Addis Ababa University
አዲስ፡አበባ፡ዩኒቨርሲቲ
SEEK WISDOM, ELEVATE YOUR INTELLECT AND SERVE HUMANITY !

ADDIS ABABA UNIVERSITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

CENTER FOR ETHIO-MINES DEVELOPMENT

Masters in Mineral Engineering

REVERSE ANIONIC FLOTATION FOR POTENTIAL BENEFICIATION OF SEKOTA IRON ORE, NORTHERN ETHIOPIA

BY

TAREKEGN DURE TESSEMA

A PROJECT SUBMITTED TO CENTER FOR ETHIO-MINES DEVELOPMENT IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF ENGINEERING IN MINERAL ENGINEERING

September, 2023

Addis ababa, Ethiopia

ADDIS ABABA UNIVERSITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

CENTER FOR ETHIO-MINES DEVELOPMENT

Masters in Mineral Engineering

REVERSE ANIONIC FLOTATION FOR POTENTIAL BENEFICIATION OF SEKOTA IRON ORE, NORTHERN ETHIOPIA

BY

TAREKEGN DURE TESSEMA

ADVISORS: ABUBEKER YIMAM (Dr.)

A PROJECT SUBMITTED TO CENTER FOR ETHIO-MINES DEVELOPMENT IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF ENGINEERING IN MINERAL ENGINEERING

September, 2023

Addis Ababa, Ethiopia

ADDIS ABABA UNIVERSITY

ADDIS ABABA INSTITUTE OF TECHNOLOGY

CENTER FOR ETHIO-MINES DEVELOPMENT
Masters in Mineral Engineering

REVERSE ANIONIC FLOTATION FOR POTENTIAL BENEFICIATION OF SEKOTA IRON ORE, NORTHERN ETHIOPIA

BY
TAREKEGN DURE TESSEMA

Approved by

Dr. Birhanu Asefa Signature………………..Date……………
Center for Ethio-Mines Director

Dr. Abubeker Yimam Signature………………..Date…………….
Advisor

Dr. Mulugeta Sisay Signature………………..Date……………
Examiner

DECLARATION

I declare that, this project work is my own original work under the supervision of Dr. Abubeker Yimam, Addis Ababa University, Center for Ethio-Mines Development, in the year 2023. I also declare that this work has not been submitted in any form for another degree or diploma at any university or other institution. Data used from the published and unpublished work of others has been appropriately acknowledged.

Tarekegn Dure Tessema Signature………………Date……………….

This thesis has been submitted for examination with my approval as University Advisor.

Dr. Abubeker Yimam (Advisor) Signature………………Date……………….

Acknowledgements

First and foremost, I would like to thanks for Ministry of Mines who facilitates the opportunity to attend my master's degree at Addis Ababa University, Addis Ababa institute of technology. I would also want to thank my advisors Dr. Abubeker Yimam for their close suggestion, comment and support.

I acknowledge with grateful thanks the contributions of Mr. Aman Yismaw (senior geologist) for providing me chemical, mineralogical and particle size distribution analysis data. And also Temesgen Demise for their help throughout the laboratory experiment work and invaluable friendship.

I would like to thank Kirubel Shegaw, for giving me a laptop computer.

Abstract

In this project study, hematite ore sample obtained from Sekota area, Northern Ethiopia was used. Main objective of the study is to obtain iron concentrate at possible maximum grade and recovery by using reverse anionic flotation beneficiation method. The effect of oleic acid collector dosage and sieve size fractions were studied and optimized. The chemical and mineralogical characterization of the ore was also determined. Results of chemical analysis using atomic absorption spectroscopy (AAS) showed that the iron ore contains 65.12% Fe_2O_3 (45.57% Fe). Major gangue minerals were SiO_2 (20.62%) and Al_2O_3 (8.86%) while all other minerals were reported at <1%. Mineralogical descriptions using the X-ray diffraction (XRD) and QEMSCAN BMA showed predominantly hematite and goethite with quartz, kaolinite and clay as gangue minerals while others P and S are in lower amounts. The concentrate 62.96% Fe_2O_3 with highest grade of 44.07% Fe was obtained with 43.4% Fe recovery in the feed size fraction (−63 µm) adding 3g/t of oleic acid collector. Based on the results obtained from the flotation tests conducted, Sekota iron ore at a sieve size of -75µm and -63µm were able to enhancing both grade and recovery.

Keywords: Beneficiation, concentration, tailing, grade, recovery

Contents **Page**

List of Figures

List of Tables

List of abbreviations

AAS	Atomic Absorption Spectrometry
BIF	Banded Iron Formation
BMA	Bulk Modal Analysis
CMC	Carboxymethylcellulose
DMS	Dense Medium Separation
LOI	Loss on Ignition
QEMSCAN	Quantitative Evaluation of Minerals by Scanning Electron Microscopy
SEM	Scanning Electron Micrograph
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
µm	Micrometer
g/t	Gram per tone

CHAPTER ONE

1. Introduction

1.1. Background

Iron is common elements on Earth. It is mainly found in hematite, magnetite and goethite from which metallic iron is extracted economically. Iron ore occurs in three types of deposit as primary ore in gabbroic intrusion, cracks and zones of crumpling known as magmatogene and also derived from pre-existing primarily sedimentary deposits is metamorphogenic (metamorphosed) or a secondary deposits formed as a result of the weathering of iron containing rock forming minerals called exogenic deposit (Iron Ores, 2013). Most of the world's important iron ore resources occur in banded iron formations (BIFs). Ethiopian iron ores are mostly basic intrusion-hosted Fe-Ti, banded iron formation (BIF) type occurrences and secondary laterite and/or gossan-related deposits (Solomon, 2009).

Approximately 2.4 Bt (billion tonnes) of iron ore was produced in the world 2020.While ore production occurs in 50 countries, the top five largest producers were Australia (900 Mt (tonnes)), Brazil (400 Mt), China (340 Mt), India (230 Mt) and Russia (95 Mt), accounting for 80% of world iron ore production (US Geological Survey, 2021). Ethiopia remains an importer of iron oxide products. Worldwide, the steel industry consumes approximately 98 % of the iron that is produced annually. It has a rapid depletion of the reserves, known as “direct shipping ores”. With the depleting reserves of high-grade iron ore (65-77 % of Fe), various iron ore beneficiation methods have been employed to process intermediate and low-grade iron ore in an attempt to meet the rapidly growing demand.

Magnetite (Fe_3O_4), hematite (Fe_2O_3), goethite (($FeO(OH)$), limonite ($FeO(OH)\cdot n(H2O)$)), siderite ($FeCO_3$), and pyrite (FeS_2) are commonly found iron minerals. Among these, magnetite, which can be easily concentrated. However, the majority of the iron resources are in the fully oxidized hematite form. As the magnetite ore grade continues to decrease, concentrating hematite and goethite will become necessary to meet global need. This situation is what drives the urgent need to upgrade these ores in order to increase the iron grade and eliminate gangue minerals. Hence, iron ores according on mineral composition, physical properties, character and degree of liberation of the iron-bearing minerals from the gangue are directed to beneficiation including gravitational, magnetic, and flotation processes or their combination.

Physical ore processes utilize different density, shape, magnetic susceptibility and hydrophobicity of ore and gangue minerals. Jigging, up-flow classification/hindered-bed settling, dense medium separation (DMS), shaking tables and spiraling; and magnetic separation are applicable in iron separation (Rezvanipour et al., 2018; Roy, 2009). Froth flotation, which uses the difference in physico-chemical properties, is the most effective method for upgrading iron concentrates (Fardis and Mehdi, 2017). Gravity separation techniques are widely used in mineral beneficiation practices for its low-cost, ease of operation, and eco-friendly nature. They are based on the differential settling velocities of the constituting particle of the ore.

The aim of upgrading iron bearing ore is to reduce the iron concentrate containing quartz, sulfur, phosphorous and aluminum less than 2%, 0.1%, 0.01%, and 2%, respectively. This needs increased direct reduction. The high alumina and silica affect blast furnace. High alumina cause viscous slag formation during smelting (Holmes and Lu, 2015; Rachappa et al., 2015: Fardis and Mehdi, 2017).

Currently, no iron ore extraction and beneficiation operation in Ethiopia to upgrade iron ores.

1.2. problem Statement

Ethiopia has million tons of iron deposit. However, these ores are low grade, high silicate content and disseminated; and studies on mineralogy, texture, grade and beneficiation are limited. The geological researches shows that the iron ore deposits in Sekota areas are low grades. This can't be utilized to produce iron and steel by conventional blast furnace technique or direct reduced iron (DRI) processes. Gangues can be removed prior to smelting process leads to better sinter product with higher blast furnace productivity and better quality of steel. Thus, beneficiating the low grade iron ore to improveing grade is required the aforementioned problems, due to produce an acceptable feed for steel making plant.

1.3. Objectives

1.3.1. Main Objective

The main goal of this project work is beneficiation of Sekota iron ore using reverse flotation to increase the content of iron minerals.

1.3.2. Specific Objectives

To achieve the general objective, the following specific objectives have to be fulfilled:

i. Determine chemical and mineralogy charecteristics of Sekota iron deposit;

ii. To determine the effect of oleic acid (collector) dosage and mineral particle size range ; and

iii. Identify the optimal conditions for the reverse flotation can be determined by taking into account both recovery and grade.

1.4. Significance of the study

Focus on the development and commercialization of mining sector, proper characterization of the deposit will help in exploration and encourage local and foreign investment. The industrialize advantages of this process are significant. So, the result used as a reference to improve the concentrate upgrade of Iron Company in understanding more about flotation separation using anionic reverse flotation. To give additional information for other local processing company to find out the appropriate method in flotation separation. In academic communities of Universities, the outcome of this study can serve as background for next studies in the area.

1.5. Scope of the study

The scope of this research work involves collect and prepare representative sample; laboratory analysis to determine the chemical and mineralogical analysis and then determine the concentration of the ore using froth flotation method. This information will be gathered using the following tools: AAS, XRD and XRF. The research is based in a lab set up. Available geological data as well as appropriate literature on iron ore processing were collated and reviewed. The Information search made use of the following databases and web search engines: Science direct, Sci-hub, Google and Yahoo.

1.6. Limitation of the study

The following limitations are barrier to the study:

- Colour images are not available for interpretation and identification of mineral.
- Scanning electron micrograph (SEM) analysis wasn’t employed for beneficiating ore samples.
- Limited gram (minimum 150 g) of flotation concentrate.
- Time period is short.

CHAPTER TWO

2. Literature review

2.1. Fundamentals of iron

Iron is a dark and silver-grey coloured metal in group VIII, the first row transition series. It is most abundent element (after oxygen, silicon, and aluminum) in the Earth's crust, forming much of Earth's core. It has an atomic number of 26, mass of 55.85 g/mol and density of 7.874g/cm^3 at 20 °C. Iron, along with its generic products, utilized the various sectors of the world's economy (Fig.1). Iron accounts for approximately 95% of all metals used by modern industrial society. Almost all (98%) of the world's iron make steel (GSA, 2009). Steel is the best known alloy of iron. Most of steel consumptions are in real-estate 47%, machinery 19%, infrastructure 16%, transportation 8% (Basson, 2014). However, iron forms compounds such as iron chloride and nitrite, iron sulphate and iron carbonyl are used in ink production as industrial reagents, fungicide and as a catalyzer of chemical industries.

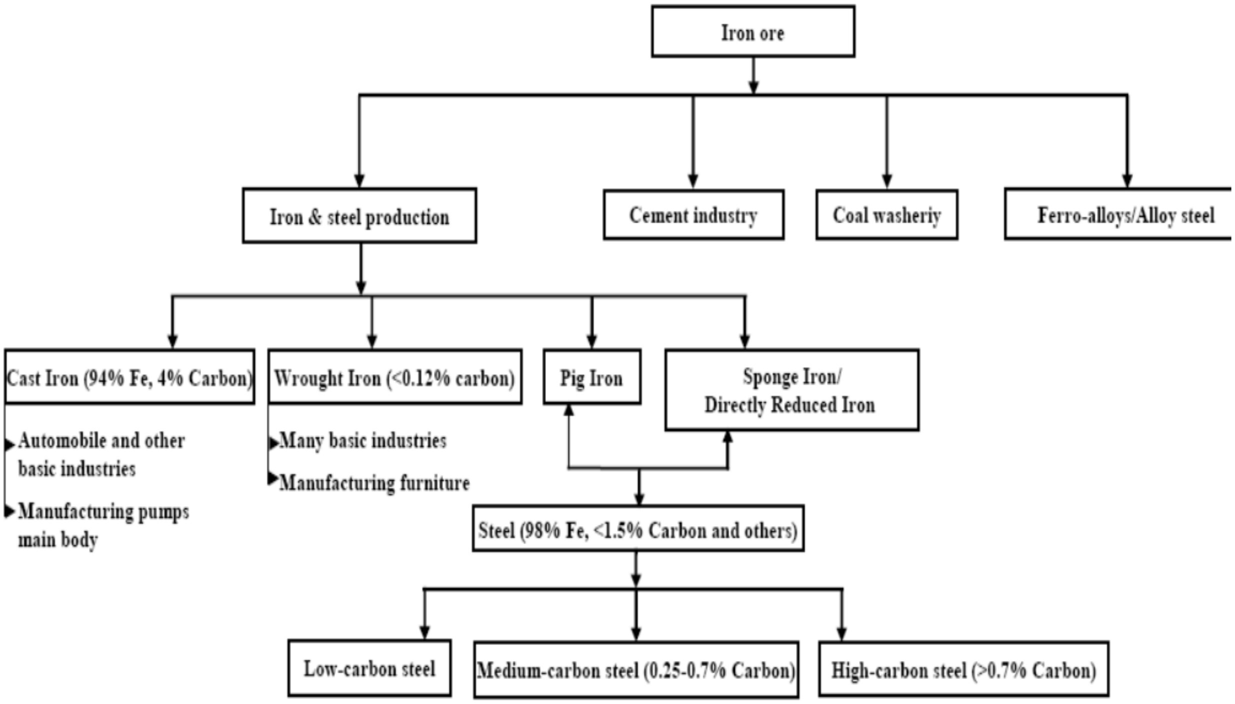
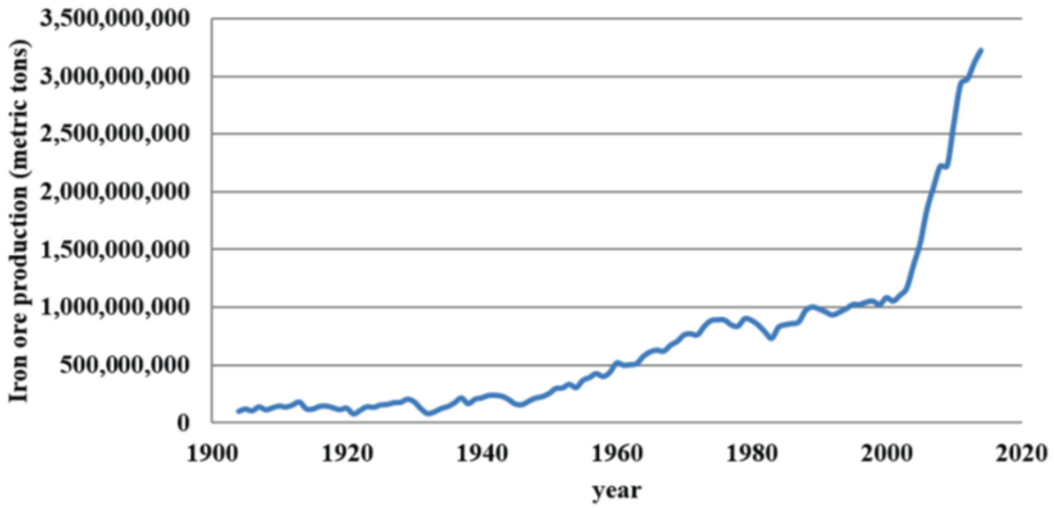


Figure 1. Various uses of iron ore.

Iron ore has a global dynamic in its production, prices, supply quantities, quality, shipping issues, and costs. Iron ore production significantly increased in the 20th century (Elamin et al., 2019). World iron ore production and consumption from 1904 to 2014 is shown in Fig. 2. Iron ore production growth rate from 2000-2013 was 1.9% (Minaei Mobtaker and Osanloo, 2015). According to UNCTAD, 2016, iron production in 2013 was 2.95 billion tons (Bt), with the major producers being China, Australia and Brazil. In 2015, decrease to 2,015 million tons (Mt), a decrease of 2.5% from 2033.2 Mt in 2014. Australia, Brazil, India and China are produced 74% of world supply in 2015 (Fig. 3).

Iron ore consumption from 1904 to 2014 is shown in figure 2 (lower) growth rate from 2000-2013 was 1.3% (Minaei Mobtaker and Osanloo, 2015). Due to increasing in world population and life quality, it is expected to have growth in iron ore consumption. The per capita consumption of iron per annum has jumped from 150 kg in 2001 to 217 in 2012, in Libya, it was 145 and now 260 (Libyan steel magazine - Issue No. 3, 2016). Such figures are indicating an increase in demand and consumption of iron and steel. The average per capita steel consumption for Ethiopia (Kg/Person) is 14.6 kg. Compared to some middle income countries like South Africa, Egypt, India with per capita iron and steel consumption value of 108.4 kg, 88.9 kg, and 57 kg respectively. Ethiopia‘s per capita consumption is found to be very low. It is even much lower than world (214.7 kg), Asian (238.8 kg) and African average (34.8 kg) (IPSRD and EiT, 2017).



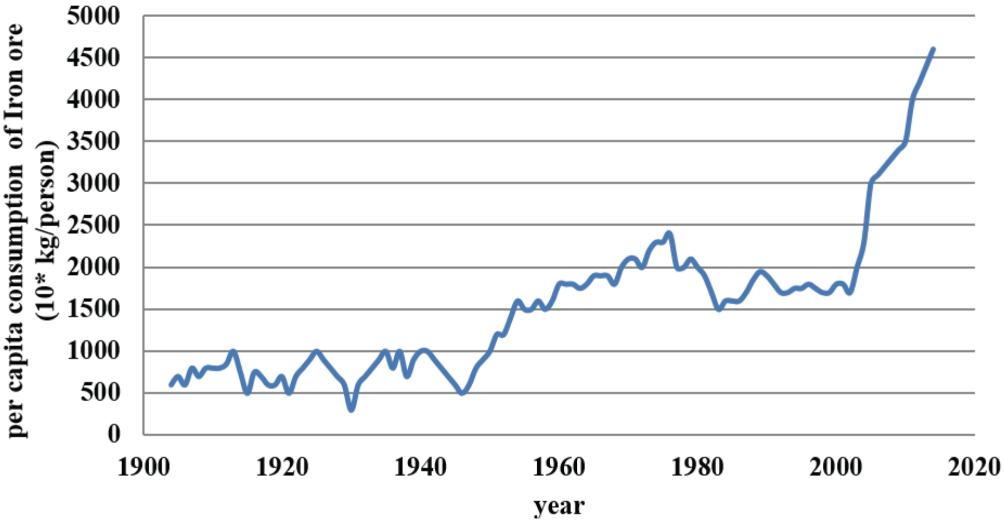


Figure 2.World iron ore production (upper) and per capita iron ore consumption from 1904 to 2014 estimated (USGS 2015) (Minaei Mobtaker and Osanloo, 2015).

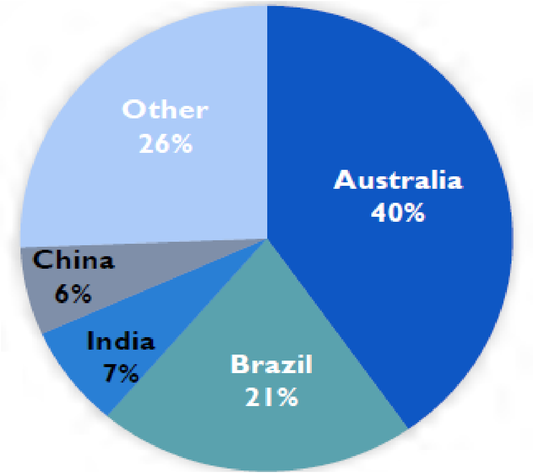


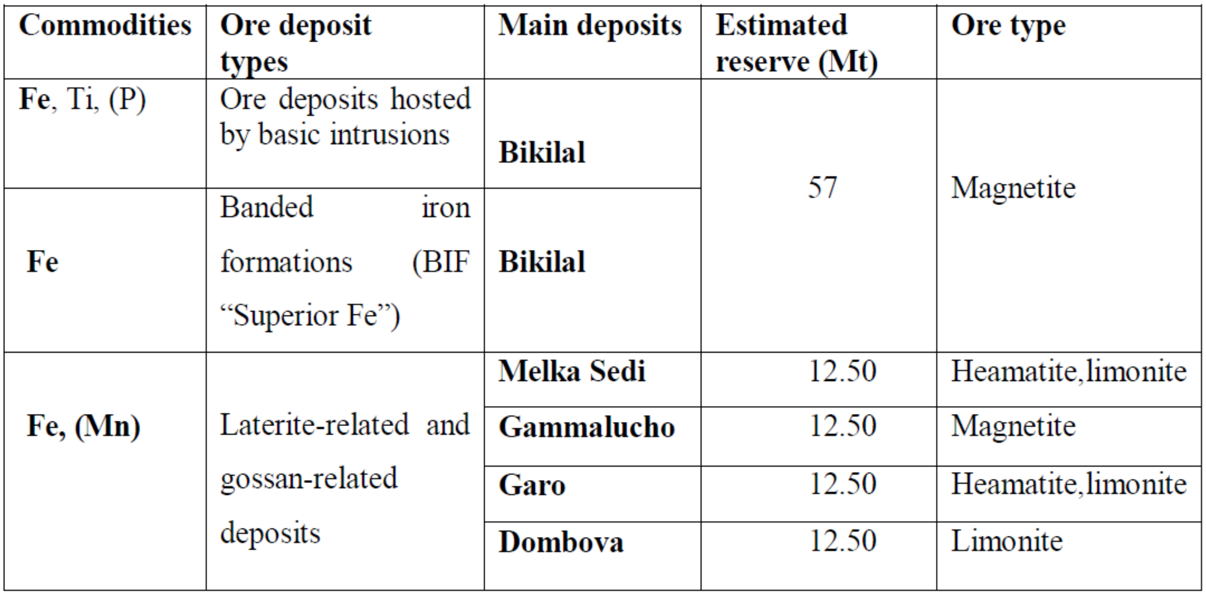
Figure 3. Top producer countries of iron ore (UNCTAD, 2016).

The world reserves base iron are estimated at about 160 Bt of iron metal. The three major types of iron ore deposits from which most ore is currently produced are deposits related to Precambrian banded iron formations (BIF) provide about 90% of all iron ore mined, and the remainder is derived from metasomatic skarn and magmatic magnetite deposits (Table 1). Other deposit types include oolitic ironstones and residual laterites are very large but low-grade resources are hosted (Morris and Ramanaidou, 2007; Gutzmer and Beukes, 2009; Filippov et al., 2014). Ethiopia iron ores deposit are mainly classified into three categories. The best known main deposit is Bikilal iron deposit. It is estimated at about 58 Mt grading 41% total Fe (EGS, 1989; Solomon, 2009). See Table 2 for iron ore deposit, type and reserve in Ethiopia.

Table 1. Major characteristics of important types of iron ore deposits (Gutzmer and Beukes, 2003).

Deposit type	Maximum deposit size (Mt ore)	Grade (wt% Fe)	Orebody shape	Remarks	Examples
Supergene/ hypogene enriched BIF					
Transvaal-Hamersley-type	> 1,000	56–69	sheet, lens	Predominant importance	Sishen (S-Africa), Serrado Carajas (Brazil) , Mt.Tom Price (WA)
Algoma-type	< 100	50–67	sheet, lens, shoot	Rare type	Buhwa (Zimbabwe)
Rapitan-type	< 1,000	50–58	sheet, lens	Rare type	Urucum (Brazil)
Metamorphosed BIF					
Taconite	> 10,000	15–40	Stratiform bed	Low grade resource	Mesabi Range (USA)
Itabirite	> 1,000	35–50	Stratiform bed	Not mined	Brazil, Australia
Ironstone					
Oolitic ironstone	500	25–45	stratiformbed	Not mined	Minette(Europe),Cinton (USA)
Reworked laterite	>1,000	50–59	Channel deposit	Significant exploration potential	Robe River (Australia)
Magmatic deposits					
In mafic and ultramafic rocks	> 1,000	30–60	plug, layer, irregularbodies	not mined	Bushveld magnetite layers and plugs; Taberg (Sweden)
In alkaline complexes	2,500	60–65	massivetabular, plug,dyke,flow	Regional significance	Kiruna,Gällivara, Grängesberg (Sweden)
Skarn deposits					
Magnetite skarn	1,000	40–60	irregular mass, vein, plug	Regional significance	Magnitaya Gora (Russia)
Siderite skarn	<1,00	20–60	irregular mass	Regional significance	Marquesado (Spain)

Table 2. Iron ore deposit, type and reserve of Ethiopia (Solomon, 2009).



Commodities	Ore deposit types	Main deposits	Estimated reserve (Mt)	Ore type
Fe, Ti, (P)	Ore deposits hosted by basic intrusions	**Bikilal**	57	Magnetite
Fe	Banded iron formations (BIF "Superior Fe")	**Bikilal**		
Fe, (Mn)	Laterite-related and gossan-related deposits	**Melka Sedi**	12.50	Heamatite,limonite
		Gammalucho	12.50	Magnetite
		Garo	12.50	Heamatite,limonite
		Dombova	12.50	Limonite

2.2 Mineralogy of iron bearing minerals

Iron is occur in a number of minerals. Over 300 minerals contain iron. Iron ore occurs mostly in oxide form. Other form are carbonates, hydroxides, sulphides and silicates. It is also found with titanium dioxide. Global iron ores are mainly contains Fe_3O_4 (magnetite), hematite (Fe_2O_3) and goethite (FeO(OH). Fe_3O_4, Fe_2O_3, (FeO(OH)), although limonite ($FeO(OH)\cdot n(H_2O)$), and siderite ($FeCO_3$) are also common iron (Fe) mineral sources. Table 3 shows iron minerals and their physical properties. These iron minerals are contains gangues and impurity elements such as Si, Al, P, S, Ca, Mg, Na, K, Ti and Mn. Quartz (silica bearing), kaolinite and gibbsite are the main alumina bearing minerals (US EPA, 1994; Sahoo et.al, 2016; Rezvanipour et al., 2018).

2.2.1. Hematite

Hematite (α-Fe_2O_3) is the stable end product of oxidation of magnetite. It has hexagonal (rhombohedral) close-packed framework and penetration to lamellar twinning without any cleavage (Haldar, 2000). Theoretical, hematite contains 69.94% iron and 30.06% oxygen. In soils, hematite (and goethite) can have concentrations of up to 5% by mass (Zhang, 2007; Maher, 2011). Hematite mainly found with silica, alumina, calcite, clay matter and phosphorus gangue minerals. The most important deposits of hematite are sedimentary and metamorphosed in origin.

2.2.2. Magnetite

Magnetite is a member of the spinel mineral group and the most magnetic of all the naturally occurring minerals on Earth. Magnetite occurs in volcanic rocks and sedimentary iron deposits (BIF) deposit, and association with sulphide mineralization. Commercially deposits include magmatic and skarns deposits (Nystrom and Henriquez, 1994). The magnetite deposit has commercial occurrences in Australia (Tallawang area). It shows coarse to ultrafine grain size (Weekes and Robertson, 1993). Magnetite is mainly use in coal washer dense medium separation and shielding of radiation X-ray equipment in hospitals.

2.2.3. Goethite

Goethite, a naturally occurring iron oxy-hydroxide mineral, is found in association with iron, manganese and bauxite ores. It is mainly formed from iron-bearing minerals through a weathering product. It has various sizes, shapes and morphology (Mohapatra et al., 2008).

The basic structural unit of goethite is a distorted octahedron. In such a structure, Al^{3+} partly replaces the central Fe^{3+} cation. It may consist different gangues includes Al_2O_3, CaO, MnO and SiO_2 (Fey and Dixon, 1981; Klein and Hurlbut, 1985).

2.2.4. Limonite

Limonite is a mixture of fine-grained hydrated iron oxides. It occurs as a pseudomorph over other minerals. Limonite is formed by weathering iron ore (Gaines et al., 1997). It is relatively soft and high water content.

2.2.5. Siderite

Siderite is a valuable carbonate of iron mineral. Its specific gravity is 3.8 and hardness varies from 3.5 to 4. It crystallizes under rhombohedral division of the hexagonal system. Siderite is an important ore constitutes only 48% iron (low iron grade) and high impurity. However, it is difficult to beneficiation using conventional methods because of low grade, easy sliming, and high impurity content. Siderite ores can be upgraded by calcination of nonmagnetic $FeCO_3$ to magnetic Fe_3O_4 or γ-Fe_2O_3 (maghemite) by using low intensity magnet, 0.17 T (Koshy, 2018).

Mineral	Formula	%Fe	Density	Hardness	Crystal System
Hematite	Fe_2O_3	69.94	5.26	5-6	trigonal
Goethite	$Fe_2O_3.H_2O$ or FeO.OH	62.85	5.07	5-5.5	orthorhombic
Limonite	$Fe_2O_3.nH_2O$	40-60	3.6-4.0	3.5	amorphous
Siderite	$FeCO_3$	48.2	3.96	4	trigonal
Magnetite	Fe_3O_4 or $Fe^{2+}O.Fe^{3+}{}_2O_3$	72.4	5.18	3-4.5	cubic
Pyrite	FeS_2	46.55	5.02	6-6.5	cubic

Table 3. Common iron minerals of economic values (After Ferenczi, 2001; Sahin, 2020).

2.3. Deleterious elements

Common deleterious elements in iron ores include phosphorus, sulphur, and titanium. Uncommon deleterious elements include vanadium, copper, zinc, chromium, nickel, arsenic, lead and tin.

2.3.1. Phosphorus

It has four major effects on iron; increases hardness and strength, lowers solidus temperature, increases fluidity and cold shortness. The strength and hardness of an iron increases with the increasing concentration of phosphorus. Phosphorus lowers the liquids temperature. Controls for high phosphorus in iron ores include avoiding high phosphorus ore minerals (Rostoker and Bronson, 1990).

2.3.2. Sulphur

Sulphur, which is usually derived from sulphide bearing minerals. Small quantity of sulphur are serious effect (Gordon, 1996). Hot short iron is brittle. Iron with over 0.03% sulphur content is avoided. Addition of manganese removes sulphur in iron ores because manganese forms a high melting sulfide at high temperatures (1610 ^{o}C) and therefore, prevents the formation of a liquid iron sulfide (Verhoeven, 2007).

2.4. Method of beneficiation

The general methods of iron ore beneficiation include beneficiation by differential crushing and screening, beneficiation by washing, wet screening and desliming, gravity concentration, magnetic separation (low and high, dry and wet), flotation and selective flocculation (Ozcan and Celik, 2016) (Fig.4). Most the high grade hematite iron ores can be use directly to iron beneficiation circuit. But, low grade ores involves processing to upgrade the iron content. Gibbsite and kaolinite gangue minerals and their soft nature increases the complexity of iron ore processing. Iron ore beneficiation methods are based on the mineralogy and gangue content of the ore minerals and their aggregation. Based on deposit type and mineralogical characteristics of the ore, different beneficiation methods are used. The most common gravity methods is employed for some, such as the separation of the heavier ore minerals from a lighter gangue (or waste) in a chosen heavy medium (Seifelnassr et al., 2012). On the other hand, the magnetite ores (magnetic) are beneficiated using LIMS (low intensity magnetic separators) (wet drum), sometimes in combination with flotation and gravity methods. Figure 5 shows beneficiation plant of primary ore.

2.4.1. Gravity separation

Non-magnetic beneficiation utilize feed sizing, shape, hardness, density and hydrophobicity (Rezvanipour et al., 2018). The main iron-bearing minerals have usually high specific gravity (hematite 5.1, magnetite 5.2, goethite 4.2, and siderite 3.85) as compared to the most common associated gangue minerals like quartz and chert 2.65, and calcite or limestone (2.7 to 2.75) (Table 3). Such differences in physical properties become suitable to appropriate gravity separation. Gravity separation is used for relatively coarse ore (diameters of approximately 0.1mm to1.5mm). The operating particle size ranges of the gravity methods are given in Fig.6. Dense media separation (DMS) is used ferrosilicon (FeSi) particles as a dense medium (Majumdar, 2008; Sarkar, 2008). The beneficiation flowsheet at Sishen mine in South Africa involved dense media separation and DMS cyclone plant. The feed size to coarse drum plant was under 90 mm and medium drum plant under 25 mm, respectively. In Australia (Mount Tom Price plant), DMS drum and cyclone plants are used to separate the low grade iron bearing ore (Maré, Bevan and Crisafio, 2015).

The shaking table is another gravity separation device. Feed particle size and table tilt are operating parameters. The feed size range is extremely wide, ranging from around 15 µm to 2 mm for mineral and up to 15 mm for coal (Burt et al., 2012). Seifelnassr et al. (2012) using shaking table a maximum grade of 44.9 % (Fe), overall recovery of 79.9 % and a stage recovery of 86.7 % at particle size fraction of -150+20 µm and table tilt angle of 5º was obtained.

In the spiral gravity technique, the range of particle size of 1mm to 0.03 mm (30µm). It is applicable for pulp density of 25-30 % solids. Spiral (one) is process up to 3 tph (Olubanbi, 2005; Raghukumar, 2012).

Jigging concenteration device is oldest one. It is used widely in the iron ore and coal industries. It separate into sections and possible for iron at the size of 30 mm to 0.5 mm (Sahin, 2020).

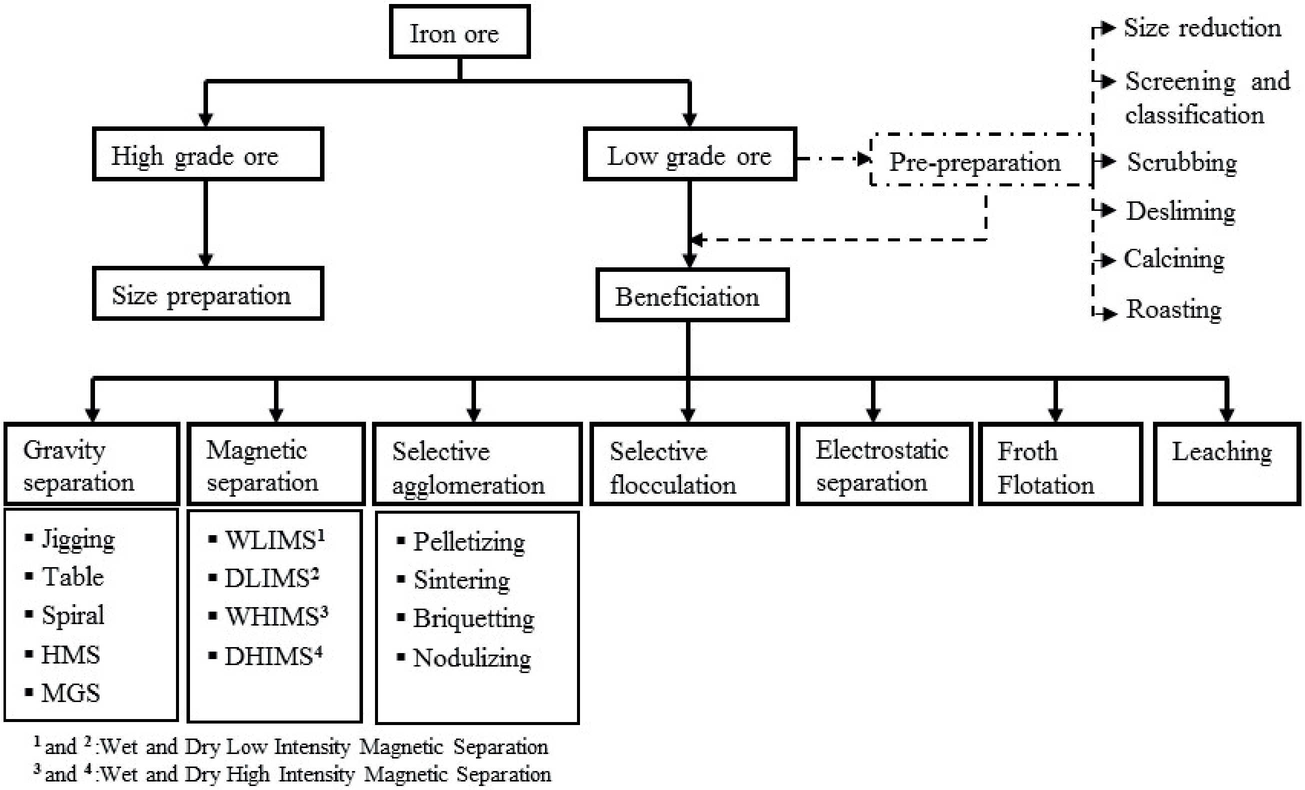


Figure 4. Beneficiation methods of high and low-grade iron ores (Yalçın and Ateşok, 1979; Sis, 2021).

2.4.2. Magnetic separation

Magnetic separation is a technique used concentrate iron ore based on the difference in their magnetic properties. The magnetic susceptibility values (10−6 m3kg-1), iron can be strongly magnetic iron ores, that is, magnetite (625-1156) and weakly magnetic iron ores (oxidized iron ores), that is, martite (6.2-13.5), hematite (0.6-2.16), specularite (3.7), limonite (0.31-1.0), and siderite (Xiong et al., 2015). Wet and dry low intensity magnetic separators (LIMS) techniques are employed to high magnetic properties minerals.The high magnetic susceptibility of magnetite, due to its high iron (72.4%) content, makes it suitable for beneficiation with magnetic separation techniques. Wet high-intensity magnetic separation (WHIMS) is efficient in concentration of paramagnetic minerals (e.g., hematite, goethite, and limonite)(Xiong et al., 2015).

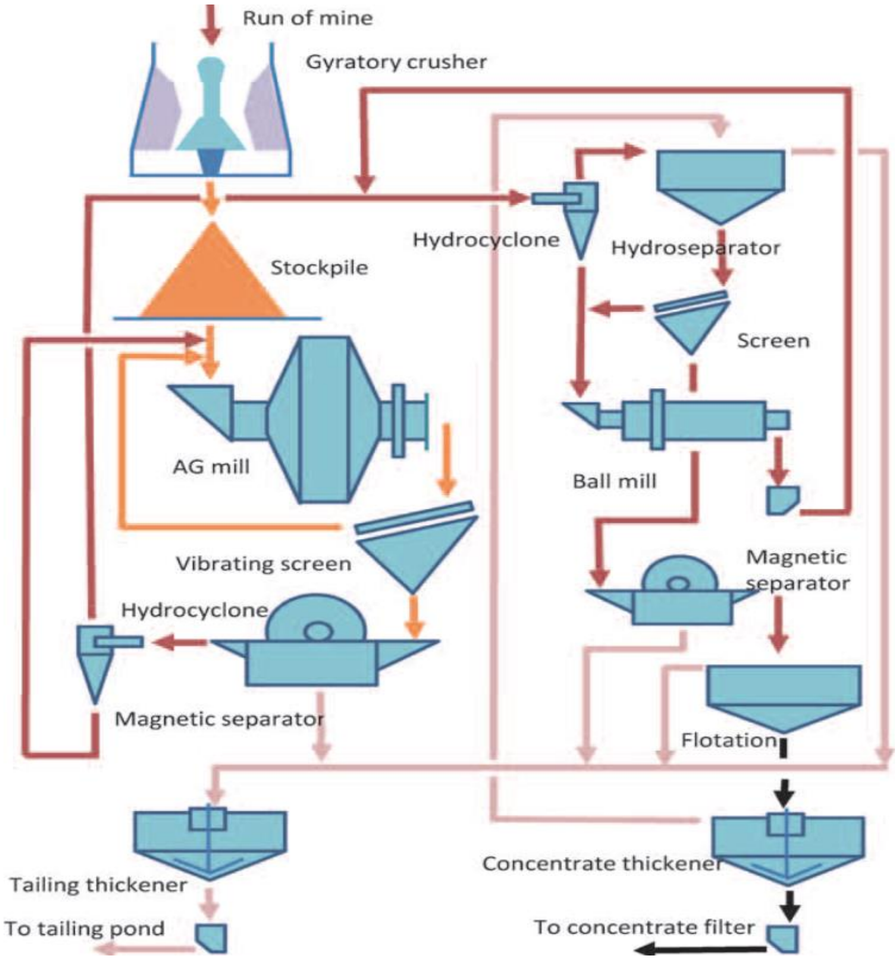


Figure 5. Beneficiation plant of the primary iron ore (Tsutomu et al., 2015).

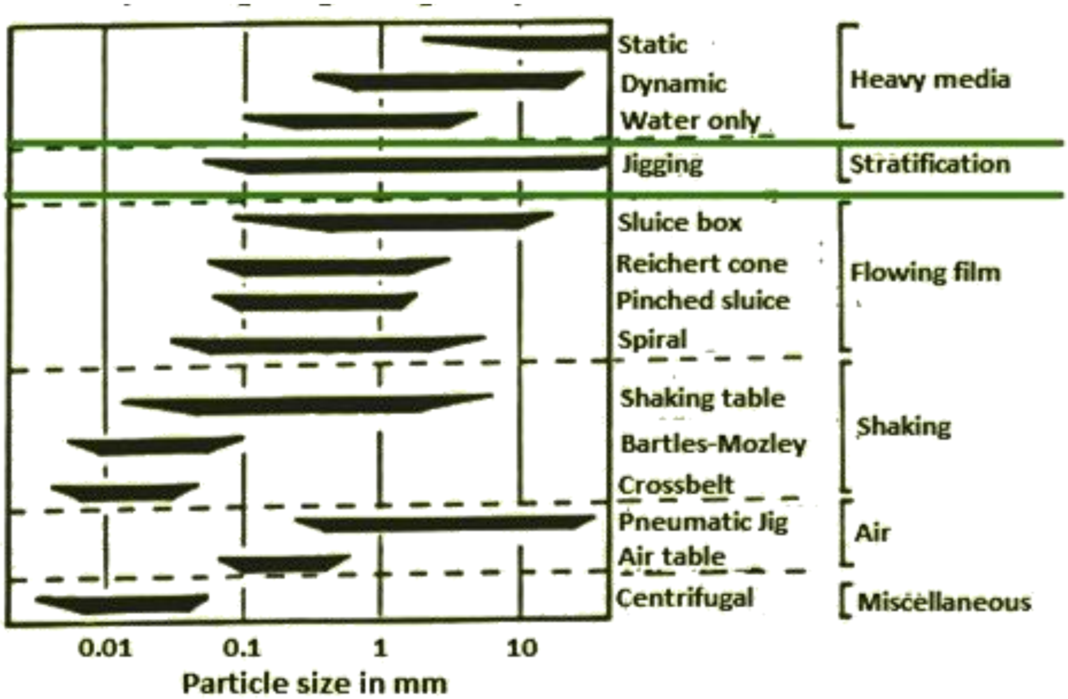


Figure 6. Operating particle size range of common gravity separators (Rath and Singh, 2007).

2.4.3. Flotation separation

Forth flotations is a technique utilizing the difference in the hydrophilicity of ore surfaces and it accomplished in mechanical cells or flotation columns. A mechanical flotation cell is shown in figure 7 which depicts overall flotation process. It is effective that do not respond to gravity or size classification units (Ma, 2012; Nakhaei and Irannajad 2018; Zhang et al., 2019; Murhula, 2023). The flotation can be direct, reverse anionic and reverse cationic flotation. In direct flotation the iron is floated using a collector while the gangue material is rejected in the underflow product. In reverse flotation the gangue mineral is floated while the iron is recovered in the underflow product (Zhang et al., 2019).

2.4.3.1. Direct flotation

Direct flotation route was used in alkaline media (Ma, 2012). Soda ash were used to maintain a pH of 9-10, and fatty acids are added as collectors. Fatty acids (approximately 91% oleic) and alkyl are used float hematite (Pattanaik and Venugopal, 2018). Direct flotation in weakly acidic media has been used by the Qidashan concentration (China). Qidashan concentrate built to concentrate from 28.94% Fe grade to 63.6% when sodium petroleum sulfonate collector and sulfuric acid pH regulator was used (Zhang and Dai, 2012).

2.4.3.2. Reverse cationic flotation

Presently this is the most accepted methodology for concentrating the iron ores. Typically, this process is performed at alkaline pH conditions. So, the iron and silica particles can remain dispersed from each other during the separation process (Zhang et al., 2019). Silica is mainly rejected, while iron oxides are depressed, with the use of collector and depressant, respectively in alkaline medium, adjusted by NaOH (Thella, 2010; Filippova et al., 2014). This process use hematite and goethite ores.

2.4.3.3. Reverse anionic flotation

In the anionic reverse flotation, the gangue minerals, mainly silica are activated by divalent calcium ions and will be floated out through the interactions with anionic collectors. In contrast, the iron-containing minerals are kept in suspension with depressant starch (Cao et al., 2013; Lima et al., 2013; Liu et al., 2016).

Reverse anionic flotation of iron ore was first developed in the U.S. by Hanna Mining and American Cyanamid using iron ore deposits from Lake Superior (Zhang et al., 2021; Filippov et al., 2014). However, this process was replaced due reagents cost and filtration problems (Filippov et al., 2014). Nowadays the reverse anionic flotation process is not very common in the U.S but it is very popular in China (Yuan et al., 2007). The Anshan-type iron ore constitutes 55% of the ore reserves in China. About 40% of this is characterized by low grades and complex mineralogy (Sun, 2006). This type of ore is processed in various Chinese concentrator plants such as the Qidashan concentrator, the Sijiayin mine, the Yuanjiacun concentrator and the Gongchangling concentrator by a process composed magnetic separation, and reverse anionic flotation (Zhang et al., 2021). Typically, calcium oxide is used as an activator, corn starch as a depressant, and a modified fatty acid as a collector, while maintaining a pH of 11.5 with caustic soda (Zhang et al., 2021).

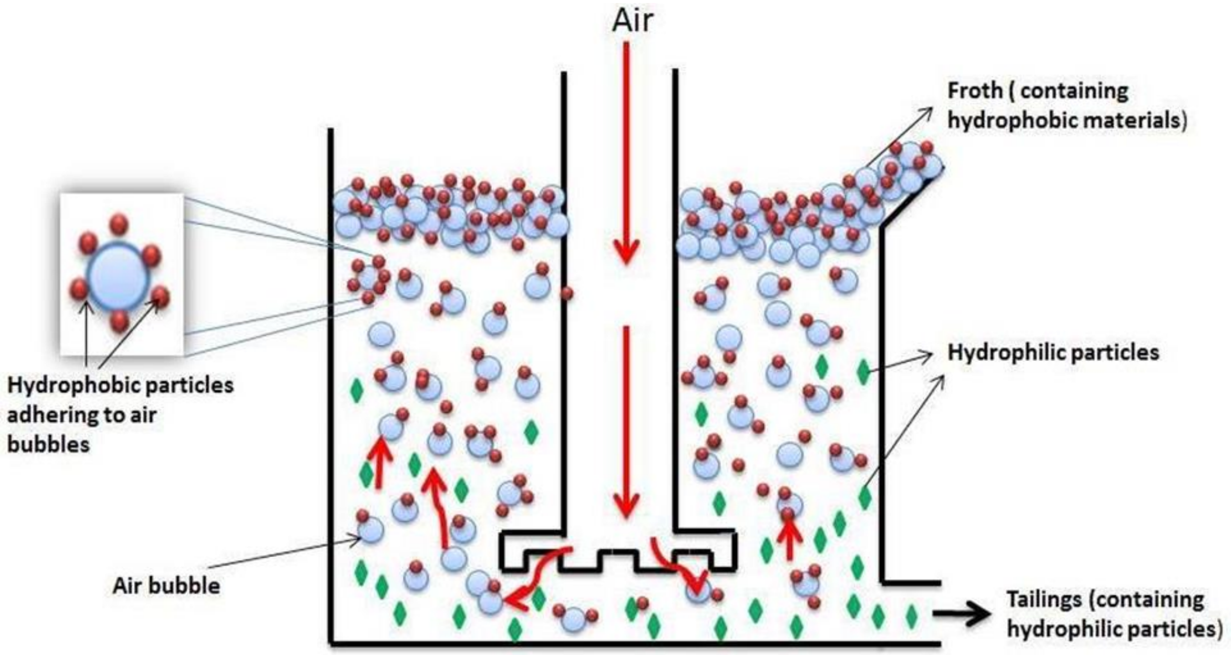


Figure 7. Mechanical flotation cell.

2.4.4. Flotation Reagents

Reagents are substances that are used to make effective flotation. For effective flotation, typical involves collectors, depressants, frothers, modifiers, dispersant, and flocculant (Damisa, 2007).

2.4.4.1. Collectors

The collectors utilized in reverse flotation can be anionic or cationic collectors.

Anionic collectors utilise a negatively charged solidophilic group. They are subdivided into two groups i.e. oxyhydryls and sulfhydryl collectors. Anionic flotation utilises the oxyhydryl anionic collectors (Bulatovic, 2007).

Cationic collectors can be fatty amines or ether amines (Vidyadhar et al., 2007).

2.4.4.2. Frothers in flotation

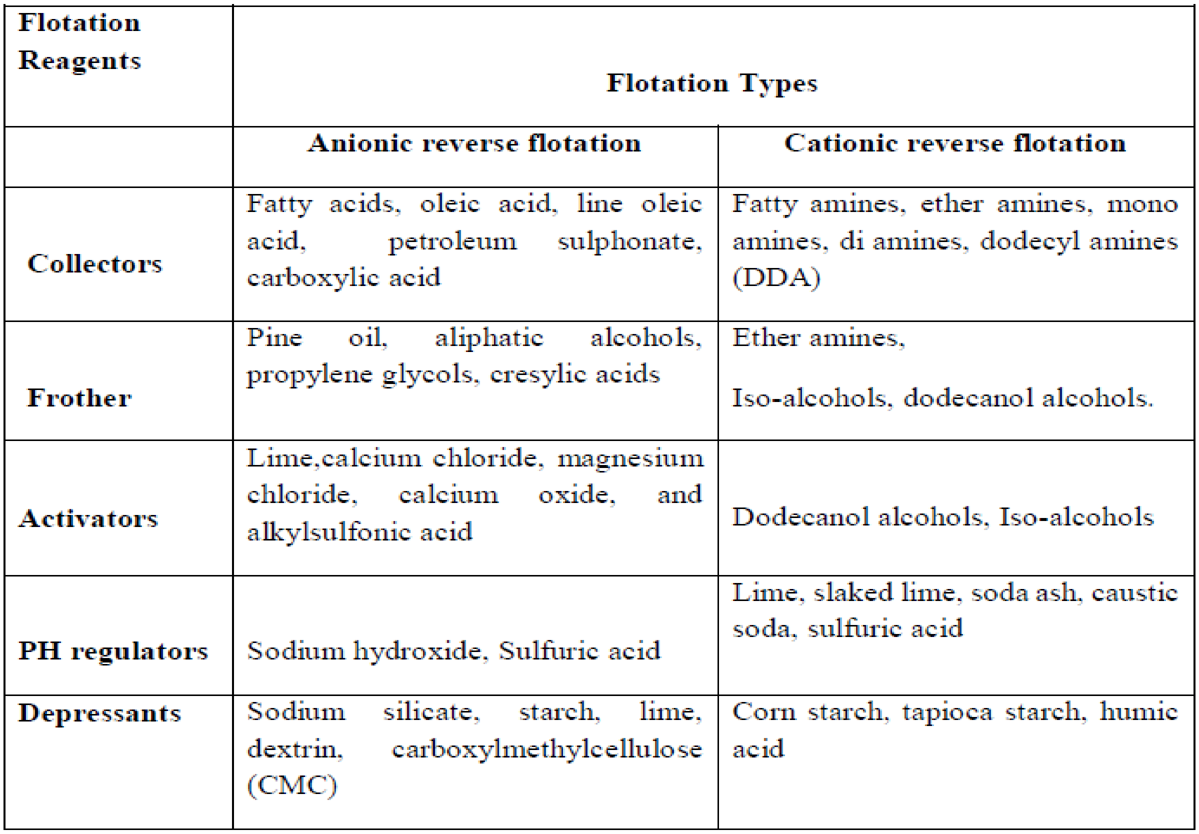
It used in creating froth, controlling the froth size, froth stability and froth mobility in flotation (Bulatovic, 2007, Vidyadhar and Singh, 2007). Propylene glycols, alkyl ethers and cresylic acids frothers are mainly used in iron ore flotations. Alcohols are the most soluble frothers in iron (Fe) flotation (Nakhaei and Irannajad, 2018).

2.4.4.3. Modifiers or Regulators

Modifiers are used to magnify or reduce the collector action to enhance the selectivity. They are classified as activators, depressants or pH modifiers (Bulatovic, 2007).

- Activators are considered regulators that react directly with mineral surfaces. The most common activator in the reverse anionic of iron ore is calcium oxide (CaO). The calcium oxide activators is effective and strong silica activation at pH > 11 (Zhang et al., 2021).
- Depressants are used to reduce the conditions for hydrophobization, or the ability to make surfaces hydrophilic, preventing them to be floated. Starch is the most popular in the reverse flotation of iron oxide (Nakhaei and Irannajad, 2018).
- pH regulators are used to render the pulp alkaline and enable attachment of silica bearing minerals to collectors during flotation. Most of the flotation processes performed under the alkaline media. The common pH regulators are NaOH, lime (CaO), soda ash and sulphuric acid (H_2SO_4) (Wills B. A., 2016).

Table 4. Reverse anionic and cationic flotations and different reagent.



Flotation Reagents	**Flotation Types**	
	Anionic reverse flotation	**Cationic reverse flotation**
Collectors	Fatty acids, oleic acid, line oleic acid, petroleum sulphonate, carboxylic acid	Fatty amines, ether amines, mono amines, di amines, dodecyl amines (DDA)
Frother	Pine oil, aliphatic alcohols, propylene glycols, cresylic acids	Ether amines, Iso-alcohols, dodecanol alcohols.
Activators	Lime,calcium chloride, magnesium chloride, calcium oxide, and alkylsulfonic acid	Dodecanol alcohols, Iso-alcohols
PH regulators	Sodium hydroxide, Sulfuric acid	Lime, slaked lime, soda ash, caustic soda, sulfuric acid
Depressants	Sodium silicate, starch, lime, dextrin, carboxylmethylcellulose (CMC)	Corn starch, tapioca starch, humic acid

2.4.5. Common factors affecting flotation

Flotation performance depends on the interaction of mineralogical characteristics, chemical and physical factors, and engineering parameters. Collector type and dosage, particle size, pH modifiers, conditioning time and pulp density are mainly affect flotation concentrate.

2.4.5.1. Collector type and dosage

The quantity/dosage of collector is one of the various factors influences the performance of flotation process. The selection of collector is determined by the mineral surface charge, collector's ability dissolve in water and its hydrocarbon chain length (Filippov et al., 2014; Zhang et al., 2019). The collector quantity continued to increase will increase the selectivity until a point when the recovery drastically decrease (Wen-gang et al., 2009).

2.4.5.2. Particle size

The flotation conenterate is strongly influenced by the feed mineral particle size. The fine feed grind size have higher liberation, which will allow increases of valuable mineral recovery. Fine particle exhibit larger adsorption area, so the reagent consumption much higher than coarse particle. The presence of slime (less than 25µm size) has deleterious effect, and reduces selectivity (Ma et al., 2012). Entrainment (particles report to the froth) is also common for particles size less than 50 µm. It is not chemically selective, hence, both the target and gangue minerals report to the froth phase.

Coarse particle exhibit slow flotation kinetics. This is due to poor mineral liberation and actual size. Similarly, the flotation kinetics of fine particles decrease with poor flotation collision (Runge et al., 2007). A maximum flotation recovery is often achieved at an intermediate particle size. Recovery as a function particle size is present in figure 8. The figure depicted that the recovery increase with increase grind particle size. However, after intermediate particle size, optimum recovery obtained, a drastic drop in recovery is observed.

2.4.5.3. pH modifiers

The surface chemistry of most minerals is affected by the pH value. Effective flotation is mostly conducted under alkaline condition. Under alkaline condition, most collectors are stable and metal corrosion is minimized (Wills and Finch, 2016).

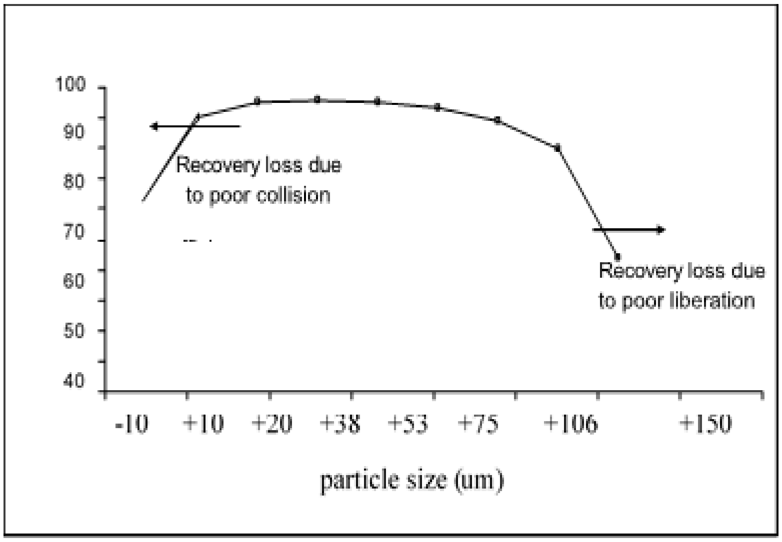


Figure 7. Typical view of flotation recovery as a function of different particle size (Runge et al., 2007; Santana et al., 2008).

Cationic reverse flotation of silica and iron oxide is stronger at alkaline medium. Anionic reverse flotation conducted at pH 11.5, hematite and quartz ultrafine particles are highly negative charged and repelling each other. It shows Fe recovery is better in the particle size <10 µm (Ma et al., 2012). Slightly negative charged hematite surface was observed in cationic reverse flotation at pH 10.5.

CHAPTER THREE

3. Material and Methods

3.1. Materials

3.1.1. Ore mineral samples and flotation reagents: Representative low-grade hematite ore sample was collected from Ethiopian Geological Institute which brought from Sekota iron deposit area, Amhara Region, Northern Ethiopia. The hematite ore was prepared by crushing and grinding in a laboratory jaw crusher and ball mill, respectively. The ground material was not deslimed. The particle size distribution was determined by screening in 75 and 63 µm sieves. The sieved product was then homogenized and weighted. The grinding product then subjected to the flotation cell. All chemicals used in the flotation experiments were purchased from commercial center. The different reagents used in the flotation experiments are presented (table 5). This includes: oleic acid ($C_{18}H_{34}O_2$) collector, ethanol which was used as a frother, Carboxylmethylcellulose (CMC) as a depressants, and sodium hydroxide, NaOH which was used to adjust pH of the pulp and calcium chloride ($CaCl_2$) were used as anionic quartz activator. Local tap water was used in all flotation experiments.

Table 5. The reagents were used in the flotation experiments.

Reagent type	Reagent name	Dosage (g/t)
Collector	Oleic acid ($C_{18}H_{34}O_2$)	1 to 6
Depressant	Carboxyl methyl cellulose (CMC)	5
Activator	Calcium chloride ($CaCl_2$)	3
Frother	Ethanol (CH_3CH_2OH)	1ml
pH modifier	Sodium hydroxide (NaOH)	5

3.1.2. Previous analysis documents: Documents that contain chemical, mineralogical and particle size distribution data's of Sekota iron ore deposit was collected from Ministry of Mines and Ethiopia Geological Institute. The analysis done for characterization of the ore sample was adapted from works of Matloga and Youlton, 2014.The chemical and mineralogical analysis were carried out using XRF, XRD and QEMSCAN Bulk Modal Analysis (BMA). The sample was crushed to 2mm size (100% passing) and 100g was pulverized for chemical and XRD analysis.

3.2. Methodology

3.2.1. Flotation Experiments

Reverse anionic flotation (flotation cell: CFB - 1000N, Wedag) using rougher stage only is use to concentrate the valuable iron mineral. It carried out with a rougher stage to study the effect of collector dosage (1, 2, 3 and 6 g/t) and particle size fraction (-75μm and -63 μm) on the iron flotation performance. A batch flotation was carried out with laboratory scale flotation machine adjusted at the agitation speed of 2000 rpm. Laboratory scaled flotation machine is showen in figure 9. Flotation tests were conducted using 300 to 330g of iron ore each particle size fractions was mixed with 2L of tap water in flotation cell for 1 min. NaOH was added to maintain the pH at about 11-12. After adjustment of pH, collector and a milliliter (ml) frother were then added. The oleic acid collector dosage was varied from 1-6g and 1ml of ethanol frother was fixed in all flotation. Modifiers such as activators 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. CMC used as depress iron bearing hematite minerals. The activators and depressant dosages were kept constant along the flotation experiments. It was used 3g and 5 g/t respectively for all experiments. All reagents were added for 3 minute interval. The total residence time for a flotation of 1batch test was 20 minutes. Concentrates were collected. The froth was scraped manually by using a plastic blade in to plastic bag. The concentrates and tailings were dried, weighed, and analyzed using AAS machine.

3.2.2. Characterization of the concentrate and flotation performance: Chemical analysis was conducted on the concentrate and tailing obtained to determine their percentage iron content. Complete silicate (major oxide) analysis of the concentrate was done using XRF.

Figure 8. Flotation cell at Addis Ababa University, Addis Ababa Institute Technological laboratory.

The effectiveness of the flotation test under different conducted condition was measured by measuring the iron grade (% of Fe by weight), calculating metal recovery and metal loss (metal in tail) as follows;

$$\mathbf{\%\ of\ Fe} = \frac{\mathbf{Molar\ mass\ of\ Fe * Number\ of\ mole\ of\ Fe}}{\mathbf{Molecular\ mass\ of\ Fe_2O_3}} * \mathbf{100} \ldots\ldots \quad (1)$$

$$\mathbf{Assay\ of\ Fe} = \frac{\mathbf{\%\ of\ Fe * assay\ of\ Fe_2O_3}}{\mathbf{100}} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

$$\mathbf{Recovery\ (R)} = \frac{\mathbf{Cc}}{\mathbf{Ff}} * \mathbf{100\%} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots. (3)$$

$$\mathbf{Tail\ (T) = 100 - R} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Where, assay of Fe – grade of Fe in the concentration, C is the weight of flotation concentrate (g), c is the assay/grade of concentrate iron (%), F is the feed of iron ore (g) and f is the assay/grade of feed iron (%).

CHAPTER FOUR

4. Results and Discussions

4.1. Ore characterization

Ore mineral characterization which gives information about the chemical, mineralogical, particle size and liberation attributes are crucial importance in studies of low grade ore deposits as it helps to determining the most suitable beneficiation methods. Therefore, chemical, size wise chemical, mineralogical and particle size analysis of Sekota iron ore was carried out before beneficiation tests.

4.1.1. Chemical Analysis (AAS and XRF)

AAS was carried out in Central Laboratories of Ethiopia Geological Institute (EGI). The AAS results of the collected sample is presented in table (table 6). The sample consists average grade of 45.57 Fe. Hematite is the dominant iron ore mineral with SiO_2 (quartz) and Al_2O_3 as main gangue minerals.

Table 6. AAS chemical analyses results of hematite iron ore in percent.

Sample No.	Composition (%)										
	Fe_2O_3	Fe	SiO_2	Al_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2
AK-1	60.12	42.04	23.98	10.6	0.32	<0.01	0.1	0.09	0.01	0.04	0.84
AK-2	70.12	49.1	17.26	7.11	0.32	<0.01	0.12	0.09	0.01	0.04	0.81
Average	65.12	45.57	20.62	8.86	0.32	<0.01	0.11	0.09	0.01	0.04	0.82

Chemical analysis was done by using XRF method. Chemical analysis and size wise distribution results of the raw ore is given in table 7 and 8. The ore sample contains 51.7% Fe_2O_3 and 35.4% SiO_2. It contains 51.7% hematite, corresponding to a low grade ore. The Fe grade doesn't meet the blast furnace requirement and there is need to upgrade the low-grade ore. XRF results depicted that silicates and alumina are main gangue minerals. The silica content is high (35.4% SiO_2) compared to the generally accepted level of 1.5% for commercial ores. In addition, the sample contains a high amount of alumina (7.79%). It is evident that iron is significantly contaminated with quartz and alumina-bearing mineral (kaolinite). The level of other impurities are not very high. The ore contains 0.04% phosphorus and 0.03% sulfur which are acceptable as compared to the upper limit of 0.04% and 0.15% . P, S and silica in the iron concentrate is acceptable less than 0.04%, 0.15 % and 1.5 %, respectively (Oliazadeh1, M and Vazirizadeh, A., 2016). Generalized contents for P, S, SiO_2 and Al_2O_3 requirements in commercial iron ores are shown in table 9.

Chemical analysis (size wise) of Sekota iron ore is presented in table 8. It shows that element is evenly distributed in different particle size fractions. The main valuable component is iron, the content of which ranges from 35.04 to 38.26% Fe in the size range -75 to +850 µm, respectively. The content of hematite iron is 50.1−54.7%. The impurity SiO2 is in greater proportion (39.5%) in the size fraction +75 µm. When observing the others components, they are major in the fraction <75 µm, mainly Al_2O_3 (9.68%) and LOI (4.07%), which are associated with the presence of kaolinite, which commonly occur in the finer particle fractions. For the studied ore, the content of phosphorus pentoxide was 0.042-0.046%. The sulfur content is <0.01−0.02%, is under the acceptable value of 0.15% for iron concentrates, and therefore, there is no need for desulfurization. The MgO, Na_2O and Cr_2O_3 values (<0.01) are generally below the detection limit.

Table 7. Major elemental analysis by XRF and sulfur (Matloga and Youlton, 2014).

Element	Detection limit (%)	%Composition
Fe_2O_3	0.01-100	51.7
SiO_2	0.01-100	35.4
Al_2O_3	0.01-100	7.79
CaO	0.01-100	0.04
MgO	0.01-100	<0.01
K_2O	0.01-100	0.04
MnO	0.01-100	<0.01
Na_2O	0.01-100	<0.01
P_2O_5	0.0023-100	0.04
TiO_2	0.01-100	0.63
Cr_2O_3	0.01-100	<0.01
V_2O_5	0.01-100	0.16
Total	0.01-120	100
LOI	50-100	3.51
S	0.01-100	0.03

Table 8. Major elemental analysis by fraction size (Matloga and Youlton, 2014).

Size fraction (μm)	composition %								
	Fe_2O_3	Fe	SiO_2	Si	Al_2O_3	Al	CaO	MgO	K_2O
+850	54.7	38.26	33.6	15.71	7.6	4.022	0.05	<0.01	0.05
+425	53	37.04	36.9	17.25	6.9	3.65	0.04	<0.01	0.04
+75	50.1	35.04	39.5	18.46	7.13	3.77	0.05	<0.01	0.04
-75	54.46	38.19	30.9	14.44	9.68	5.12	0.06	<0.01	0.06
Size fraction (μm)									
	MnO	Na_2O	P_2O_5	TiO_2	Cr_2O_3	V_2O_5	Total	LOI	S
+850	0.02	<0.01	0.045	0.61	<0.01	0.16	65.24	3.49	0.01
+425	<0.01	<0.01	0.046	0.59	<0.01	0.17	68.63	3.13	0.02
+75	0.02	<0.01	0.042	0.61	<0.01	0.14	72.78	3.12	0.02
-75	<0.01	<0.01	0.045	0.71	0.02	0.15	65.15	4.07	<0.01

Table 9. Impurities level of analyzing iron Ore.

Components	Total iron content			Impurities			
	Low	Medium	High	S	p	SiO_2	Al_2O_3
Content Mass (%)	<58	62-64	>65	0.1	0.05 - 0.07	6	3-4

4.1.2. Ore mineralogy

X-Ray Diffraction

The X-Ray Diffraction spectrums of Sekota iron ore is presented in figure 10. Using XRD analysis, it reveled that the predominant crystalline minerals is hematite (Fe_2O_3), with lesser amount of quartz (SiO_2), which showed a medium grade of >50% and 10-20%, respectively (Table 10). Minor amount of 10% kaolinite $Al_4(Si_4O_{10})(OH_8)$ is also occurred. The major constituents were hematite as an iron-bearing mineral, quartz and kaolinite are gangue, and the rest were minor minerals. This is in line similar / with the XRF major oxide elemental analyses results, which displayed elevated contents of iron oxide (51.7%), silica (35.4%) and alumina (7.79%). No magnetite and goethite was detected in Sekota iron sample during the XRD analysis. The BMA data indicate that the sample contains significant amount goethite.

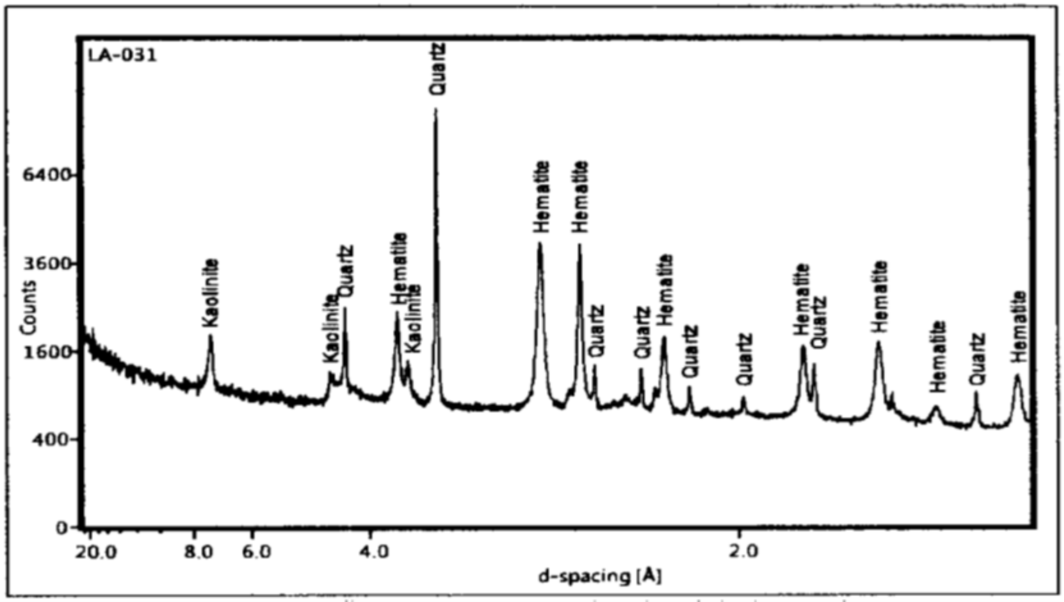


Figure 10. X-ray diffraction analysis image of the ore (Matloga and Youlton, 2014).

Bulk Modal Analysis

The bulk mineralogy determined by QEMSCAN BMA analysis is presented in table 11. Based on the BMA results, the iron sample mainly composed of Hematite 36.15, Quartz 30.94%, Goethite 17.44% and Kaolinite 14.37% with trace amount of feldspar, rutile/ilmenite and mica. Quartz exists as the major gangue mineral associated with these hematite bearing ores.

Table 10. XRD Analysis result of representative sample.

Mineral	Chemical Formula	%Composition
Hematite	Fe_2O_3	>50
Quartz	SiO_2	10-20
Kaolinite	$Al_4(Si_4O_{10})(OH_8)$	3-10

Table 11. QEMSCAN bulk modal analysis results.

Mineral	% Composition
Hematite	36.15
Goethite	17.44
Quartz	30.94
Kaolinite	14.37
Feldspar	0.73
Rutile/ilmenite	0.23
Mica	0.09
Other silicates	0.03
Other sulphides	0.01
Other	0.03

4.2. Flotation of iron

As was indicated in the previous sections, flotation tests were conducted in order to obtain a reasonable grade and recovery values. With the aim of determining the optimum flotation conditions yielding maximum possible recovery and grade, eight (8) flotation test by varying different parameters were performed. According to previous studies and literature review, sodium hydroxide as the pH regulator, CMC depressant, $CaCl_2$ activator, $C_{18}H_{34}O_2$ collector and CH_3CH_2OH frother was used. During the flotation tests variable operational parameters were chosen as collector dosage and particle size fraction. Corresponding effects of each parameter on flotation performance were investigated by changing each of them one by one while keeping rest of all parameters as constant.

4.2.1. Effect of collector dosage on Fe grade

Effect of the different quantities of oleic acid were studied (1, 2, 3, 6 g/t). On the flotation separation of Fe four batch flotation test was done for each particle size fraction (-75µm and -63 µm). The chemical analysis result obtained on four batch flotation tests conducted on the effect of oleic acid quantities are shown in table 12 and table 13. The rough concentrates was analyzed using AAS.

Table 12. AAS analysis result of the concentrate by particle size fraction -75 µm.

Oleic acid dosag	Major oxide										
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O
1g/t	21.56	9.36	61.24	0.16	<0.01	0.08	<0.01	<0.01	0.03	0.73	0.95
2	21.51	9.6	61.81	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.72	0.4
3	19.92	10.01	62.14	0.8	<0.01	<0.01	<0.01	<0.01	0.04	0.73	0.91
6	22.3	8.07	62.08	0.16	0.03	0.1	<0.01	<0.01	0.05	0.72	0.78

Table 13. AAS analysis result of the concentrate by particle size fraction -63 µm

Oleic acid dosage (g/t)	Major oxide										
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2O
1	21.4	10.01	61.3	0.01	0.03	0.06	0.04	0.01	0.01	0.75	0.98
2	20.08	9.98	62.17	<0.01	<0.01	<0.01	0.01	<0.01	0.02	0.81	0.8
3	17.34	7.7	62.96	0.22	<0.01	0.11	<0.01	<0.01	0.01	0.72	0.78
6	18.18	8.5	62.56	0.16	0.32	<0.01	<0.01	<0.01	0.46	0.73	0.95

Table 12 and 13, gives the AAS chemical analysis result of reverse flotation test using varied oleic acid collector amount for the feed size ranges -75 µm and -63 µm. Based on the assay of the hematite (Fe_2O_3) concentrate shown in table 12 and 13, Fe grade and recovery are computed using equation 2 and 3. The effect of oleic acid quantity on iron grade and iron recovery is shown in table 14.

Figure 11 shows Fe grade and recovery were obtained from -75 µm size fraction test. It depict increasing collector (oleic acid) quantity, the grade of iron slightly increase. The Fe grade 42.86% and 43.26% (-75 µm size fraction) are obtained with 1g/t of oleic acid collectr and oleic acid 2g/t, respectively. The recovery of Fe at these collector quantity are 43.26% and 42.06%, respectively. The iron grade continued to increase with increasing oleic acid collector amount until it reached to 43.49%. The Fe grade then reach to 43.49% maximum value at a collector dosage 3g/t with the recovery of 40.48%. However, slightly decrease in Fe grade is observed with over-dosage collector quantity (6g/t). At this quantity, the Fe grade is 43.45% with a recovery of 40.36%. Over-dosing with collector can lead also to froth stability issues (immobility to collapse), and can cause bubble clustering. So, further collector dosage increase didn’t improved the iron grade.

Table 14. The result of the flotation test using -75 µm size fraction.

Oleic acid quantity (g/t)	Weight of feed (g)	Products	Weight products (g)	Assay of Fe_2O_3 (%)	Fe grade (%)	Fe recovery (%)
1	320	Concentrate	154.5	61.24	42.86	42.36
		Tailings	165.5	NA	NA	57.63
2	320.1	Concentrate	153.3	61.81	43.26	42.06
		Tailings	167.8	NA	NA	57.94
3	325.6	Concentrate	150.2	62.14	43.49	40.48
		Tailings	175.4	NA	NA	59.51
6	330	Concentrate	151.8	62.08	43.45	40.36
		Tailings	178.2	NA	NA	59.64

Note: NA not analyzed

According to flotation result were showed in table 14 and presented in figure 11, Fe iron grade and recovery has inverse relationship. Increasing the iron concentrate (sink) grade result in decrease the hematite recovery.

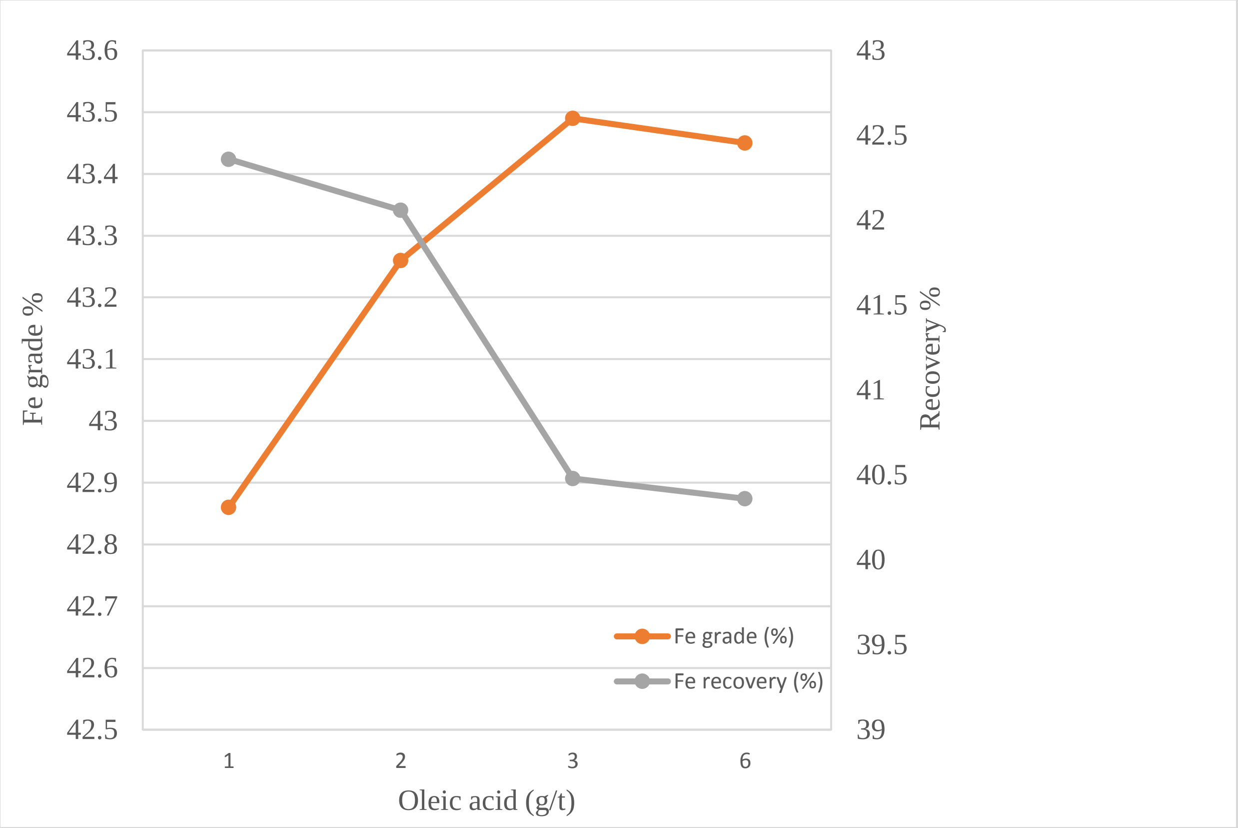


Figure 9. Effect of collector quantity on Fe grade and recovery for -75 µm size fraction.

Table 15 and figure 12 shows the batch reverse flotation test result obtained for -63 µm feed sample. As shown in -63 µm size fraction test, the oleic acid collector quantity has also similar impact on Fe grade and recovery. In general, increasing the collector quantity led to slightly increase concentrate (sink) grades and decrease recovery percent. The optimum conditions for Fe grade, from -63 µm particle size is obtained by adding 3g/t oleic acid. A result show sink (concentrate) assaying 62.96% hematite (Fe_2O_3) with 43.4 % Fe recovery and 44.07 % Fe grade at this collector dosages.

Table 15. Flotation test result using -63 µm particle size fraction.

Oleic acid quantity	Weight of feed (g)	Products	Weight products (g)	Assay of Fe_2O_3 (%)	Fe grade (%)	Fe recovery (%)
1	300	Concentrate	155	61.3	42.9	45.34
		Tailings	145.5	NA	NA	55.86
2	300	Concentrate	153.9	62.17	43.52	45.01
		Tailings	146.1	NA	NA	54.99
3	300	Concentrate	150	62.96	44.07	43.4
		Tailings	155	NA	NA	56.6
6	300	Concentrate	151	62.56	43.79	45.2
		Tailings	149	NA	NA	54.8

Note: NA not analyzed

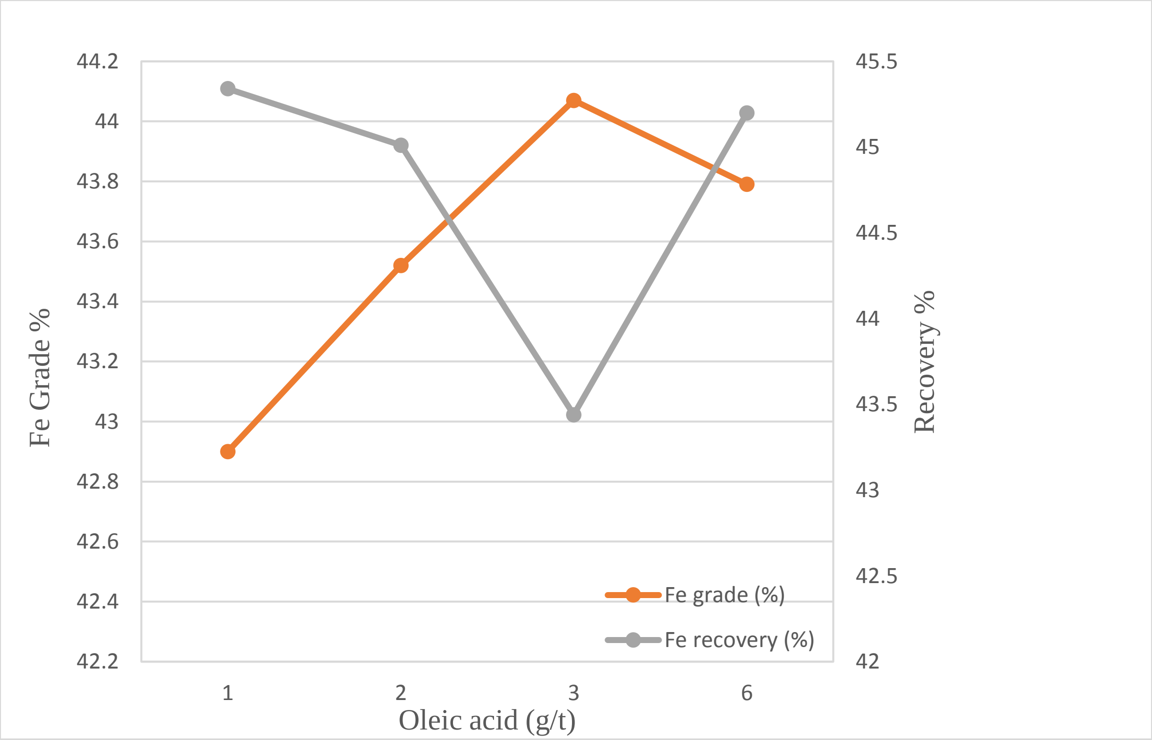


Figure 10 . Effect of collector quantity on Fe grade and recovery for -63 µm size fraction.

4.2.2. Effect of -75 µm and -63 µm particle size on Fe grade

Batch flotation tests on feed size fraction 100% -75 µm and -63 µm were carried to study the effect of size fraction on Fe concentrate grade and recovery. During these reverse flotation performance study, reagent dosage and conditioning time was kept similar. The computed % Fe assay and recovery values in each sieve size -75µm and -63µm fractions are based on Fe_2O_3 sink (concentrate) grade obtained by AAS analysis shown on table 12 and table 13. At these sieve size fractions, the iron concentrate grade and recovery percentages is compared in table 16, and in figure 13 and 14.

Table 16. Comparison of Fe grade and recovery at -75µm and -63µm size fractions.

Oleic acid quantity	Particle size (µm)	Weight of concentrates (g)	Assay of Fe_2O_3 (%)	Fe grade (%)	Fe recovery (%)
1	-75	154.5	61.24	42.86	42.36
	-63	155	61.3	42.9	45.34
2	-75	153.3	61.81	43.26	42.06
	-63	153.9	62.17	43.52	45.01
3	-75	150.2	62.14	43.49	40.48
	-63	150	62.96	44.07	43.4
6	-75	151.8	62.08	43.45	40.36
	-63	151	62.56	43.79	45.2

Flotation feed size fraction decreases, the highest assay of hematite is 62.96% Fe_2O_3 with a concentrate maximum grade of 44.07% Fe. When testing the feed -75µm size fraction (screen undersize), a recovery of 42.36% (on weight of product (sink) 154.5g) with a low grade of 42.86 % Fe was observed. The low recovery is may due to poor liberation occur with the feed sieve size. Feed sieve size decreases from -75μm to -63μm the grade of the Fe increase from 42.86 % to 44.07 % and a recovery percent decrease to 40.36%. Recovery and grade was slight difference to be decrease -63 μm size fraction range. Because as the sieve feed size decreases, the degree of liberation between hematite mineral particles and associated gangues are increases.

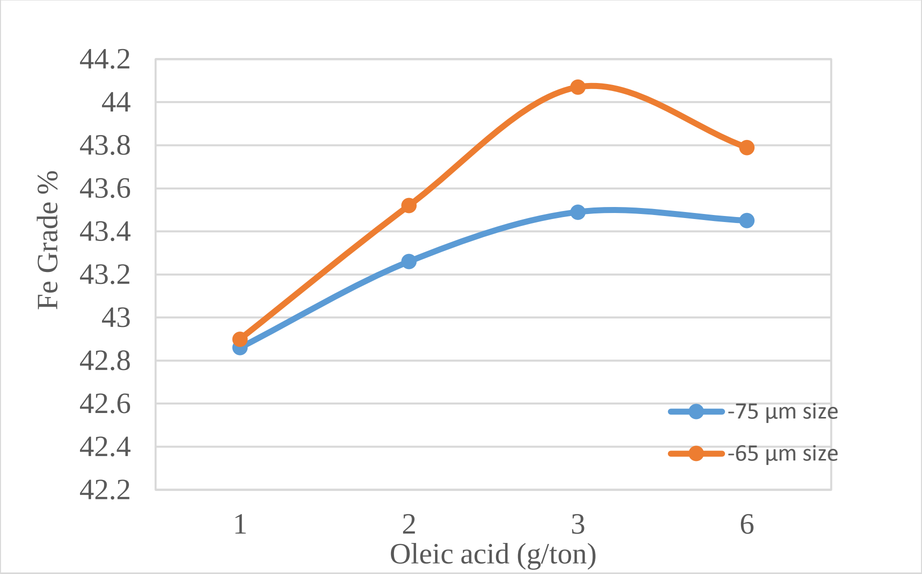


Figure 11. Grade of iron as a function of particle size range.

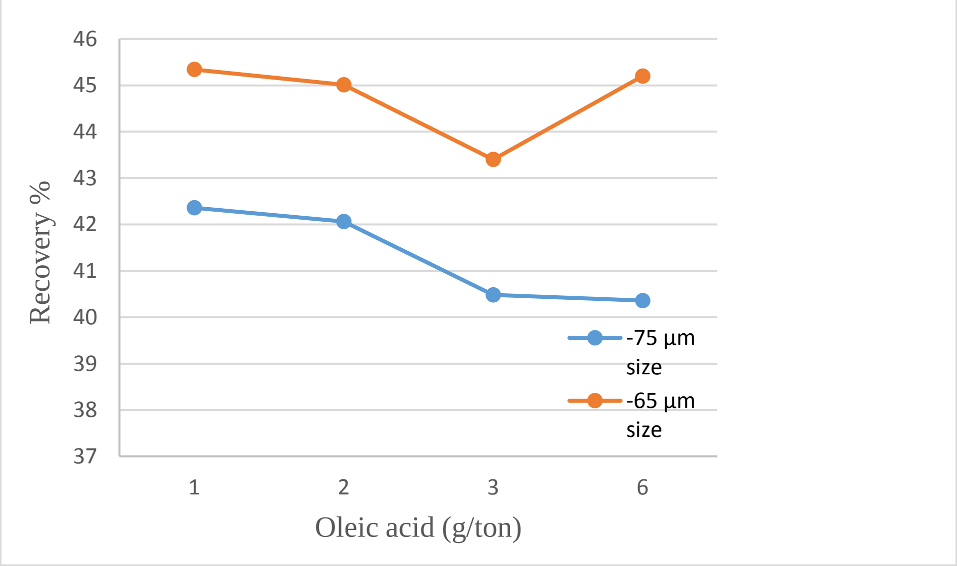


Figure 12. Recovery of iron as a function of particle size range.

CHAPTER FIVE

5. Conclusions and Recommendations

5.1. Conclusion

Beneficiation of hematite iron ore from Sekota, Amhara Region was studied by utilizing flotation via reverse anionic flotation method. The ore containing mainly hematite as valuable mineral. Two different particle size fractions used for sink (concentrate) and the results were compared in terms of grade and recovery. The following conclusions were drawn from the tests;

1. The chemical analysis revealed that the iron ore contains 51.7% Fe_2O_3 and 35.4% SiO_2. The result indicates that the iron ore is low grade with high amount of silica and alumina (7.79). However, the ore contains low level S and P.

2. The mineralogical revealed that the iron bearing minerals are hematite and goethite while quartz and kaolinite are present as gangue minerals. No magnetite was detected in the sample.

3. The iron minerals was obtained to have evenly distributed in the sieve size fractions (+850µm to -75µm). So, the liberation of hematite grains (19%) are poorly liberated, and 72% reports to middlings. Fine grinding will be necessary.

4. Increase the quantity of oleic acid have a slight increase in hematite grade concentrate. Considering the high hematite concentrate (62.96% Fe_2O_3), a maximum grade of 44.07% Fe with a recovery of 43.4% was obtained in the feed fraction adding 3g/t of the collector (oleic acid). At 6g/t, the grade was dropped, so there is no need longer added of oleic acid collector.

5. The sieve size fraction -75 and -63µm was subjected to flotation cell. A concentrate with Fe grade of % 44.07 and Fe recovery of % 45.34 on size fraction finer than 63 µm and -75µm with grade % 43.49 Fe and recovery % 42.36 was obtained. The flotation results of the -63µm feed sample was achieved a high grade and recovery of Fe compared to -75µm feed fraction. The result obtained shows that Sekota iron ore couldn’t be upgraded using reverse flotation separation method. This suggest that the Fe minerals are not liberated at a screen size of -63µm.

5.2. Recommendation

Based on the mineralogical and chemical analysis was carried out, the hematite ore is a complex ore. Therefore, the iron is finely associated with silica and alumina can’t be effectively liberated. Hence, it is recommended that finely grinding (under size -63µm) and de-slimmed techniques need to be employed.

Processing ore samples haven’t notable goethite minerals (detected by AAS). Therefore, more sensitive characterization technique should be applied to ascertain the actual content.

References

AusIMM. (2011). Monograph 27 - Cost estimation handbook.

Basson, E. (2014). World steel in figure, world steel association. Worldsteel [online].

Burt, K., Lockyer, C., McShane, K. and Fong, O.T. (Eds.). (2012). Cost estimation handbook, second ed. Australasian Inst. of Mining and Metallurgy. Monograph 27.

Elamin F., Elderrat H. and Abushaala S. (2019). The role of iron and steel industry for achieving the industrial development in Libya. Journal of engineering research and applied sciences (JERAS), 7th edition- volume (1)- December 2019.

Fardis, N. and Mehdi, I. (2017). Reagents types in flotation of iron oxide minerals: A review, Mineral Processing and Extractive Metallurgy Review.

Ferenczi, P. (2001). Iron ore, manganese and bauxite deposits of the northern Territory. Northern Territory Geological Survey, Report 13, December 2001. pp. 13-41. Government Printer of the Northern Territory Darwin.

Filippov, L. O., Severov, V. V. and Filippova, I. V. (2014). “An overview of the beneficiation of iron ores via reverse cationic flotation”, International journal of mineral processing, 127, 62-69.

Gaines, R.V., Skinner, C.W., Foord, E.E., Mason, B. and Rosenzweig, A., with sections by V.T. King. (1997). Dana’s new mineralogy - The system of mineralogy of James Dwight Dana and Edward Salisbury Dana: Eighth edition, John Wiley & Sons, New York, 1819 p.

GSA (2009). PIRSA minerals, Government of South Australia (GSA), Primary Industries and Minerals SA; 2009.

Gundewar, J.S. (2011). Iron and steel vision 2020. (Sumesh, M., Ed.), Nagpur: Aqua Process, pp. 36-87.

Gutzmer J. and Beukes, N.J. (2003). Iron and manganese ore deposits: Mineralogy, Geochemistry, and Economic Geology. Geology 4, 43-69.

Gutzmer, J. and Beukes, N.J. (2009). Iron and manganese ore deposits: mineralogy, geochemistry, and economic geology. In: Geology, vol. 4. Eolss Publishers Co. Ltd/UNESCO, Oxford, United Kingdom.

Haldar S.K. (2020). Introduction to mineralogy and petrology (Second Edition).

Industrial Policy Study and Research Department (IPSRD) and Ethiopian Institute of Technology -EiT, Mekele. (2017). Study of Development of Iron ore and Metallurgy Industries: Challenges, prospects, and policy/strategy options (2015-2025).

J.S. Thella, Mukherjee A.K. and Rajshekar, Y. (2010) Recovering iron ore values from iron ore slimes using cationic and anionic collectors, Proceedings Of The Xi International Seminar On Mineral Processing Technology (MPT-2010), Jamshedpur, 2010, pp. 247–254.

Kisore, K. (1993). Performance evaluation of Mozley mineral separator for treating coal, MTech thesis (unpublished), Indian School of Mines, Dhanbad, 61 p.

Klein, C. and Hurlbut, C. S. Jr. (1985). Manual of mineralogy, 21th edn. John Wiley and Sons, New York, 391p.

Koshy, P., Dankwah, J. B., Dankwah, J. and Dankwah, J. R. (2018). “Calcination Behaviour of Guizhou Siderite Ore and its Amenability to Reduction by Waste Polymers”, Ghana Journal of Technology, Vol. 3, No. 1, pp. 63- 68.

Libyan steel magazine - Issue No. 3 - March 2016. Research and development department of the Libyan Iron and Steel Company (Arabic language).

Maré, E., Beven, B. and Crisafio, C. (2015). Developments in non-magnetic physical separation technologies for hematitic/goethitic iron ore. Iron Ore, 309–338. doi:10.1016/b978-1-78242-156-6.00010-1.

Matloga, N.C. and Youlton, B.J. (2014). Mineralogical testwork on iron ore sample from Ethiopia. SGS Laboratories: SGS South Africa (Pty) Ltd.

Ma, M. (2012). Froth flotation of iron ores. International Journal of Mining Engineering and Mineral Processing 1 (2):56–61. doi:10.5923/j. mining.20120102.06.

Minaei Mobtaker, M. and Osanloo, M. (2015). Chaos in iron ore price prediction. Proceeding of 6th international conference on sustainable development in mineral industry (SDIMI), Greece, 30 June- 3 Julye, 2013, Taylor and Francis, United Kingdom, pp 7-11.

Murhula, E., Hashan, M. and Otsuki, A. (2023). Effect of solid concentration and particle size on the flotation kinetics and entrainment of quartz and hematite. Metals 2023, 13, 53. https://doi.org/ 10.3390/met13010053.

Mohapatra, B. K., Jena, S., Mahanta, K. and Mishra, P. (2008). “Goethite morphology and composition in banded iron formation, Orissa, India.” Resource Geology, 58, pp. 325–332.

Morris, R.C. and Ramanaidou, E.R. (2007). Genesis of the channel iron deposits (CID) of the Pilbara region, Western Australia. Aust. J. Earth Sci. 54, 733–756.

Nakhaei, F. and M. Irannajad. (2018). Reagents types in flotation of iron oxide minerals: A review. Mineral Processing and Extractive Metallurgy Review 39 (2):89–124. doi:10.1080/08827508.2017.1391245.

Olatunji, K. J. and Durojaiye, A. G. (2010). Determination of bond index Birnin-Gwari iron ore in Nigeria. Journal of Minerals and Materials Characterization and Engineering, Vol.9, No.7, Pp. 635-642.

Oliazadeh1, M. and Vazirizadeh, A. (2016). Removing impurities from iron ores: methods and industrial cases. Conference Paper 2016.

Ozcan, O. and Celik, İ.B. (2016). Beneficiation routes for upgrading iron ore tailings with teetered bed separator. Separation Science and Technology 51, 2844-2855.

Pattanaik A. and Venugopal R. (2018). Investigation of adsorption mechanism of reagents (Surfactants) system and its applicability in iron ore flotation – An Overview, Colloid and Interface Science Communications, Volume 25, 2018, Pages 41-65. https://doi.org/10.1016/j.colcom.2018.06.003.

Rachappa, S., Prakash, Y. and Amit, T. (2015). Iron ore recovery from low grade by using advance methods. Procedia earth and planetary science, 11, 195-197.

Rath, R.K. and Singh, R. (2007) Gravity concentration of iron ore. In: Processing of Iron Ore.

Rezvanipour, H., Mostafavi, A., Ahmadi, A., Karimimobarakabadi, M. and Khezri, M. (2018). Desulfurization of iron ores: Processes and challenges. Steel Research International, 89(7), 1700568. doi:10.1002/srin.201700568.

Roy, S. (2009). Recovery improvement of fine iron ore particles by multigravity separation. The Open Mineral Processing Journal 2, 17–30.

Roy, S., Das, A. and Mohanty, M. (2007). Feasibility of producing pellet grade concentrate by beneficiation of iron ore slime in India, Separat. Sci. Technol., 2007, vol. 42, no. 14, pp. 3271–3287.

Roy, S. and Das, A. (2008). Characterization and processing of low-grade iron ore slime from the Jilling area of India, Miner. Proc. and Extract. Metall. Rev., 2008, vol. 29, no. 3, pp. 213–231.

Sahin R. (2020). Beneficiation of low/off grade iron ore: areview. International Journal of Research -Granthaalayah, 8(8),328-335.

Sahoo, H., Rath, S. S., Rao, D. S., Mishra, B. K. and Das, B. (2016). “Role of silica and alumina content in the flotation of iron ores”, International Journal of Mineral Processing, 148, 83-91.

Seifelnassr, A. A. S., Moslim, E. M. and Abouzeid, A. Z. M. (2012). Effective processing of low-grade iron ore through gravity and magnetic separation techniques. Physicochem. Probl. Miner. Process. 48 (2), 567-578.doi.org/10.5277/ppmp120220.

Sis, H., Karaağaç, T., Birinci, M. and Kıyak, T. (2021). Enrichment of low-grade magnetite ore by magnetic and gravity separations: Effect of particle size. Bilimsel Madencilik Dergisi , vol.60, no.1, 31-39.

Suresh, N, Tripathi, D. and Venkatesh, A S. (2001). Characterisation and beneficiation of Boula-Nuasahi chromites, in proceedings international conference on challenges in coal and mineral beneficiation (eds: R Venugopal and V K Saxena), pp 23-28 (Tata McGraw-Hill Publishing Company Limited).

Summerfield, D. (2020). Australian resource reviews: Iron Ore 2019. Geoscience Australia, Canberra.

Tsutomu, N., Norihito, Y., Takeshi, F. and Yuta, T. (2015). Beneficiation plants and pelletizing plants for utilizing low grade iron ore.

UNCTAD (2016). The iron ore market 2016 flyer. Retrieved December 13, 2016, from http://unctad.org/Sections/ditc_commb/docs/suc2016_IronOre_Flyer_en.pdf.

US EPA (1994). Extraction and beneficiation of ores and minerals. Technical resource document: iron. Vol 3. U.S. Environmental Protection Agency (USEPA), EPA 530-R-94-030, NTIS PB94-195203, pp. 122.

USGS. (2015). Iron and steel mineral commodity summary. U.S. Geolocial Survey.

USGS. (2021). Mineral commodity summaries 2021.

Vidyadhar, A. and Singh, R. (2007). Froth flotation and its application to concentration of low grade iron ores.

Weekes G.J., and Robertson A.C. (1993). Magnetite production by commercial minerals limited in Queensland and New South Wales. In: Woodcock J.T. and Hamilton J.K. eds. Australasian Mining and Metallurgy; the Sir Maurice Maw by Memorial Volume, pp. 1393–1396. Australasian Institute of Mining and Metallurgy Monograph 19.

Yellishetty, M., Mudd, G., Mason, L., Mohr, S., Prior, T. and Giurco, D. (2012). Iron resources and production: technology, sustainability and future prospects. Prepared for CSIRO Minerals Down Under Flagship, by the Department of Civil Engineering (Monash University) and the Institute for Sustainable Futures (University of Technology, Sydney), October 2012. ISBN 978-1-922173-46- 1.

Zhang, S. and Yin, H. (2007). Current situation and existing problems of blast furnace iron making in China. Iron and Steel 42 (9), 1-8 (In Chinese).