\text{Molar mass of Fe}*\text{Number of mole of Fe}}{\text{Molecular mass of the Compound Fe2 O3}} * 100$$

Therefore % of Fe = $\frac{55.85*2}{(55.85*2)+(16*3)} * 100$

This gives 69.94 % Fe approximately 70% Fe in Fe_2O_3 and 30.0%O

Based on the analytical values above in table 9 it is possible to determine the grade of the economic mineral and also the percentages of major unwanted minerals present in the sample. As shown in table 10 the amount of such gangue minerals are below the internationally acceptable values for iron and steel production purposes since these unwanted materials are the main deleterious elements in iron ore processing methods.

The grade of iron in the concentrate for the sample code BIC/75/1 is then 70*86.59/100 = 61.0%Fe and for BIC/75/2 is 60.0

Similar calculations can be carried out to get all the sample types examined and all the numerical data are listed in tabular form in table 10.The average grade of iron from 3 round experiments for each particle size range is 61.4%Fe

Table 5 Shows the iron grade with the corresponding major gangue minerals.

Concentrate sample types (%)	Major mineral compositions			
	Fe	P	Si	Al
BIC/75/1	61.0	0.08	2.7	1.2
BIC/75/2	60.0	0.13	2.02	0.98
BIC/75/3	61.0	0.10	2.6	1.07
BIC/63/1	62.1	0.08	2.0	1.32
BIC/63/2	63.0	0.07	2.4	0.75
BIC/63/3	61.5	0.05	2.3	0.95
Average Grade	61.40	0.085	2.4	1.04

From the table the average grade of iron in -75 micron is 60.6% whereas in -63micron the average grade is 62.2%, which is somehow better result than in -75 micron sieve size products. According to the available parameters the recovery of iron can be calculated as

Recovery, R = 100Cc%/Ff

Or 100c (f-t) %/f(c-t)

F = mass of feed

C = mass of Concentrate

T = mass of tailing

f = Feed assay

c = concentrate assay

t = tailing assay (gram)

$$R\ (75/1\mu m) = \frac{100*250kg*0.61}{500g*0.41}$$

R = 74.4

$$R\ (-63/1\mu m) = \frac{100*215g*0.62}{250g*0.41}$$

R = 65.6%

Similar calculations can be performed to get all recoveries for each experimental runs.

Table 6 Iron grade and recovery from the concentrate after flotation has conducted.

Run of Experiment	Fe Grade (%)	Fe Recovery (%)
BIC/75/1	61.0	74.4
BIC/75/2	60.0	73.1
BIC/75/3	61.1	74.4
BIC/63/1	62.1	65.6
BIC/63/2	63.0	66.1
BIC/63/3	61.4	65

From the above table it can be understood that the grade and recovery has inverse relationship as the higher the grade the lower the recovery and the vice versa. Figure 15 below shows this opposite relation of the grade and recovery.

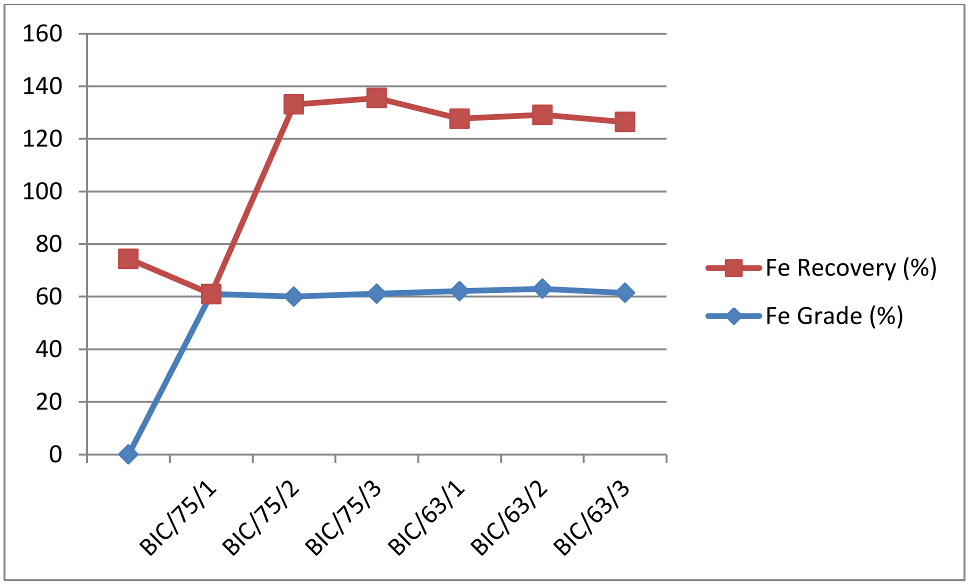

Figure 4 The relation between the grade and recovery of iron concentrate.

The chemical analysis results of the tailings have been also indicates the presence of a variety of gangue minerals in the ore with different compositional amounts. As listed in table 12 silica is a major gangue mineral followed by Alumina with a compositional values ranging from 44.21-45.48% and 15.01-18.07% respectively.

Table 7 Complete silicate (major oxide) analysis results of the flotation tailings from Bikilal iron ore.

Tailing sample types (%)	Composition												
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O	LOI	Total Weight (%)
BIT/75/1	45.48	18.07	16.6	10.25	5.73	0.88	<0.01	<0.01	0.87	1.19	0.49	0.80	100
BIT/75/2	43.50	17.22	17.31	12.32	6.10	0.76	<0.02	<0.01	0.82	1.24	0.50	0.72	100
BIT/63/1	43.34	15.52	15.36	10.20	10.48	1.34	<0.02	0.10	0.80	1.33	0.28	1.53	100
BIT/63/2	44.21	15.01	14.65	11.11	10.79	0.93	<0.01	0.12	0.65	1.10	0.36	1.13	100

Note: BIT   Bikilal Iron Tailing

5.4. Conclusion and Recommendations

5.4.1. Conclusion

From this Project work it is possible to deduce that the ultimate goal of the study has been achieved by the selected beneficiation method and the procedures used to perform all the entire activities. The chemical reagents chosen to do this research has also assured that the Bikilal low grade iron ore could be improved with fatty acids used as a collector, calcium chloride as an activator of silica to change the naturally negative charge silica to an activated positively silica so that it can be absorbed by a negatively charged anionic collector (Oleic acid) and it moves up from the cell to remove as a major waste mineral. Sodium hydroxide has used as a PH control to set in the range (11-12) for effective flotation performance. According to the chemical analysis of the upgraded iron ore there is a promising result with an iron grade of (60-63%) and the recoveries (65-74.4%). Even though the result obtained from this project is economically viable to mine and process it can be improved more than this outcomes because the sample taken to do this paper is from the Geological Survey of Ethiopia which is stored for many years almost for

18 years so there might be some contamination as a result affects the iron contents and the recoveries as well. Due to this factors the ore sample that is going to be examined should be a fresh sample and have to be taken onsite so that a pure and representative samples can be collected. In general from the present study it can be concluded that the major detrimental elements such as P, Al and Si has been reduced to 0.085, 1.04 and 2.5 respectively, which is below the allowable amount for iron and steel production.

5.4.2. Recommendations

- The present project work gives a promising result to exploit the potential resource of the country so as to promote the economy of the country. However further studies on how to change this resource into a useable product form is recommended.
- There is a huge gap to study a detailed and well recognized work on Bikilal iron ore deposit area as well as in the country level; therefore a wider and scientific approach should be done to clearly understand the processing techniques and proper management of waste mineral disposals
- This project work depends on the major oxide chemical analysis (AAS) method to obtain the above results, but this is not enough to confidently deduce about the results, so additional Methods such as x-ray defractometry (XRD), scanning electron microscope (SEM) and X-ray fluorescence (XRF) methods are recommended to fully understand the qualitative and quantitative mineral compositions.
- In Bikilal iron ore deposit there are many associated major minerals and rare earth elements including gold metal. So in addition to Fe metal there is a high possibility to produce other economic minerals as a bi-product like Phosphorous from phosphate, Aluminum from Alumina and gold and other minor minerals. Therefore further investigations regarding to the processing of this additional minerals is essential to encourage the growth of the mining industry.

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Appendices

Appendix 1

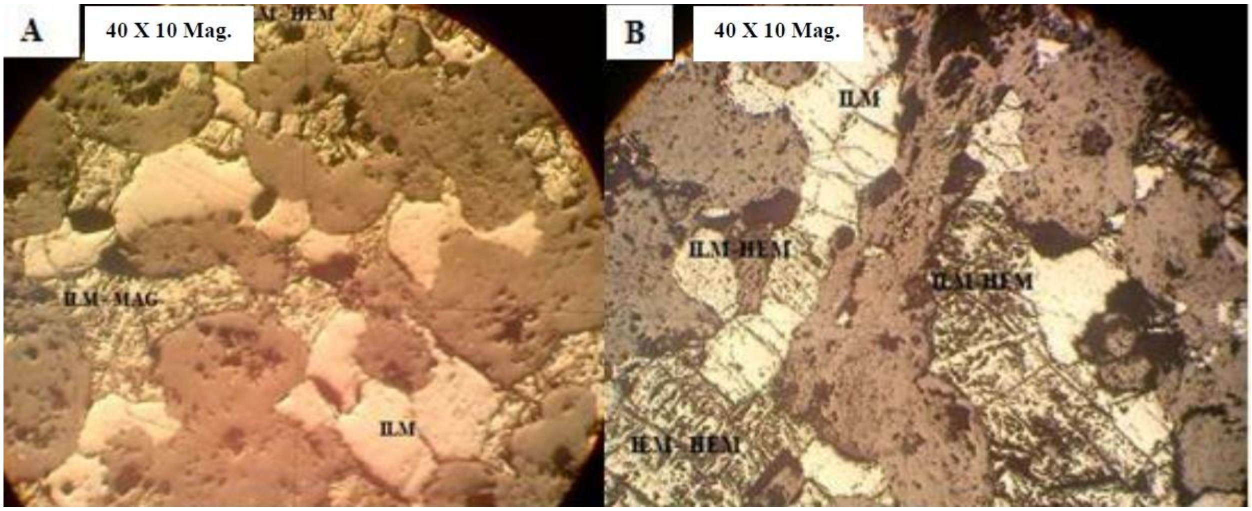

Figure 4.1.Photomicrographs of magnetite, pyrite and oxidation textures of ilmenite (A), Exsolution lamellae of ilmenite in magnetite oxidation (B).

Appendix 2

Table 4.1. Magnetite separation test analysis of ore, concentrate and tailings according to Zewdneh, T. (1990).

Mineral types (%)	**Composition**										
	Fe	TiO_2	V_2O_3	S	P_2O_5	SiO_2	Al_2O_3	MgO	CaO	Na_2O	K_2O
Ore	40.7	15.9	0.18	0.80	0.35	12.5	7.0	5.1	2.4	0.45	0.45
Concentrate	60.0	5.5	0.38	0.85	0.20	3.8	3.7	1.8	0.5	0.15	0.05
Tailing	30.0	21.9	0.07	0.77	0.43	16.3	9.1	6.6	3.4	0.62	0.22