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ADDIS ABABA INSTITUTE OF TECHNOLOGY

CENTER FOR ETHIO-MINES DEVELOPMENT

M.Sc. PROGRAM IN MINERAL ENGINEERING

TITLE: CHARACTERIZATION AND ANIONIC REVERSE FLOTATION OF BOREDA KAKISHA IRON ORE, MELO KOZA, GOFA ZONE, SOUTH ETHIOPIA REGION

By

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June 9, 2025

AAU, Ethiopia

APPROVAL SHEET

TITTLE: CHARACTERIZATION AND ANIONIC REVERSE FLOTATION OF BOREDA KAKISHA IRON ORE, MELO KOZA, GOFA ZONE, SOUTH ETHIOPIA REGION

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DECLARATION

I declare that this project work, entitled “Characterization and Anionic Reverse Flotation of Boreda Kakisha Iron Ore, Melo Koza, Gofa Zone, South Ethiopia Region” is my own original work submitted for the award of Partial Fulfillment of the Requirements for the Degree of Master of Engineering in Mineral Engineering at CEMD, Addis Ababa Institute of Technology, AAU. I further declare that this work has never been submitted in any form to any university or other institution for consideration for another degree or diploma. The required acknowledgment has been given to the data used from other people's published and unpublished work.

Declared by:	Confirmed by:
Name: Wondishaw Wosine Wolka	Name: Dr. Kebede Gamo
Signature: -------------------------	Signature: ------------------------
Date: ----------------------------	Date: ---------------------------

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List of Acronyms and Abbreviations

AAiT --- Addis Ababa Institute of Technology

AAS --- Atomic Absorption Spectroscopy

AAU -- Addis Ababa University

AMU -- Arba Minch University

BK -- Boreda Kakisha

BKIC ---Boreda Kakisha Iron Concentrate

BKIO -- Boreda Kakisha Iron Ore

CEMD -- Center for Ethio – Mines Development

CMC --- Carboxymethyl Cellulose

GPS -- Global Positioning System

GSE -- Geological Survey of Ethiopia

GZWIMDD -- Gofa zone water, irrigation and mines development department

JXSCMC --- Jiangxi Shicheng Mining Company

MKWWIMDO --- Melo koza woreda water, irrigation and mines development office

ml --- millilitres

Mt --- Million tonnes

µm -- micrometers

Plc --- Private limited company

rpm --- revolutions per minute

USGS -- United States Geological Survey

XRD -- X-Ray Diffraction

XRF --- X-ray Fluorescence

ABSTRACT

Iron ore, a mineral or rock from which metallic iron is extracted, mainly consists of iron in the form of oxides, hydroxides, carbonates, and sulfides. The presence of impurities like silica, alumina, phosphorus, sulfur, and trace elements can affect the ore's quality and suitability for iron and steel production, often requiring processing to reduce impurities. Anionic reverse flotation was used in this work to examine the impurity removal procedure for low-grade BK iron ore. BK iron ore sample is the low-grade and contains Fe_2O_3 (26.26 – 31.20 wt%, average 29.73 wt %) as iron-bearing mineral and SiO_2 (43.16 – 51.78 wt %, average 45.78 wt %) and Al_2O_3 (11.46 – 13.60 wt %, average 12.42 wt %) as major associated impurities based on AAS analyses. The XRD mineralogical analysis showed that the iron ore sample primarily consisted of hematite and goethite as the main valuable minerals, with quartz being the dominant gangue mineral. The effect of varying particle size range was studied in the flotation test. BK iron ore was upgraded through anionic reverse flotation using the following conditions: 2 ml of 2% corn starch solution, 2 ml of oleic acid, 2 ml of 1% calcium chloride solution, and 2 ml of pine oil, pH of 11 and particle size of -250+150 microns, which is optimal particle size range because of highest concentrate grade due to mineral liberation and less affected by slimes. Under these conditions, a concentrate with 23.92% Fe grade and 97.21% iron recovery was achieved. The average iron (Fe) grade of 20.78% in the ore was increased to 23.17% in the concentrate, while the average iron oxide (Fe_2O_3) grade of 29.73% in the ore was upgraded to 33.15% in the concentrate. The percentage increase in iron content from the initial ore grade to the final concentrate grade was calculated to be 11.5%. This means that the average iron concentration in the concentrate has increased by approximately 11.5% compared to the original ore. However, further separation is necessary to enhance the iron content and eliminate impurities to meet the minimum iron grade specifications needed for blast furnaces and steel manufacturers.

Key words: Iron ore; Low grade ore; Beneficiation; Impurities; Cationic reverse flotation; Anionic reverse flotation.

CHAPTER ONE

1. INTRODUCTION

1.1 Background

Iron (Fe) is the fourth most common element in the crust of the earth, after oxygen, silicon and aluminum. It makes up roughly 5.6% of the earth's crust, making it the second most plentiful metal after aluminum. Iron is among the most extensively used metals in the world and the main raw material for the steel industry (Mishra & Swain, 2020). Iron never occurs in its pure form in nature but is typically found combined as oxides, hydroxides, carbonates, sulfides, and other compounds. Iron ore is a mineral or rock from which metallic iron can be economically extracted, serving as the primary raw material for steel and iron production, with about 98% used in steelmaking and the remaining 2% for various other applications (Harraz, 2023). Though iron is present in over 300 minerals, pyrite (FeS_2), siderite ($FeCO_3$), limonite ($FeO(OH).n(H_2O)$), goethite ($FeO(OH)$), magnetite (Fe_3O_4), and hematite (Fe_2O_3) are the primary sources of iron (Journal et al., 2018; Harraz, 2023). Around 82 billion tons of metallic iron are available for extraction from the world's 170 billion tons of iron ore (USGS, 2017; Indian Bureau of Mines, 2018). The top ten countries in the world in terms of iron ore resources are Australia, Russia, Brazil, China, India, Canada, Ukraine, Sweden, USA, and Iran (Mishra & Swain, 2020). The global quantity of iron ore reserves in 2021 is 179,230 Mt. Out of this amount Australia 51,000 Mt (28.5%), Brazil 34,000 Mt (19.0%), Russia 25,000 Mt (13.9%), China 20,000 Mt (11.2%), Ukraine 6,500 Mt (3.6%), Canada 6,000 Mt (3.3%), India 5,500 Mt (3.1%), United States 3,000 Mt (1.7%), Iran 2,700 Mt (1.5%), Peru 2,600 Mt (1.5%) and other countries 22,930 Mt (12.8%). Australia led the world in 2021 with 900 Mt (34.6%) of iron ore production, followed by Brazil (380 Mt, 14.6%), China (360 Mt, 13.8%), India (240 Mt, 9.2%), Russia (100 Mt, 3.8%), Ukraine (81 Mt, 3.1%), Kazakhstan (64 Mt, 2.5%), South Africa (61 Mt, 2.4%), Canada (57 Mt, 2.3%), Iran (50 Mt, 1.9%), and other nations with 244 Mt (9.6%) (Bedinger, 2020; Harraz, 2023). Iron ore deposits in Ethiopia are distributed across various regions, including Indaselassie and Mentebteb, Adwa, Axum, and Enticho (Tigray); Bikilal, Gordana-Dima, Koree, Worakalu, and Chago (Wollega); Melka Arba (Bale); Werfedo (Harar); Dime; as well as Gamalucho and Garo (Jimma), Dambova, and several other locations (GSE, 2020). According to Agegnehu et al. (2023) BK iron deposit is estimated to contain approximately 950.84 Mt of iron ore with an average grade of 32% iron, where the iron mineralization is primarily hosted in rhyolite and composed mainly of goethite ($FeO(OH)$). High-quality iron ore, commonly referred to as direct shipping ore, typically contains more than 60% iron in the form of hematite or magnetite, allowing it to be directly introduced into blast furnaces for iron production with little to no additional processing

(Harraz, 2023). Because high-grade iron ore sources are being depleted and the need for steel production is rising globally, low-grade iron ore beneficiation has recently attracted a lot of attention (Behnamfard & Khaphaje, 2019). Medium and low-grade iron ores in Ethiopia need beneficiation to enhance iron content and remove impurities, yet there are no significant studies focused on iron ore extraction and beneficiation in the country, forcing Ethiopia to import iron ore products.

Flotation is a physical-chemical method of separation that takes use of the variations in surface properties between valuable and undesirable gangue minerals (Napier-Munn & Wills, 2006). Iron ore flotation is mainly divided into direct flotation, which floats iron minerals, and reverse flotation, which floats gangue and is further classified into cationic and anionic types based on the collector used, with reverse flotation currently being the most widely employed technique for iron ore concentration. Compared to cationic reverse flotation, anionic reverse flotation offers advantages such as lower sensitivity to slimes, reduced collector costs, and greater selectivity due to calcium's dual role in activating quartz and depressing iron ore (Zhang et al., 2021). The choice of processing method is influenced by the ore's type and complexity, along with considerations like simplicity of the process, cost efficiency, processing speed, and environmental effects (Dehghani et al., 2022). Based on the above and other reasons anionic reverse flotation was preferred for BK iron ore to upgrade the iron content and minimize the impurities.

1.2 Statement of the problem

Although Ethiopia has abundant iron resources, they are often of low quality, containing high levels of unwanted minerals, and require further processing. The country faces an imbalance between iron ore and its product's supply and demand due to industrial growth and rising living standards. As a result, Ethiopia is compelled to use imported iron ore products. Relying solely on imported resources to achieve the desired societal-based economic development is challenging. To address this, processing local medium and low-grade iron ore is essential to reduce dependence on imports and meet the growing demand. Iron ore processing has been widely researched globally, but studies in Ethiopia, particularly around BK, are limited. According to Agegnehu et al. (2023), the BK iron deposit is estimated to contain around 950.84 million tonnes of ore with an average iron content of 32%, which is low grade. This indicates a strong potential for the deposit to serve as a domestic iron source for Ethiopia. However, the absence of modern processing research has left this potential mostly unexploited. Historically, local communities have extracted the ore for use in making traditional agricultural tools, but it has yet to be developed or utilized on a modern, industrial scale. This study seeks to fill existing gaps by examining how different particle size ranges influence anionic reverse flotation in order to enhance iron content through impurity removal, offering a basis for iron ore beneficiation in Ethiopia, specifically targeting the BK region.

1.3 Objectives

1.3.1 General objective

The general objective of this study was to characterize the BK iron ore from Melo Koza in the Gofa Zone of South Ethiopia and to evaluate the effectiveness of anionic reverse flotation for enhancing the quality of ore by efficiently eliminating impurities.

1.3.2 Specific objectives

In order to accomplish the general objective, the following specific objectives must be met:

- To analyze the mineralogical composition of ores.
- To analyze the chemical composition of ores and concentrates.
- To determine the optimal particle size range for effective anionic reverse flotation of iron ore.

1.4 Research questions

- What are the mineralogical compositions of ore?
- What are the chemical compositions of ores and concentrates?
- What is the optimal particle size range for maximizing grade of iron during anionic reverse flotation of ore?

1.5 Significance of the study

The study of characterization and anionic reverse flotation of BK iron ore is significant for several reasons, as it addresses key challenges and provides solutions for improving the efficiency and sustainability of the iron ore beneficiation process. Anionic reverse flotation significantly improves iron ore quality by selectively removing gangue minerals such as silicates and alumina, resulting in a higher-grade iron concentrates with fewer impurities, which is crucial for efficient downstream processing in steel production. The method utilizes anionic collectors like fatty acids to effectively target gangue minerals, optimizing flotation conditions and reagent use, which enhances process efficiency, reduces iron losses, and minimizes costs. This technique is particularly valuable in addressing the depletion of high-grade ore reserves by efficiently processing lower-grade materials, prolonging the lifespan of existing deposits. Additionally, anionic reverse flotation is more environmentally sustainable than traditional methods, using less harmful reagents and generating less waste, which reduces environmental impact and tailings disposal concerns. The higher-grade iron concentrates produced also bring significant economic benefits, improving market competitiveness and expanding the potential for low-grade ore extraction. Moreover, the study's findings advance both academic knowledge and industrial practices, contributing to better flotation operations and increased productivity. In regions like BK, such advancements can positively impact local economies by creating jobs, improving resource management, and supporting sustainable mining. On a global scale, the improved beneficiation process has a direct influence on the steel industry, ensuring a more reliable and sustainable supply of high-quality iron ore for steel production.

1.6 Scope of the study

This study focuses on characterizing BK iron ore from Melo Koza, Gofa zone, South Ethiopia, and applying anionic reverse flotation, including steps such as sample collection, crushing and grinding, mineralogical and chemical analysis, flotation tests at varying particle sizes, and evaluation of iron grade and recovery to determine the process's efficiency and economic feasibility.

1.7 Description of iron deposit area

1.7.1 Location and accessibility

The ore deposit is situated 580 kilometers from Addis Ababa, the capital of Ethiopia, and 117 kilometers from Sawla, the capital of Gofa zone. It is accessed through Addis Ababa - Wolayta Soddo – Sawla – Melo Koza – Boreda Kakisha.

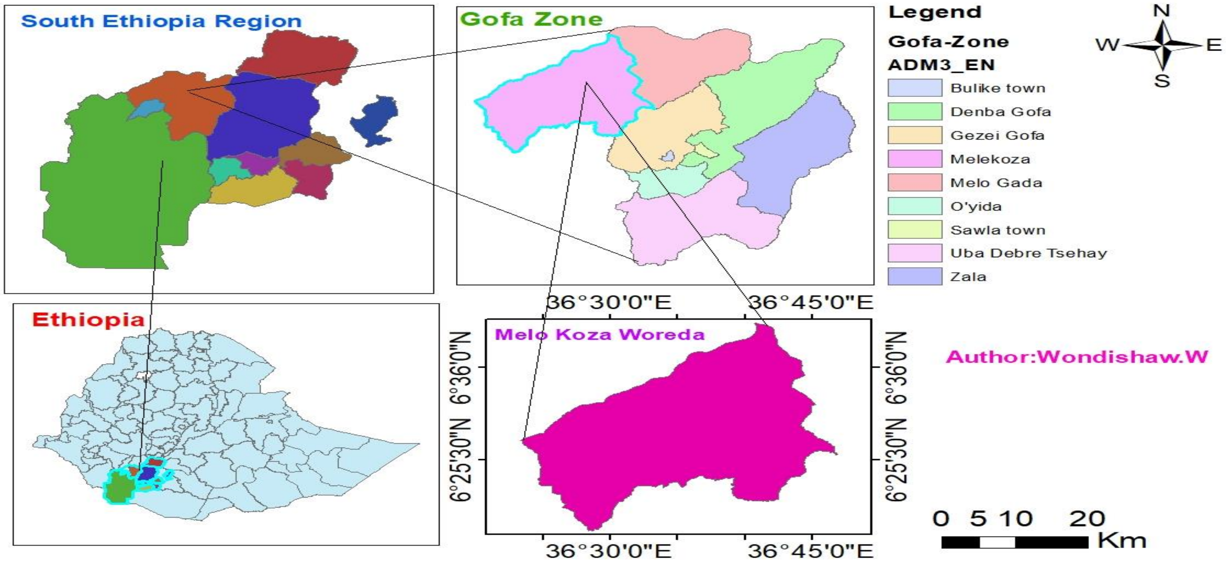

Figure 1: Location map of Melo Koza

1.7.2 Physiography of the deposit area

The region is characterized by a rough and irregular topography that includes mountains, hills, and valleys. Vegetation cover is generally sparse, with dominant plant species including eucalyptus, junipers, Podocarpus gracilior, Veronica, and various dwarf plants. The main agricultural products cultivated in the area are maize, teff, sorghum, coffee, chat and others (Agegnehu et al., 2023).

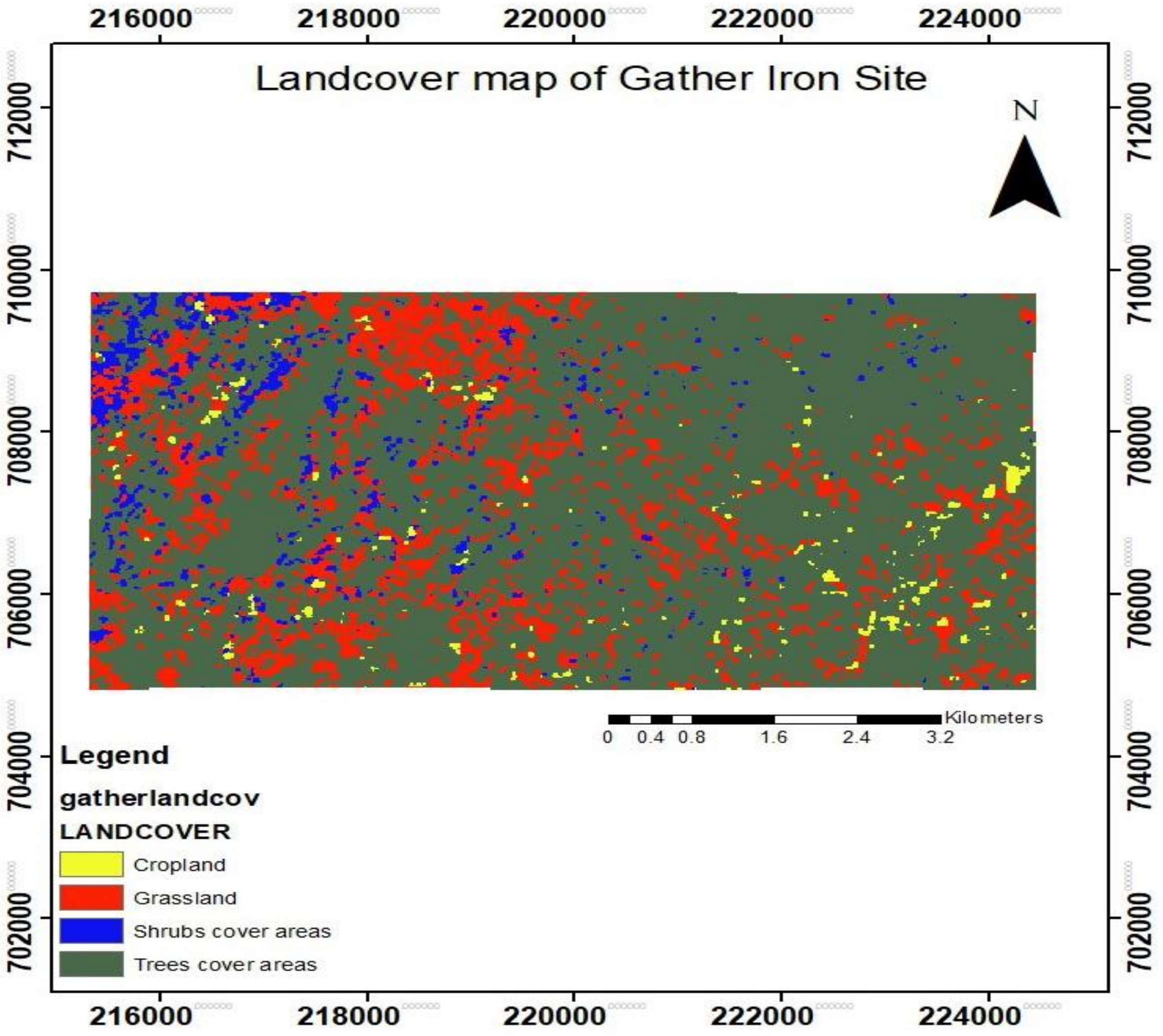

Figure 2: Land cover map of Gather iron site (Agegnehu et al., 2023)

1.7.3 Drainage of the deposit area

The area is drained by two major rivers, Endala and Sala, which flow southward in the western part, fed by numerous fresh tributaries originating from nearby rocks, and the overall drainage pattern is dendritic.

CHAPTER TWO

2. LITERATURE REVIEWS

2.1 Geology

2.1.1 Regional geology

Ethiopia's geology is characterized by a diverse rock record ranging from Precambrian to Recent, comprising Precambrian metamorphic and igneous rocks (25% of landmass), Paleozoic–Mesozoic sedimentary basins (25%), and extensive Cenozoic volcanic rocks and sediments (50%). The Precambrian basement, divided into the Lower Complex (Mozambique Belt) and Upper Complex (Arabian-Nubian Shield), features high-grade gneisses and low-grade volcano-sedimentary belts along major shear zones, with rock ages mostly between 900–500 Ma. Major sedimentary basins; namely, Ogaden, Abay, and Mekele are formed during the breakup of Gondwanaland, accumulating thick marine and non-marine sequences. Cenozoic volcanism began in the late Eocene with massive flood basalt eruptions linked to mantle plumes, covering vast areas and reaching volumes of ~300,000 km³, followed by shield volcanoes and later bimodal volcanic activity along the tectonically active Main Ethiopian Rift (Tadesse Alemu, 2012).

2.1.2 Local geology

In the Boreda kakisha, especially gazer area, the dominant lithologies exposed along river cuts, hillsides, and road cuts are rhyolite, basalt, granite, andesite, and ignimbrite, with rhyolite being the most prevalent and often associated with iron-hosted bodies (Agegnehu et al., 2023).

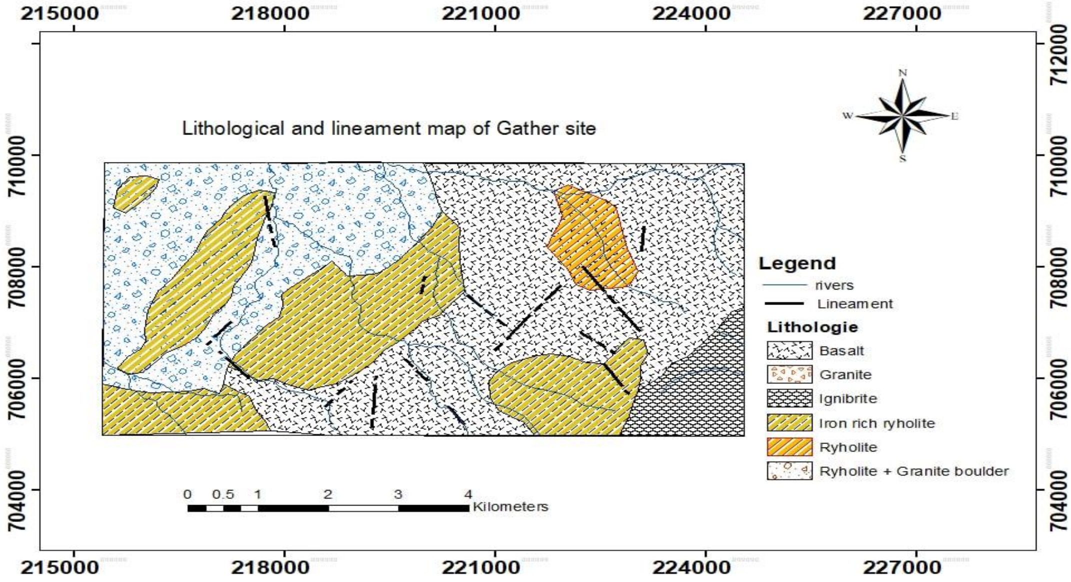

Figure 3: Geological map of Boreda Kakisha iron deposit area

2.1.3 Lithological Descriptions

There are different lithological units were exposed in BK iron deposit area such as, basalt, andesite, rhyolite, granite, and ignimbrite.

(A) **Basalt units:** Basalt is a dark-colored, fine-grained, mafic extrusive igneous rock composed mainly of pyroxene and plagioclase, with low silica content, and it forms from the rapid cooling of low-viscosity lava, making it the most common volcanic rock on Earth. In the study area it has dark grey to black color, and aphanitic texture with vesicular nature.

(B) **Andesite units:** Andesite is a fine-grained, extrusive igneous rock with intermediate silica content, typically gray in color, composed mainly of plagioclase feldspar along with smaller amounts of amphibole or pyroxene. In the study area this units have light grey color, coarse grain and form large boulders. In some part this unit associated with trachyte unit.

(C) **Rhyolite units:** Rhyolite is a light-colored, fine-grained volcanic igneous rock rich in silica, primarily made up of quartz, feldspar, and mica, and it forms when thick, high-viscosity lava cools quickly at or near the Earth's surface. In the majority of the region, it was exposed.

(D) **Granite:** Granite is a coarse-grained, light-colored, intrusive igneous rock that is mostly made up of mica, feldspar, and quartz. It has a high silica concentration. It is created by the gradual cooling of lava found deep within the Earth's crust.

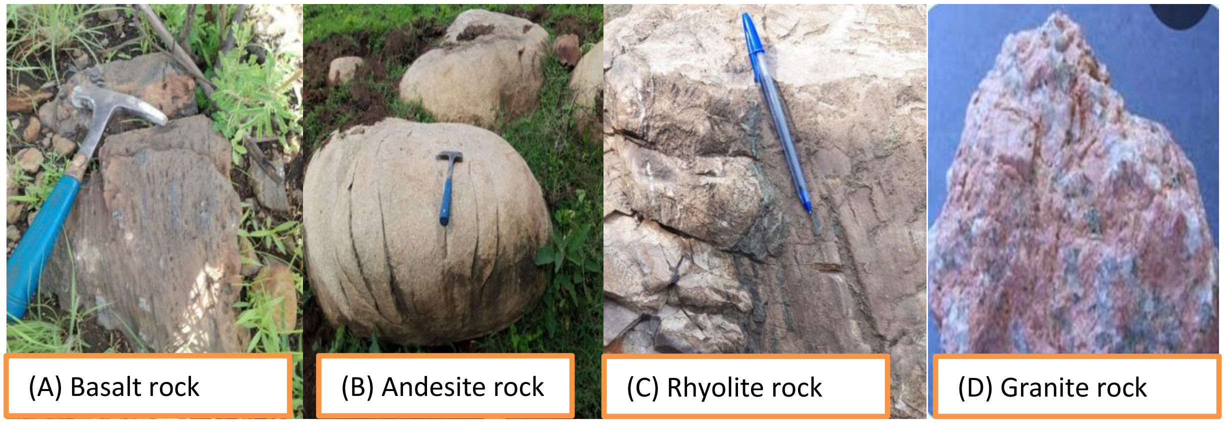

Figure 4: Lithological units exposed in BK iron deposit

2.2 Iron ore

Iron ore is a mineral or rock that can be economically mined for metallic iron. Approximately 98% of it is used in the production of steel, with the remaining 2% going to other uses, making it the primary raw material for the production of iron and steel (Harraz, 2023). The most common iron-bearing minerals used in industry are hydroxides and oxides, including limonite ($FeO(OH).nH_2O$), goethite ($FeO(OH)$), hematite

(Fe_2O_3), and magnetite (Fe_3O_4), but siderite ($FeCO_3$), a carbonate mineral, is utilized less (Filippov et al., 2014). Iron ore is primarily used to produce steel, pig iron, and sponge iron, and also serves various industries including foundry, glass manufacturing, coal washing, ferroalloys, cement, and vanaspati production (Indian Bureau of Mines, 2018). Iron ore is converted into iron through beneficiation and extraction, with high-grade ores generally needing only crushing and washing prior to extraction, while low-grade ores require beneficiation to increase their iron content (Siaplay, 2017). Iron and steel combined make up the largest manufactured products globally, playing a crucial role in every industry and being essential to all aspects of modern civilization (Sethumadhav, n.d.). Iron and steel are the main drivers of industrial development in every nation, and the health of the sector has a significant impact on a nation's economic standing (Indian Bureau of Mines, 2018). The steel industry is currently shifting toward increased use of direct reduction methods alongside electric furnace production, which demands iron ore with less than 2% silica (SiO_2), 0.1% sulfur, 0.01% phosphorus, and 2% aluminum (Nakhaei & Irannajad, 2018). For optimal blast furnace performance, the alumina content in the ore feed should be kept below 2% and the alumina-to-silica ratio should be less than 1.0 (Mehrotra, 2019; Ghosh et al., 2021). The goal of upgrading iron-bearing ore is to lower the levels of impurities such as quartz, sulfur, phosphorus, and aluminum in the iron concentrate to below 2%, 0.1%, 0.01%, and 2%, respectively (Tarekegn, 2023).

2.2.1 Iron ore reserves and production

According to USGS data in 2024, global iron ore reserves are estimated at approximately 190.942 billion metric tons, with the largest reserves held by Australia, followed by Brazil, Russia, China, Ukraine, Canada, India, Iran, the United States, and other countries (Table 1). In 2023, global iron ore production was estimated at 2,544 million tons with iron content of 1,561.5 million tons, led by Australia as the top producer, followed by Brazil, China, India, and other countries (Table 1).

Table 1: World Mine Production and Reserves (USGS, 2024)

No	**Name of countries**	**Mine production (Thousand metric tons)**				**Reserves (million metric tons)**	
		Iron ore		Iron content		Iron ore	Iron content
		2022	2023	2022	2023		
1	United States	39,000	44,000	24,700	28,000	3,100	1,300
2	Australia	944,000	960,000	584,000	590,000	58,000	27,000
3	Brazil	435,000	440,000	276,000	280,000	34,000	15,000
4	Canada	69,000	70,000	41,400	42,000	6,000	2,300

5	Chile	17,700	18,000	11,100	11,000	NA	NA
6	China	272,000	280,000	170,000	170,000	20,000	6,900
7	India	251,000	270,000	156,000	170,000	5,500	3,400
8	Iran	78,300	77,000	51,300	50,000	3,300	1,500
9	Kazakhstan	53,600	53,000	8,890	8,800	2,500	900
10	Mauritania	12,700	13,000	7,950	8,100	NA	NA
11	Mexico	10,800	12,000	6,800	7,600	NA	NA
12	Peru	19,300	19,000	12,900	13,000	2,600	1,200
13	Russia	84,200	88,000	55,800	58,000	29,000	14,000
14	South Africa	63,700	61,000	40,500	39,000	990	620
15	Sweden	38,900	38,000	27,700	27,000	1,300	600
16	Turkey	17,700	17,000	10,700	10,000	152	99
17	Ukraine	34,100	36,000	21,300	22,000	6,500	2,300
18	Other countries	57,200	48,000	32,200	27,000	18,000	9,500
World total		2,498,200	2,544,000	1,539,240	1,561,500	190,942	86,619

NA: Not available

2.2.2 Iron resource potential of Ethiopia

The major Ethiopian locations known for their significant iron ore resources are Melka Arba, Shire, Bikilal, Sekota, Mertolemaryam, and Mekaneselam. These areas have a high potential for economically viable iron ore deposits (Table 2).

Table 2: Location and distribution of iron ore resources and reserves of Ethiopia (Getahun, 2023)

Deposit Name	Location	Ore minerals	Estimated iron ore (million ton)
Bikilal	Wollega	Magnetite	57
Gamalucho	Kaffa	Magnetite	12.5
Garao	Kaffa	Hematite & limonite	12.5
Melka Arba	Sidamo	Hematite, Magnetite & Limonite	111
Shire	Tigray	Goethite & Limonite	192
Enthicho	Tigray	Limonite	14.23
Metro lemareyam		Hematite & Goethite	32.2

Sekota	Waghemera zone	Hematite & Goethite	57.01
Zikuala	Waghemera zone	Hematite & Goethite	31.4
Abergele	Waghemera zone	Hematite & Goethite	15.73
Mekane Selam	S. Wello Zone	Hematite & Goethite	184

2.2.3 Uses of iron ore and its products

Iron ore is primarily used to produce steel, an essential material that supports a wide range of global industries including construction, transportation, machinery, energy, healthcare, defense, and the arts by enabling the development of infrastructure, vehicles, appliances, tools, medical equipment, packaging, and artistic works (World Steel Association, 2023).

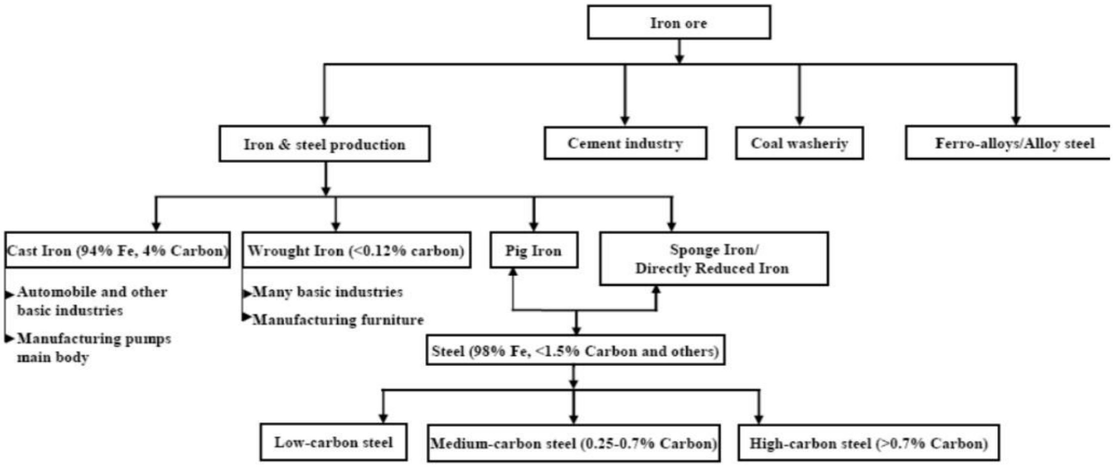

Figure 5: Uses of iron ore (Tarekegn, 2023)

2.2.4 Main iron ores

Iron is found in over 300 minerals, primarily in oxide forms such as magnetite (Fe_3O_4), hematite (Fe_2O_3), and goethite ($FeO(OH)$), with other forms including carbonates, hydroxides, sulphides, and silicates (Tarekegn, 2023).

a) Magnetite

The iron oxide mineral magnetite (Fe_3O_4), often known as lodestone is opaque, magnetic, usually black, brown, or dark gray, has a metallic sheen, crystallizes in an isometric system, and has a hardness of 5.5 to 6.5. Steel producers greatly value the concentrate made from this iron ore, which is the second most prevalent after hematite and requires sophisticated processing to get 68–70% iron (Harraz, 2023). Magnetic separation is the most often used method for the beneficiation of iron ores, including magnetite and hematite (Dehghani et al., 2022). Wet and dry low-intensity magnetic separators (LIMS) are used to process ores containing minerals with strong magnetic properties, such as magnetite and titanomagnetite (Nakhaei & Irannajad, 2018).

b) Hematite

Hematite, the most significant iron ore mineral and main source for industries, is composed of iron oxide with traces of titanium, has a reddish color comes from the Greek word "haima" meaning blood, a reddish streak, a specific gravity of 4.9 to 5.3 g/cm^3, a metallic to dull luster, and exhibits weak magnetic and paramagnetic properties (Harraz, 2023).

c) Goethite

Goethite (FeO(OH)) is produced by weathering of other iron-rich minerals. It typically appears in yellow, brown, or reddish-brown colors, contains about 62.9% iron, has a specific gravity ranging from 3.3 to 4.3, and a hardness of 5.0 to 5.5. Although it is an important iron ore mineral, it contains less iron compared to hematite and magnetite.

d) Limonite

Limonite is a brown iron ore formed through the weathering of iron minerals, consisting of amorphous Fe-hydroxides with varying water content, and is commonly found in rocks with brown, yellow, or tan colors, resulting from the oxidation of ferrous iron (Fe^{2+}) to trivalent iron (Fe^{3+}) in a process known as limonitization, often forming an iron-rich crust above hematite and siderite deposits, and serving as an important source of low-grade iron and nickel ore (Harraz, 2023). Limonite, a hydrated oxide of iron, is commonly brown to ocherous yellow, though it may be black, dark brown, or reddish brown, commonly known as "brown iron ore," with a specific gravity ranging from 3.3 to 4.3, a hardness of 5.5, and containing 10 to 14.5 percent combined water; it is a secondary mineral formed by the alteration of other minerals, occurring as thick capping due to weathering and hydration of the underlying ore-body and can be changed into hematite or magnetite upon calcination (Indian Bureau of Mines, n.d.).

e) Siderite

Siderite, also known as "spathic ore," is an iron carbonate with a color range from ash grey to brown, often featuring yellow and red stains due to oxidation and hydration, a specific gravity of 3.8, a hardness of 3.5 to 4, crystallizes in the rhombohedral division of the hexagonal system, and typically occurs as sedimentary or replacement deposits (ibid.). Siderite (FeCO3) is a valuable mineral, which containing 48% iron, and is free from sulfur, phosphorus, zinc, and manganese (Temesgen, 2023)

f) Pyrite

Pyrite is a metallic-lustered, golden-yellow mineral with a specific gravity of 5.1 and a hardness of 6 to 6.5. It can be used as an iron ore when sulfur is removed by calcination, though it is not currently a popular choice (Indian Bureau of Mines, n.d.).

Table 3: Common iron- bearing minerals and its properties (Ram Mohan et al., 2011)

Mineral	**Composition**	**Iron content (%Fe)**	**Specific gravity**	**Hardness (Moh's scale)**	**Color**
			Oxides		
Hematite (Alpha) and Martite	Alpha-Fe_2O_3	70	4.9 – 5.3	5.5 – 6.5	Steel grey to red
Turgite	2 $Fe_2O_3.H_2O$	66.1	4.2 – 4.6	6.5	Brown to red
Goethite	$Fe_2O_3.H_2O$	62.9	3.4 – 4.2	5 – 5.5	Brown to red
Limonite	2 $Fe_2O_3.H_2O$	60	3.4 – 4.2	5 – 5.5	brown to yellow
Lepidocrosite	$Fe_2O_3.H_2O$	62	4.09	5	Brown to reddish brown
Ilmenite	$FeTiO_2$	36.8	4.5	5 – 6	Black to brownish black
Maghemite	Gamma-Fe_2O_3	69.9	4.88	5	Brown
Magnetite	Fe_3O_4	72.4	5.17	5.5 – 6.5	Black, blue or brown black
			Carbonates		
Siderite	$FeCO_3$	48.2	3.7 – 3.9	3.5 – 4.5	Ash grey to brown
			Sulphides		
Pyrite	FeS_2	46.6	4.8 – 5.1	6 – 6.5	Brass yellow
Marcasite	FeS_2	46.6	4.9	6 – 6.5	Light brass yellow
Pyrrhotite	FeS_2	61.6	4.4 – 4.65	3.5 – 4.5	Bronze yellow
			Silicates		
Chamosite	$(Mg.Fe.Al)_6$ $(Si.Al)_4$	33.42	3 – 3.5	3	Green to light yellow

2.2.5 Impurities

The impurities in iron ore consist of slag- forming constituents and other minerals like arsenic, titanium, vanadium, copper, lead, zinc, tin, chromium, nickel, phosphorus, sulfur, etc. Silica is the primary acid ingredient of slag-forming minerals, whereas alumina, lime, and magnesia produce the basic slag. For optimum performance of the blast furnace, these two constituents in the burden must be well balanced in accordance with a limited and well-defined ratio (Indian Bureau of Mines, n.d.). There are four main slag forming constituents in iron ores, these include the acidic oxide silica (SiO_2) and the basic oxides lime (CaO) and magnesia (MgO) as well as the neutral alumina (Al_2O_3) (Siaplay, 2017). The iron ore contains alumina-bearing minerals such as gibbsite, kaolinite, montmorillonite, illite, diaspore, and corundum (Fan et al., 2020). Increased coke consumption and decreased blast furnace productivity are the results of using high alumina slag, which is extremely viscous and requires a larger amount of flux (10% MgO) and comparatively bigger slag volumes (Scientist et al., 2013).

Table 4: Gangue minerals associated with iron ores (Pattanaik & Venugopal, 2018)

Associated gangues	Elemental forms
Alumina	gibbsite (hydrated aluminum oxide), kaolinite (a layered aluminosilicate mineral of general chemical formula $Al_2\ Si_2O_3(OH)_4$) and other gangues like ferruginous clay in minor quantities
Silica	Quartz, quartzite, chert, kaolinite, and other than these minerals sminnesotaite, greenalite are also silicate minerals which are found associated in very minor quantities with iron ore
Phosphorous	Apatite , hydroxylapatite, fluropatite, Chloropatite, Vollophane and bromapatite
Sulphur	Found in the form of iron sulphide minerals such as Pyrite, Marcasite and pyrrohotite

2.3 Flotation

Flotation is a physical and chemical concentration technique that takes use of the variations in surface properties between gangue and valuable minerals (Napier-Munn & Wills, 2006). Flotation is a method used to separate materials, especially in the case of complex or low-quality ores. It is particularly effective when the particles are too small for gravity-based separation or have similar densities. This technique is widely used in areas such as wastewater treatment, removing ash-forming minerals from coal, extracting silicate minerals from iron ores, and separating sulphide minerals from silica gangue and other sulphides (Fuerstenau & Urbina, 2018 ;Adeleke, 2023). It is the most cost-effective and technologically advanced method for improving the quality of iron concentrates (Filippov et al., 2014). Flotation has significant advantages over gravity separation techniques with very fine (< 150µm) particles, while magnetic separation can also be made to work at fine particle sizes, but using magnetic separation alone will reject the non-magnetic portion of the iron ore (Zhang et al., 2021). The most common separation method used for ores in the fine size range (<149µm) is flotation (Viana, 2005). The market requirements for higher grade concentrates of iron with limited contents of silica, alumina, and other impurities to improve the productivity of the iron and steel industry has increased the importance of the flotation process with respect to the conventional pre-concentration of ore by gravity or magnetic separation (Nakhaei & Irannajad, 2018). Flotation has been considered to be the most promising method of obtaining useful, high-grade iron from low-grade, finely disseminated iron ores. It can be used alone or to complement other separation methods, such as gravity and magnetic separation (Zhang et al., 2021). In reverse cationic flotation, over 92% metal loss occurs for ultrafine particles (<10 µm) due to hematite entrainment. Reverse anionic flotation more effectively rejects quartz ultrafines with only ~42% metal loss in the 5–10 µm range. However, cationic flotation performs slightly better for coarse particles (>210 µm), rejecting more quartz (Ma et al., 2011).

2.3.1 Principles of flotation

Flotation is a separation technique that exploits natural and engineered differences in the surface properties of minerals, specifically whether they are hydrophilic (water-attracting) or hydrophobic (water-repelling). In flotation concentration, direct flotation recovers the valuable minerals by allowing them to attach to the froth and rise, leaving the waste material (gangue) behind in the tailings. In contrast, reverse flotation removes the gangue by floating it, while the valuable minerals remain in the tailings (Wills & Partner, 2016).

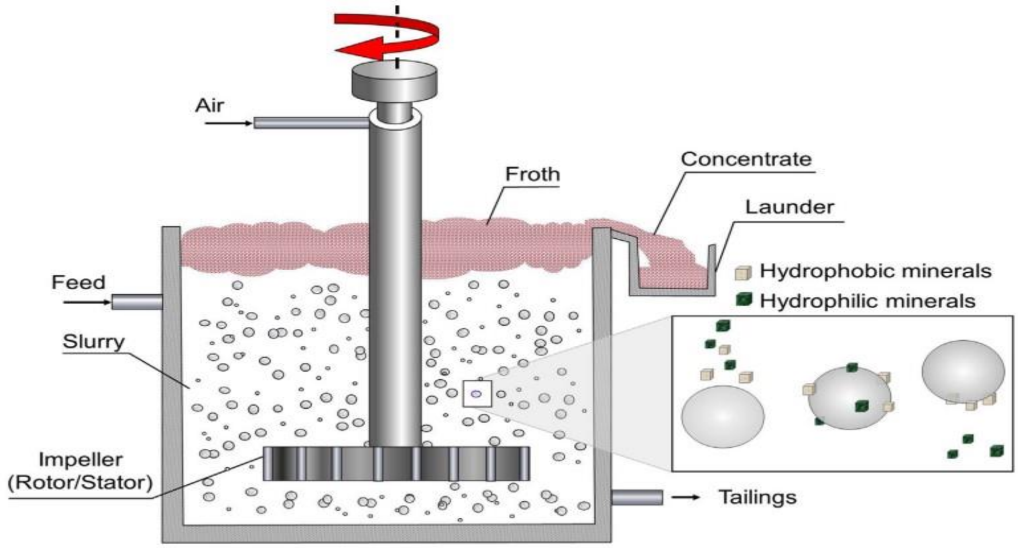

Figure 6: Principle of froth flotation (Wills & Partner, 2016)

2.3.2 Factors affecting flotation

i. Particle size

Coarse particles struggle to stay suspended and detach easily from bubbles due to their weight and high dropout force, while fine particles have low collision rates and poor attachment caused by the hydration layer, resulting in weak flotation performance for both under standard conditions (JXSCMC, 2018). Reverse quartz flotation has been effectively used in the iron ore sector for particle sizes smaller than 150 µm following the de-sliming procedure (Lima et al., 2013).

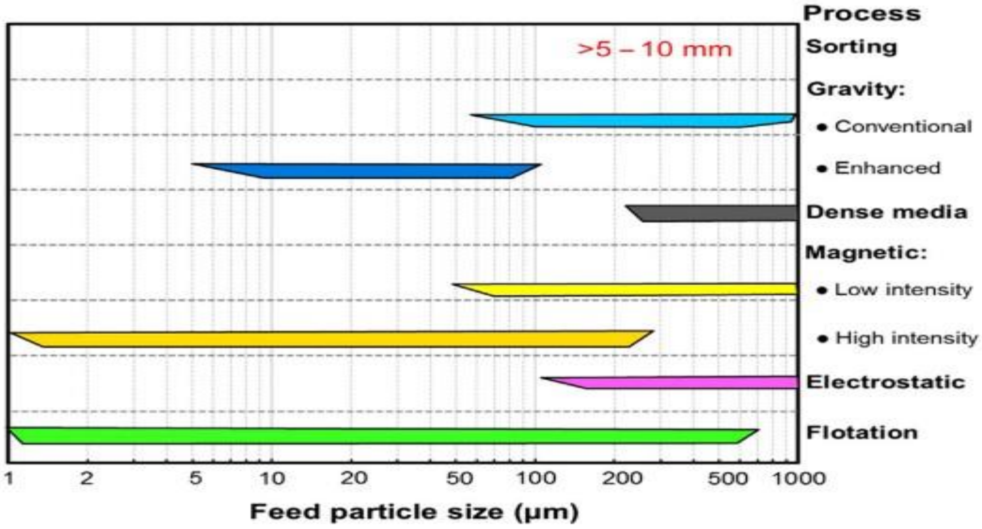

Figure 7: Size ranges of efficient concentration processes (Wills & Partner, 2016)

ii. Pulp pH value

Pulp pH, indicating its acidity or alkalinity, affects mineral surface charge and reagent solubility, influencing particle-bubble attachment. Each mineral has a critical pH range for effective flotation, so controlling pH helps optimize separation (JXSCMC, 2018).

iii. Aeration and Mixing

Air input and stirring influence bubble formation and particle suspension, enhancing flotation efficiency, but excessive aeration and agitation can cause bubble merging, reduced concentrate quality, increased energy use and wear, and particle detachment, so optimal levels must be determined experimentally based on the flotation machine's design (ibid.).

iv. Mineral characteristics

The inherent properties of the minerals being floated, such as their surface chemistry, hydrophobicity, and shape, directly affect flotation performance.

v. Slurry density

Slurry density influences particle-bubble attachment and movement, with optimal levels improving separation efficiency, while low density may cause minerals to wash out prematurely and high density can hinder air dispersion and slurry suspension (ibid.).

vi. Flotation time

Flotation time is the period needed to achieve target recovery and concentrate quality, with longer durations generally increasing recovery but reducing grade, while shorter times raise tailings losses, making experimental determination of optimal time essential for each mineral (ibid.).

vii. Pulp temperature

Flotation is usually conducted at room temperature, but heating the slurry may be necessary for effective separation in certain cases, and when required, it is best to utilize available waste heat or exhaust gases based on local conditions (ibid.).

viii. Pulp concentration

Pulp concentration affects flotation performance, as low levels reduce recovery, overly high levels impair aeration and flow, with lean slurry enhancing concentrate quality and rich slurry increasing capacity and efficiency, making it essential to determine the optimal concentration based on ore type and process requirements (ibid.).

ix. Water quality

Water quality is essential in flotation, with different types like soft, hard, salt, and backwater needing specific conditions, free from excess particles, soluble substances, and microorganisms, while dissolved oxygen levels also influence the process outcomes (ibid).

x. Flotation reagents

Flotation reagents are chemical substances essential to the froth flotation process, enabling the separation of valuable minerals from waste by modifying surface properties of minerals and air bubbles, and without them, modern mineral processing and the mining industry itself would not be possible (Bulatovic, 2007).

a) Collectors

Flotation reagents must be added to the pulp because most minerals are not naturally water-repellent, with collectors being the most essential as they adsorb onto mineral surfaces, render them hydrophobic (or aerophilic), and promote attachment to air bubbles (Napier-Munn & Wills, 2006)

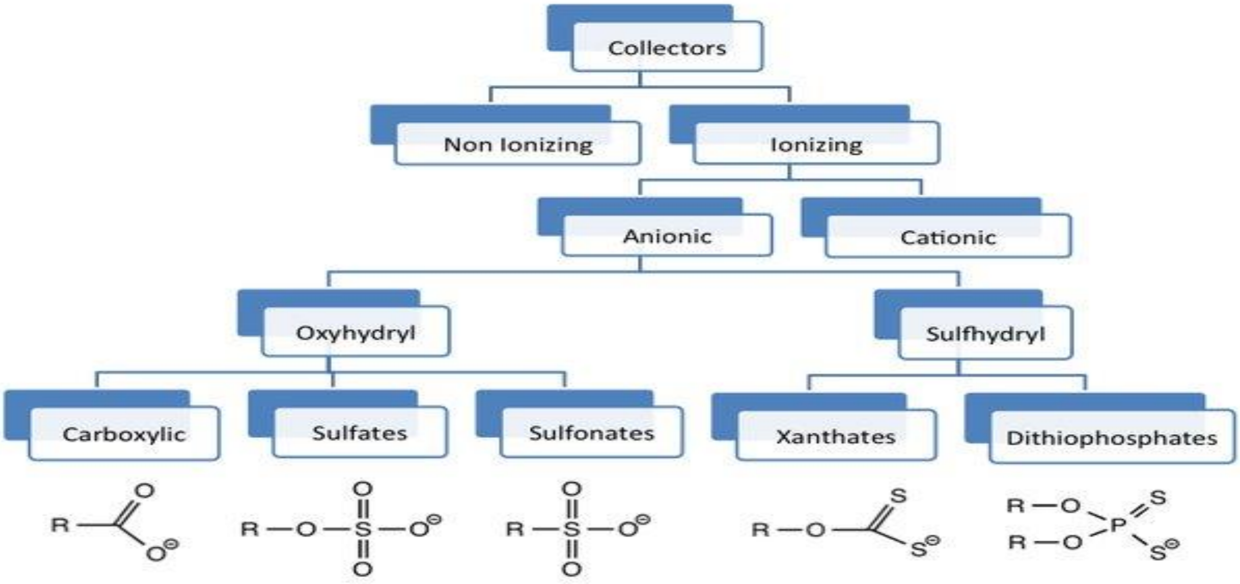

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Figure 8: Classification of collectors

b) Frothers

Frothers are essential for controlling bubble size, froth mobility, and froth stability in flotation (Bulatovic, 2007). Various frothers are widely used in iron ore flotation, including pine oil, aliphatic alcohols, propylene glycols, alkyl ethers of polypropylene glycol, and cresylic acids (Nakhaei & Irannajad, 2018; Mondal et al., 2021; Zhang et al., 2021)

c) Regulators (Modifiers)

During flotation, regulators (activators, depressants, or pH modifiers) adjust mineral adhesion to air bubbles and control pH. Activators change mineral surfaces to hydrophobic, depressants prevent certain minerals from floating, and pH modifiers regulate pulp pH (Napier-Munn & Wills, 2006). Starch, dextrin, guar, and carboxy methyl cellulose are common iron-oxide depressants (Turrer & Peres, 2010; Nakhaei & Irannajad, 2018).

2.3.3 Flotation routes for iron ore

Iron ore flotation methods are classified into direct, reverse anionic, and reverse cationic flotation, where direct flotation collects iron ore in the froth, while reverse flotation removes gangue minerals through froth separation (Zhang et al., 2021). Due to depletion of reserves of high-grade iron ore, low-grade ores which are unsuitable for direct use in iron and steel production must be upgraded through various beneficiation techniques to reduce gangue and enhance Fe content, meeting the growing global demand (Nakhaei & Irannajad, 2018).

i. Direct flotation of iron ore

In this flotation process, iron ore is recovered in the froth using fatty acids as collectors and soda ash to maintain a pH of 9–10, offering a simple method that can work without de-sliming, though its separation efficiency decreases at higher pH due to the similar surface properties of gangue minerals like sulfur, phosphorus, fluorine, and carbonate (Mondal et al., 2021; Zhang et al., 2021). Direct flotation in acidic conditions offers more effective separation but is highly affected by slimes, whereas in alkaline conditions, its efficiency is limited due to similar surface charges between gangue and valuable minerals (Zhang et al., 2021). Anionic collectors are used to float iron due to its net positive surface charge. The flotation of iron ore, which began with the direct flotation of iron oxides using anionic collectors like petroleum sulfonates, fatty acids, and hydroxamates, typically employs fatty acids, resin acids, soaps, alkyl sulfates, and sulfonates to float iron-bearing minerals, particularly hematite (Nakhaei & Irannajad, 2018).

ii. Reverse flotation of iron ore

In recent years, the reverse flotation of iron ores using anionic collectors has gained significant attention because anionic collectors are less expensive than cationic ones and this flotation method is less affected by the presence of slime-sized materials (Behnamfard & Khaphaje, 2019). Reverse flotation is generally classified into two types based on the choice of collector: cationic reverse flotation and anionic reverse flotation (Zhang et al., 2021).

Table 5: A comparison between direct and reverse flotation (Bulayani et al., 2024)

Aspect	**Direct Flotation**	**Reverse Flotation**
Flotation Target	Iron oxides are floated	Gangue minerals are floated with depression of iron oxides
Common Reagents	Anionic reagents (petroleum sulfonates, fatty acids)	Cationic reagents (primary amines, quaternary ammonium salt); sometimes, anionic reagents are used depending on the gangue
pH Modifiers (calcium hydroxide, sodium carbonate, sulfuric acid)	Creates favorable conditions for the flotation of iron oxides	Creates favorable conditions for the flotation of gangue minerals
Depressants (starch, dextrin, sodium silicate)	Prevents flotation of gangue minerals	Prevents flotation of iron oxides
Activators (copper sulfate, lead nitrate)	Not typically used	May be used prior to the addition of collectors to activate gangue minerals
Collectors (dodecanoic acid, sodium dodecyl, amines, fatty acids, xanthates, sulphate)	Aimed at increasing iron oxides hydrophobicity	Aimed at increasing the hydrophobicity of gangue minerals

a) Cationic reverse flotation

Cationic reverse flotation uses a cationic collector to separate and discard negatively charged silica (Zhang et al., 2021). Reverse cationic flotation offers benefits over reverse anionic flotation, such as faster flotation rates and improved efficiency in hard water, but it is more influenced by slime content, whereas the anionic approach is more sensitive to the ionic makeup of the pulp (Nakhaei & Irannajad, 2018). Cationic reverse flotation provides advantages like higher flotation rates, simpler reagent systems, reliable low-temperature operation, and ease of use, but its drawbacks include lower selectivity, excessive foaming, and higher toxicity, leading to product loss, thick and sluggish froth, and environmental pollution (Zhang et al., 2021). Reverse cationic flotation is the predominant flotation method in the iron ore industry, but it faces common challenges, including metal loss during the critical de-sliming stage and the high cost of amine collectors (Ma et al., 2011).

b) Anionic reverse flotation

Anionic reverse flotation uses anionic collectors to eliminate positively charged silica, which typically becomes positively charged only at low pH but can also acquire this charge through the specific adsorption of calcium or magnesium ions provided by activator reagents (Zhang et al., 2021). In the anionic reverse flotation method, the flotation of quartz is achieved by using an activator (usually lime) combined with an anionic collector at elevated pH value (Mondal et al., 2021). Anionic reverse flotation, in comparison to cationic reverse flotation, is less affected by slimes, has lower collector costs, and provides higher selectivity due to calcium's dual role in activating quartz and depressing iron ore, but it requires a large amount of activator and operates at higher temperatures and alkalinities (Zhang et al., 2021). In contrast to reverse cationic flotation, which operates at a pH of 10–10.5, reverse anionic flotation is conducted at a higher pH range of 11–12 (Ma et al., 2011)

CHAPTER THREE

3. MATERIALS AND METHODS

3.1 Materials

3.1.1 Flotation Reagents

The flotation reagents; depressant (corn starch), collector (oleic acid) and frother (pine oil) were purchased from Fine chemical general trading plc. The rest of the flotation reagents (calcium chloride and sodium hydroxide) were obtained from mechanical unit operation laboratory in AAiT, which is one of AAU campus. Flotation reagents used in the flotation experiments were presented in table 6.

Table 6: The flotation reagents were used in the flotation experiments

No	Reagent type	Obtained from	Reagent dosage (in ml)	Remark
1	pH modifier (Sodium hydroxide)	Mechanical unit operation laboratory, AAiT	A drop was added to adjust pH= 11 by using 5% NaOH solution	5 grams of NaOH dissolved in 100 milliliters of water
2	Depressant (Corn starch)	Purchased from Fine chemical general trading plc.	2ml with 2% solution	2 grams of corn starch dissolved in 100 milliliters of water
3	Activator (Calcium chloride)	Mechanical unit operation laboratory, AAiT	2ml with 1% solution	1 gram of $CaCl_2$ dissolved in 100 milliliters of water
4	Collector (Oleic acid)	Purchased from Fine chemical general trading plc.	2ml	Obtained in a liquid form
5	Frother (Pine oil)	Purchased from Fine chemical general trading plc.	2ml	Obtained in a liquid form

In anionic reverse flotation of iron ore, sodium hydroxide adjusts the slurry pH to an alkaline condition, enhancing selectivity. Corn starch acts as a depressant, preventing the flotation of iron ore and allowing impurities to float. Calcium chloride, as an activator, enhances the attachment of silicates to the collector. Oleic acid, the collector, makes silicates hydrophobic for flotation. Pine oil, the frother, reduces surface tension to generate stable foam, ensuring efficient separation and high-quality concentrate. In figure 9 A stands for pH modifier, B stands for depressant, C stands for activator, D stands for collector, and E stands for frother.

Figure 9: The flotation reagents used for this study

3.1.2 Equipment and Instruments

Equipment and instruments used in this study include: GPS, Geological hammer, Jaw crusher, Double cone mixer, Cutting mill, Ball mill, Sieve shaker, Sieves, Riffle splitter, Electronic balance, Flotation cell, pH Meter, Air compressor, Electric oven, Volumetric flask, Measuring cylinder, XRD analyzer, XRF and AAS. The lists of equipment, purpose in this study and their locations were presented in table 7.

Table 7: Equipment and instruments used in this study

No	Name of equipment	Purpose in this study	Available area
1	GPS	Used to indicate the exact location (latitude, longitude, and altitude) of iron ore	GZWIMDD
2	Geological hammer	Used to break, chip, or extract rock and iron ore samples from natural formations	MKWWIMDO
3	Jaw crusher	Used to break down large, hard material (iron ore) into smaller, more manageable pieces.	Mechanical unit operation laboratory, AAiT, AAU
4	Double cone mixer	Used to blend (mix) materials (crushed iron ore samples)	Mechanical unit operation laboratory, AAiT, AAU
5	Cutting mill	Used to cut or grind material (iron ore) into	Mechanical unit operation

		smaller pieces	laboratory, AAiT, AAU
6	Ball mill	Used to grind and blend iron ore sample	Mechanical unit operation laboratory, AAiT, AAU
7	Sieve shaker	Used to mechanically shake a set of sieves containing crushed or grounded iron ore sample	Mechanical unit operation laboratory, AAiT, AAU
8	Sieves (500, 250, 150, 100 and 63µm)	used for the separation and classification of materials according to particle size	Mechanical unit operation laboratory, AAiT, AAU
9	Riffle splitter	used to divide and mix samples to achieve homogeneity	Mechanical unit operation laboratory, AAiT, AAU
10	Electronic balance	Used to weigh the reagents, materials and ore	Mechanical unit operation laboratory, AAiT, AAU
11	Flotation cell	A vessel or tank used for conducting the flotation experiments	Mechanical unit operation laboratory, AAiT, AAU
12	pH Meter	Used to measure and adjust the pH of the flotation pulp	Mechanical unit operation laboratory, AAiT, AAU
13	Air compressor	Used to inject air into flotation cell	Mechanical unit operation laboratory, AAiT, AAU
14	Electric oven	Used to remove moisture (water) from flotation concentrates	Mechanical unit operation laboratory, AAiT, AAU
15	Volumetric flask	Used to mix solid reagents with water and make a solution	Mechanical unit operation laboratory, AAiT, AAU
16	Measuring cylinder	Used to measure the volume of water	Mechanical unit operation laboratory, AAiT, AAU
17	XRD analyzer	Used to determine the mineralogical composition of the iron ore	Chemistry department laboratory, 4kilo campus, AAU
18	AAS	Used for chemical analysis of iron ores	Geochemical laboratory desk, GSE
19	XRF	Used for chemical analysis of concentrates	XRF laboratory, Geology department, AMU

3.2 Methods

3.2.1 Sample Collection

The iron ore sample was collected from the iron ore deposit located in BK, Melo Koza, Gofa zone, South Ethiopia region. The iron ore sample was taken from two different places with a geographic coordinate of E 219775, N 706975 and E 219788, N 706896 in the deposit area.

3.2.2 Sample Preparation

The iron ore samples were subjected a series of size reduction and sizing steps to achieve the desired particle size for flotation. Initially, the samples were crushed using a jaw crusher to reduce the ore into smaller pieces. These pieces were then blended in a double cone mixer for 30 minutes to ensure uniformity in the material. To further reduce particle size, the blended sample was passed through a laboratory cutting mill within a closed-circuit system. Any material coarser than 500μm was returned to the cutting mill for additional size reduction. After the cutting mill, the sample was subjected to further reduction in a ball mill within a closed circuit, ensuring that the particle size was sufficiently fine for flotation. The product from the cutting mill and ball mill stages were sieved using different mesh sizes (500μm, 250μm, 150μm, 100μm and 63μm) to separate the material into various fractions (-500+250, -250+150, -150+100, -100+63 and -63μm). The final product from the crushing, grinding and sieving process was subjected to flotation in a flotation cell. The flow sheet of the sample preparation is showed in figure 10.

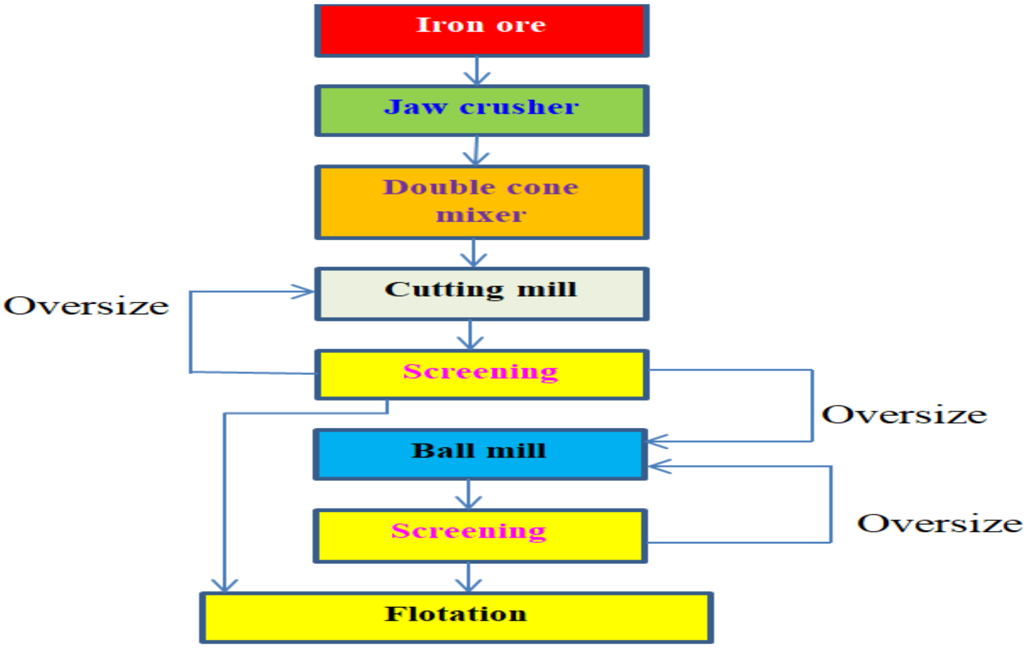

Figure 10: Flow sheet of comminution and sizing

3.2.3 Characterization of iron ore

The mineralogical and chemical analysis of the iron ore were performed using a XRD analyzer and AAS respectively.

3.2.4 Flotation tests

Batch flotation tests were carried out in a flotation cell having 3 liters volumetric capacity. The flotation tests were carried out at -500+250, -250+150, -150+100, -100+63 and -63µm particle size. But the other parameters were constant throughout the flotation tests. The solid-liquid ratio was 10% for all experiments. For both conditioning and flotation, the impeller speed was 1500 rpm. The flotation time was kept constant at 10 minutes. Two liters (2000 mL) of tap water were added to a 3L flotation cell, which was then turned on, followed by the addition of a 200 grams iron ore sample, and the pH of the pulp was adjusted to 11 by adding a 5% NaOH solution (5 grams of NaOH dissolved in 100 mL of water) before introducing other flotation reagents. The pulp (mixture of iron ore and water) was conditioned for several minutes during the pH adjustment in a 3 liters flotation cell, after which depressant (corn starch) was added and conditioned for 3 minutes, activator (calcium chloride) was added and conditioned for 3 minutes, collector (oleic acid) was added and conditioned for 3 minutes, frother (pine oil) was added and conditioned for 2 minutes, and finally, the air valve was opened, and the froth was scraped manually and continuously using a plastic blade. The remaining pulp, containing the iron concentrate, was collected for further analysis. The flotation experiment was shown in figure 11.

Figure 11: Flotation experiments

3.2.5 Dewatering

After flotation, the concentrates were dried in a laboratory oven at 105 °C, weighed, and subjected to chemical analysis to determine the iron content and other impurities.

3.2.6 Characterization of concentrates

The Chemical analysis using XRF were performed on dried and weighed flotation concentrates to determine the concentrations of iron, silica, alumina, and other elements.

3.2.7 Recovery and Grade Calculation

The flotation recovery (the percentage of iron recovered) and grade (the concentration of iron in the concentrate) were calculated by using dry weight and chemical analysis of concentrates. In mineral processing, recovery or recovery rate is the mass fraction of a valuable mineral that is carried over in a beneficiation process from the ore feedstock to the concentrate. The recovery, when dealing with nonmetallic ores, refers to the percentage of the total mineral contained in the ore that is recovered into the concentrate.

$$\text{Recovery (\%)} = \left(\frac{\text{Mass of valuable mineral in concentrate}}{\text{Mass of valuable mineral in feed}}\right) \times 100 \text{ ----------------- } (1)$$

The recovery, in the case of a metallic ore, is the percentage of the total metal contained in the ore that is recovered to the concentrate.

$$\text{Recovery (\%)} = \left(\frac{\text{Mass of elemental metal in concentrate}}{\text{Mass of elemental metal in feed}}\right) \times 100 \text{ ------------------------ } (2)$$

In terms of the usual symbols recovery R is given by:

$$R = \frac{Cc}{Ff} \text{ -- } (3)$$

Where: C is weight of concentrate, F is the weight of feed, c is the assay of metal or mineral in the concentrate, and f is the assay of metal/mineral in the feed.

Grade or assay indicates the content of a marketable commodity in a stream (e.g., feed, concentrate). For metallic ores, it's often shown as % metal; for very low-grade ores like gold, it's expressed in ppm or g/t. Some metals are reported by oxide content (e.g., %WO_3, %U_3O_8). In nonmetallic ores, grade refers to mineral content (e.g., %CaF_2 in fluorite). Diamond ores are graded in carats/100t (1 carat = 0.2 g), while coal grade is based on ash content (Napier-Munn & Wills, 2006).

Grade of Concentrate: The concentration of valuable mineral or metal in the concentrate.

$$\textbf{Grade (\%)} = \left(\frac{\underline{\textbf{Mass of valuable mineral in concentrate}}}{\textbf{Mass of concentrate}}\right) \textbf{ x 100} \text{ ------------------------------ } \textbf{(4)}$$

$$\textbf{Grade (\%)} = \left(\frac{\underline{\textbf{Mass of elemental metal in concentrate}}}{\textbf{Mass of concentrate}}\right) \textbf{ x 100} \text{ ---------------------------- } \textbf{(5)}$$

CHAPTER FOUR

4. RESULTS AND DISCUSSIONS

4.1 Characterization of iron ore

4.1.1 Mineralogical analysis of ore

The XRD analysis was used to determine the mineralogical composition of the sample, with the XRD spectrum shown in Figure 12. The result indicates that the ore mineral in the sample is hematite, while quartz exists as the main gangue mineral in the iron ore sample, which is less than 63 µm particle size ranges. In X-ray diffraction (XRD) analysis, the major peak for hematite typically appears at 2θ = 33.2°, while the major peak for quartz is usually found at 2θ = 26.6°.

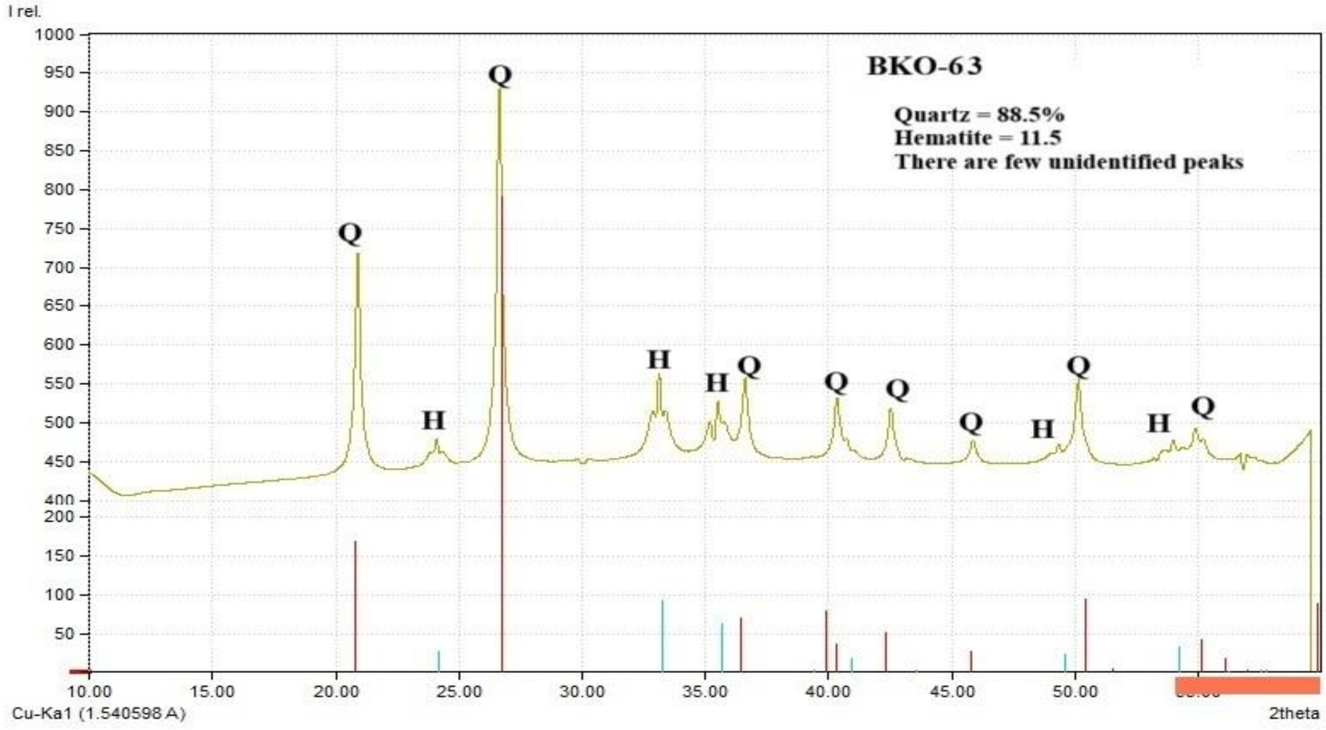

Figure 12: XRD analysis of the ore sample at a particle size of -63 µm

The mineralogical composition of the sample was determined by the XRD analysis. Figure 13 shows the XRD spectrum for -500+250 µm particle size sample. The main peak of the constituting minerals of the sample is specified in this spectrum. It can be seen that the sample contains hematite and goethite as valuable minerals, with quartz as the main gangue mineral.

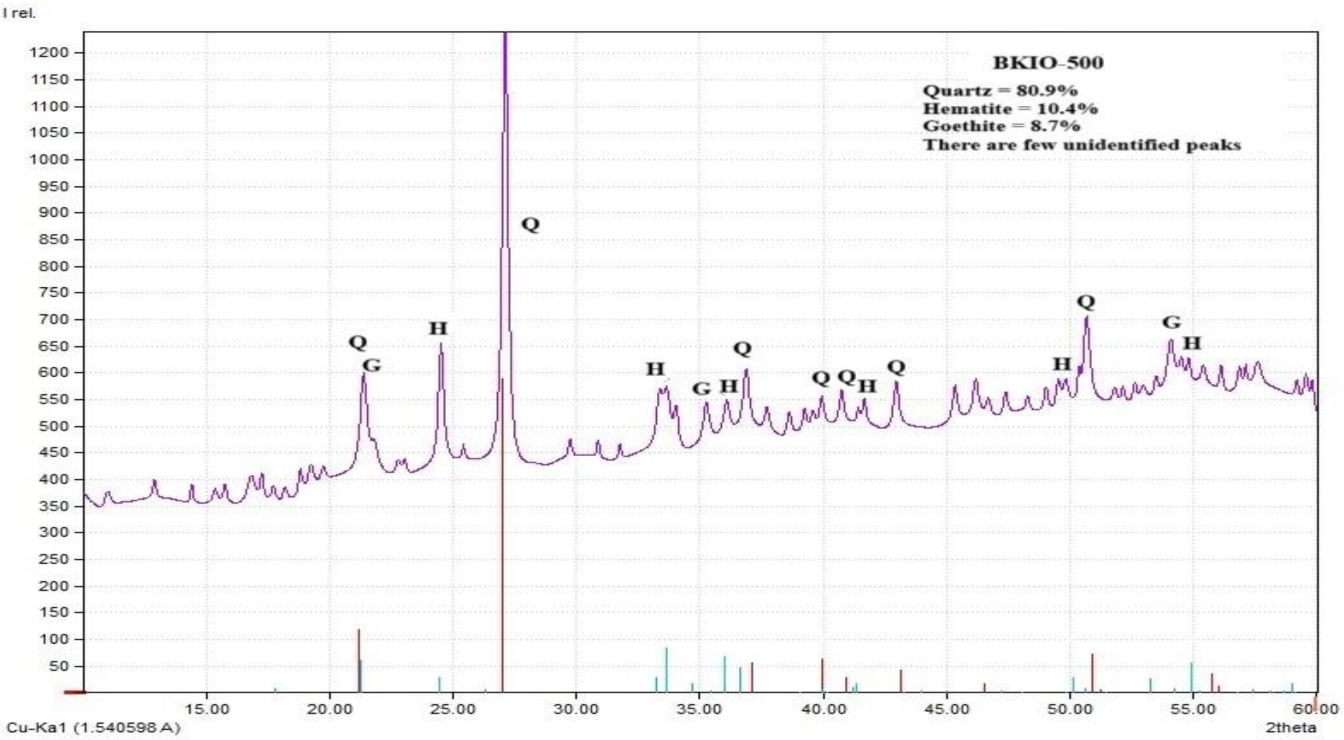

Figure 13: XRD analysis of the ore sample at a particle size of -500+250 µm

4.1.2 Chemical analysis of ore

The chemical analysis of the iron ore sample for this study using AAS is shown in Table 8, where the main impurities are SiO_2 and Al_2O_3, with an average of 29.73% iron oxide (Fe_2O_3) as ore mineral, which is low grade ore.

Table 8: AAS chemical analysis of iron ore

Sample code	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	Na_2O (%)	K_2O (%)	MnO (%)	P_2O_5 (%)	TiO_2 (%)	H_2O (%)	LOI (%)
BKIO(-500+250)	43.16	13.60	31.20	0.40	<0.01	<0.01	0.54	1.10	0.42	0.54	1.73	7.74
BKIO(-250+150)	43.24	12.36	30.98	0.36	0.58	0.36	0.67	1.14	0.44	0.46	1.97	8.52
BKIO(-150+100)	44.14	12.44	30.12	0.30	<0.01	0.10	0.94	1.04	0.42	0.46	1.74	7.78
BKIO(-100+63)	46.60	12.24	30.08	0.34	1.16	<0.01	<0.01	0.88	0.36	0.48	2.10	7.16
BKIO(-63)	51.78	11.46	26.26	<0.01	<0.01	0.72	0.91	<0.01	0.32	0.46	2.05	7.30

Chemical analysis results of -500+250, -250+150, -150+100, -100+63 and -63 µm particle size raw ore was given in table 8. The ore sample contains 43.16% SiO_2, 13.60% Al_2O_3 and 31.20% Fe_2O_3 for -500+250µm particle range, 43.24% SiO_2, 12.36% Al_2O_3 and 30.98% Fe_2O_3 for -250+150µm particle range, 44.14% SiO_2, 12.44% Al_2O_3 and 30.12% Fe_2O_3 for -150+100µm particle range, 46.60% SiO_2, 12.24% Al_2O_3 and 30.08%

Fe_2O_3 for -100+63µm particle range and 51.78% SiO_2, 11.46% Al_2O_3 and 26.26% Fe_2O_3 for -63µm particle range as main components. AAS results indicated that iron oxide (Fe_2O_3), which is low grade as valuable component and silica (SiO_2) and alumina (Al_2O_3) as main gangue minerals. The steel industry is increasingly shifting toward direct reduction paired with electric furnace production, which requires iron ore with less than 2% SiO_2, 0.1% sulfur, 0.01% phosphorus, and 2% aluminum (Nakhaei & Irannajad, 2018). The silica content (43.16% - 51.78%) and alumina content (11.46% - 13.60%) is high compared to the generally accepted level of < 2% SiO_2 and < 2% aluminum respectively. The levels of other impurities are not very high. The iron (Fe) grade must be upgraded because it does not fulfill the minimum blast furnace requirements.

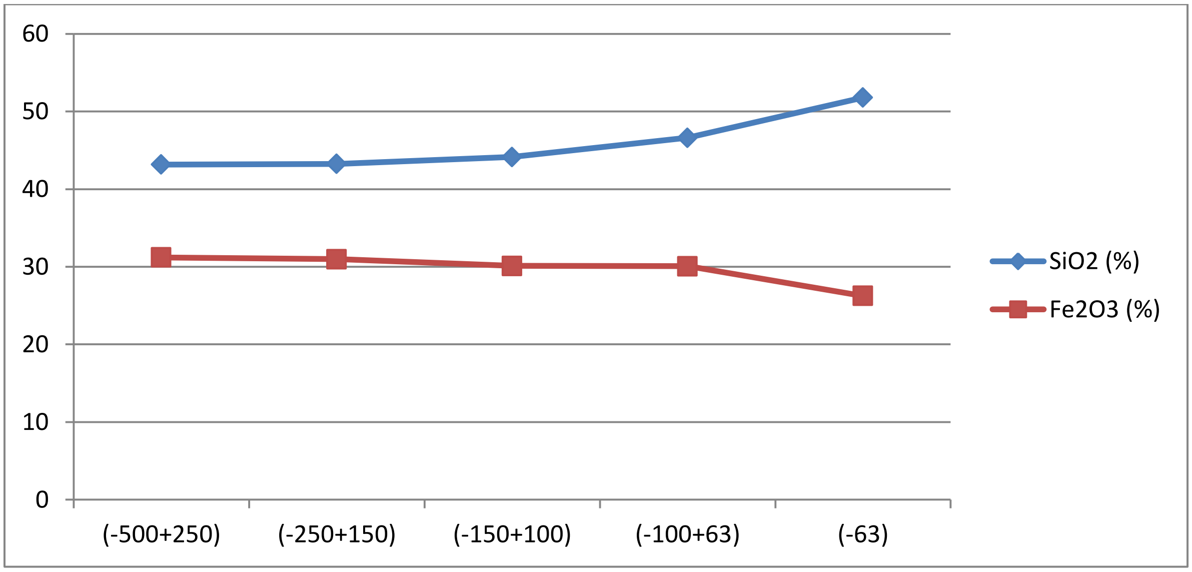

Figure 14: Relationship of silica, iron oxide & particle size in the ore

The relationship between silica, iron oxide and particle size of ore is presented in figure 14. The iron oxide (Fe_2O_3) content decreases progressively with decreasing ore particle size, dropping from 31.20% in the coarsest fraction to 26.26% in the finest. This trend indicates that iron is predominantly concentrated in the coarser particles, while the finer fractions are enriched in gangue minerals, primarily silica. This distribution suggests a degree of mineral liberation, where iron-bearing minerals are more effectively preserved in the coarse particles and separated from the gangue during crushing and grinding. Silica (SiO_2) content increases with finer particle size, confirming that the finer fractions are more siliceous, likely due to the higher presence of quartz or other silicate gangue minerals.

Table 9: Geochemical analysis of iron ore samples (Agegnehu et al., 2023)

Sample code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2
GZ-24	19.34	18.8	42.32	<0.01	0.08	0.56	2.64	5.44	<0.01	<0.01
GZ-24	20.28	11.2	46.4	<0.01	0.08	<0.01	2.4	7.52	0.36	<0.01
GZ-05	55.6	6.38	24.8	<0.01	0.4	5.6	2.48	0.32	<0.01	<0.01
GZ-01	55.78	15.96	16.64	<0.01	0.52	0.84	1.4	1.4	<0.01	0.1
GZ-02	52.38	6.08	22.44	<0.01	0.24	<0.01	1.56	1.4	<0.01	0.07
GZ-30	72.44	1.96	16.4	1.44	0.48	1.04	3.6	<0.01	<0.01	0.04
GZ-16	63.8	8.94	16.32	<0.01	0.08	<0.01	3.04	<0.01	0.04	0.03
GZ-32	54.48	20.45	16.89	1.37	2.03	1.23	1.09	<0.01	0.58	1.53
GZ-26	9.46	4.16	65.04	0.56	0.32	0.32	2.4	0.48	0.22	0.06
GZ-25	28.36	5.22	53.01	<0.01	<0.01	<0.01	0.41	<0.01	0.07	<0.01

The chemical composition of iron ore from the BK area is summarized in Table 9. The samples show concentration of Fe_2O_3 (16.32 – 65.04 wt%, average 32.02 wt %), SiO_2 (9.46 - 72.44 wt %, average 43.2 wt %), and Al_2O_3 (1.96 - 20.45 wt %, average 9.92 wt %) as major constituents, but CaO (<0.01- 1.44 wt %), MgO (<0.01 – 2.03 wt %), Na_2O (<0.01 - 5.6 wt %), K_2O (0.41 – 3.6 wt %), MnO (<0.01 – 7.52 wt %), P_2O_5 (<0.01 – 0.58 wt %) and TiO_2 (<0.01 – 1.53 wt %) as minor constituents (Agegnehu et al., 2023). In figure 15 there is minimal variation between the average contents of iron oxide, silica, and alumina reported by Agegnehu et al. (2023) and the AAS results obtained in this study.

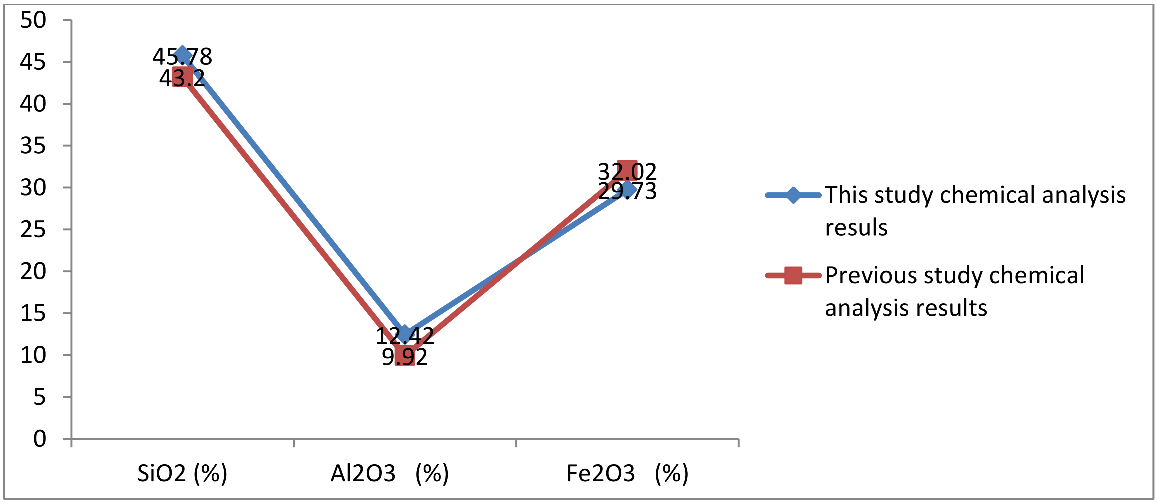

Figure 15: Comparison of this study with previous chemical analysis results

4.2 Flotation experiments

Flotation experiments were performed in order to get recovery values and a reasonable grade. Fifteen (3 experiments for each particle size range) flotation tests by varying particle size range and keeping rest of all parameters as constant were performed. The flotation tests results were presented in table 10.

Table 10: Flotation test products for each particle size ranges

Experimental code	**Run**	**Particle size ranges (μm)**	**Weight of iron ore (in grams)**	**Weight of Concentrates (in grams) after dried**	**Remark**
BKIC-001	1	-250+150	200	176	
BKIC-002	2	-250+150	200	158.2	
BKIC-003	3	-150+100	200	140.5	
BKIC-004	4	-500+250	200	186	
BKIC-005	5	-150+100	200	160.3	
BKIC-006	6	-100+63	200	148	
BKIC-007	7	-63	200	128.3	
BKIC-008	8	-100+63	200	148	
BKIC-009	9	-500+250	200	189	
BKIC-010	10	-63	200	135.3	
BKIC-011	11	-250+150	200	169.3	
BKIC-012	12	-500+250	200	191.1	
BKIC-013	13	-100+63	200	154.1	
BKIC-014	14	-150+100	200	156.7	
BKIC-015	15	-63	200	119.2	

4.3 Chemical analysis of Concentrates

From the above flotation concentrates in table only five (5) were selected for the chemical analyses by using XRF. The results of chemical analyses on concentrates were shown in Table 11. The concentrates contains 11.63% SiO_2, 1.85% Al_2O_3 and 32.26% Fe_2O_3 for -500+250µm particle size, 13.25% SiO_2, 2.29% Al_2O_3 and 34.22% Fe_2O_3 for -250+150µm particle size, 15.16% SiO_2, 2.41% Al_2O_3 and 33.50% Fe_2O_3 for -150+100µm particle size, 17.60% SiO_2, 2.23% Al_2O_3 and 32.80% Fe_2O_3 for -100+63µm particle size and 20.52% SiO_2, 2.46% Al_2O_3 and 32.95% Fe_2O_3 for -63µm particle size. Both the main gangue minerals (silica and alumina) are removed during flotation and the percentages are decreased in concentrates. The iron contents in concentrates are greater than iron content in ore. However, the percentage of main gangue minerals were above the acceptable limit and the iron grade was low after flotation. Therefore further separation is needed to satisfy the accepted value.

Table 11: XRF Analyses of flotation concentrates

Sample code	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	K_2O (%)	Cr_2O_3 (%)	TiO_2 (%)	MnO (%)	P_2O_5 (%)	Ta_2O_5 (%)
BKIC-005	15.16	2.41	33.50	0.08	0.00	0.93	0.05	0.39	1.00	0.81	0.00
BKIC-012	11.63	1.85	32.26	0.10	0.00	0.71	0.05	0.47	0.87	0.00	0.00
BKIC-001	13.25	2.29	34.22	0.07	0.00	0.87	0.06	0.38	1.24	0.77	0.00
BKIC-010	20.52	2.46	32.95	0.00	0.00	0.79	0.06	0.42	0.86	1.15	0.00
BKIC-013	17.60	2.23	32.80	0.07	0.00	0.83	0.06	0.37	1.19	0.00	0.00

4.4 Optimization of particle size range

Particle size is essential factor to improve the grade and recovery of minerals in the flotation. To obtain the optimum particle size range, the flotation experiments were done in the alkaline pH value (pH =11) using sodium hydroxide as pH modifier, corn starch as depressant, calcium chloride as activator, oleic acid as collector and pine oil as frother. The iron grade and recovery were calculated for all particle size range to determine the optimum particle size range.

$$\textbf{Mass of } Fe_2O_3 \textbf{ in Feed} = \textbf{Mass of Feed x} \left(\frac{\% \; Fe_2O_3 \textbf{ in Feed}}{100} \right) \text{ ------------------------------ } (6)$$

$$\textbf{Mass of } Fe_2O_3 \textbf{ in Concentrate} = \textbf{Mass of Concentrate x} \left(\frac{\% \; Fe_2O_3 \textbf{ in Concentrate}}{100} \right) \text{ ----------------- } (7)$$

$$\textbf{Mass of Fe} = \textbf{Mass of } Fe_2O_3 \textbf{ x } 0.699 \text{ -- } (8)$$

Based on the above equations mass of iron oxide (Fe_2O_3) & iron metal (Fe) in feed, mass of iron oxide (Fe_2O_3) & iron metal (Fe) in concentrate and recovery & iron grade were presented in table 12, table 13 and table 14 respectively.

Table 12: Mass of iron oxide (Fe_2O_3) and iron metal (Fe) in feed

Sample Code	**Particle size range**	**Mass of feed (in grams)**	**Percent of Fe_2O_3 in feed (%)**	**Mass of Fe_2O_3 in feed (in grams)**	**Mass of Fe in feed (in grams)**
BKIO(-500+250)	-500+250	200	31.20	62.40	43.62
BKIO(-250+150)	-250+150	200	30.98	61.96	43.31
BKIO(-150+100)	-150+100	200	30.12	60.24	42.11
BKIO(-100+63)	-100+63	200	30.08	60.16	42.05
BKIO(-63)	-63	200	26.26	52.52	36.71

Table 13: Mass of iron oxide (Fe_2O_3) and iron metal (Fe) in concentrates

Sample Code	**Particle size range**	**Mass of concentrate (in grams)**	**Percent of Fe_2O_3 in concentrate (%)**	**Mass of Fe_2O_3 in concentrate (in grams)**	**Mass of iron (Fe) in concentrate (in grams)**
BKIC-012	-500+250	191.1	32.26	61.65	43.09
BKIC-001	-250+150	176	34.22	60.23	42.10
BKIC-005	-150+100	160.3	33.50	53.70	37.54
BKIC-013	-100+63	154.1	32.80	50.54	35.33
BKIC-010	-63	135.3	32.95	44.58	31.16

Table 14: Iron grade and recovery

Mass of feed (in grams)	**Mass of iron (Fe) in feed (in grams)**	**Mass of concentrate (in grams)**	**Mass of iron (Fe) in concentrate (in grams)**	**Grade (%)**	**Recovery (%)**
200	43.62	191.1	43.09	22.55	98.78
200	43.31	176	42.10	23.92	97.21
200	42.11	160.3	37.54	23.42	89.15
200	42.05	154.1	35.33	22.93	84.02
200	36.71	135.3	31.16	23.03	84.88

The iron mass recoveries in the concentrate were as follows: 98.78% at the -500+250 µm range, 97.21% at the -250+150 µm range, 89.15% at the -150+100 µm range, 84.02% at the -100+63 µm range, and 84.88% at the -63 µm range. The recovery decreases as the particle size decreases. Figure 14 demonstrates the effect of particle size range on the iron ore flotation. The optimal particle size range was determined to be between -250 and +150 µm, based on the grade and recovery. The average iron (Fe) grade of 20.78% in the ore was increased to 23.17% in the concentrate; while the average iron oxide (Fe_2O_3) grade of 29.73% in the ore was upgraded to 33.15% in the concentrate. To calculate the improvement in grade, the percentage increase from the initial ore grade to the final concentrate grade was calculated by using the following formula.

$$\textbf{Percentage increase} = \left(\frac{\textbf{Final Grade} - \textbf{Initial Grade}}{\textbf{Initial Grade}} \right) \textbf{x 100} \text{ -------------------------- } \textbf{9}$$

For Iron (Fe) Grade:

$$\text{Percentage increase} = \left(\frac{23.17 - 20.78}{20.78} \right) \text{x } 100 = 11.5\%$$

Therefore, the average iron content in the concentrate has increased by approximately 11.5 % compared to the original ore.

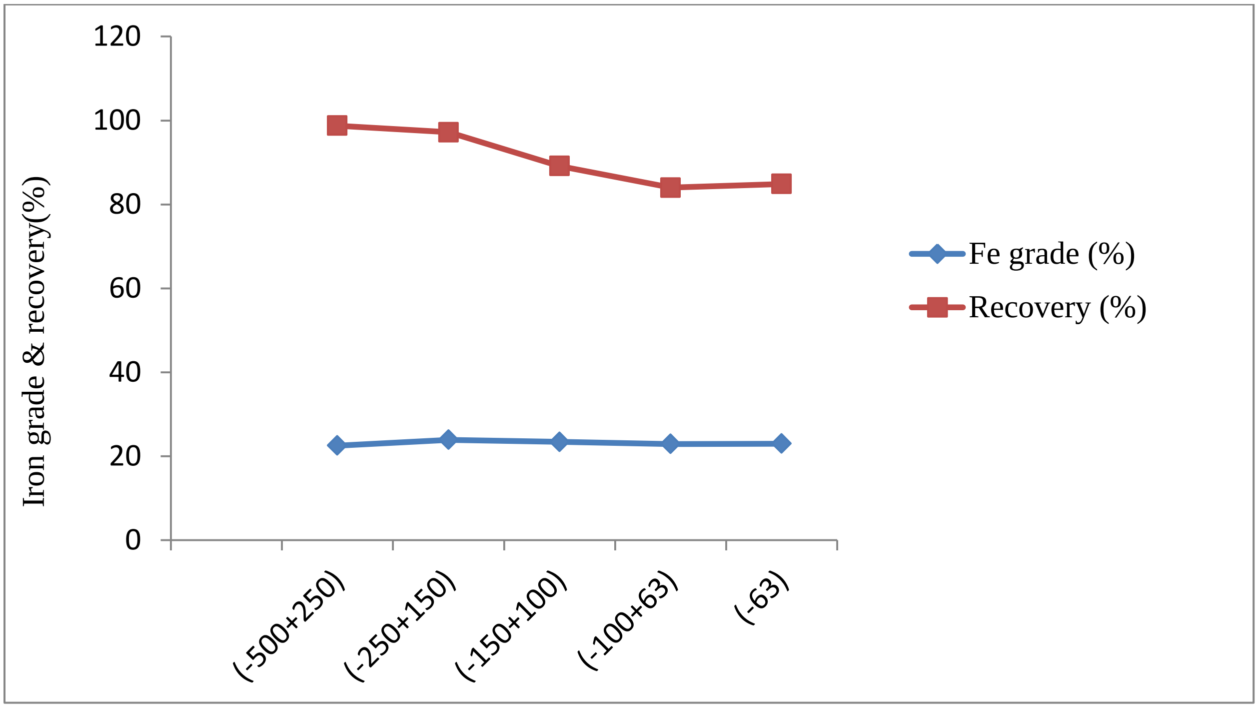

Figure 16: Effect of particle size range on Fe grade and recovery

CHAPTER FIVE

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Mineralogical analysis via XRD revealed that the BK iron ore sample predominantly contained hematite and goethite as the valuable minerals, with quartz as the main gangue mineral. AAS chemical analysis showed that the ore contained Fe_2O_3 (26.26 – 31.20 wt%, average 29.73 wt %) as the primary iron-bearing mineral, along with SiO_2 (43.16 – 51.78 wt%, average 45.78 wt %) and Al_2O_3 (11.46 – 13.60 wt%, average 12.42 wt %) as the major impurities. The low-grade BK iron ore was upgraded through anionic reverse flotation using the following conditions: 2 ml of 2% corn starch solution, 2 ml of oleic acid, 2 ml of 1% calcium chloride solution, and 2 ml of pine oil, pH of 11 and particle size of -250+150 microns, which is optimal particle size range because of highest concentrate grade due to mineral liberation and less affected by slimes. Under these conditions, a concentrate with 23.92% Fe grade and 97.21% iron recovery was achieved. The average iron (Fe) grade of 20.78% in the ore was increased to 23.17% in the concentrate, while the average iron oxide (Fe_2O_3) grade of 29.73% in the ore was upgraded to 33.15% in the concentrate. The percentage increase in iron content from the initial ore grade to the final concentrate grade was calculated to be 11.5%. This means that the average iron concentration in the concentrate has increased by approximately 11.5% compared to the original ore. However, further separation is necessary to enhance the iron content and eliminate impurities to meet the minimum iron grade specifications needed for blast furnaces and steel manufacturers. This study not only provides valuable insights into the beneficiation of BK iron ore but also serves as a foundation for future research and development in this area.

5.2 Recommendations

The study's conclusions lead to the following recommendations for further investigation. It is highly recommended that sophisticated equipment be utilized for comprehensive mineralogical and chemical investigations of the BK iron ore and concentrates, providing a deeper understanding of their composition and potential beneficiation techniques. It is strongly recommended that the number of samples collected from iron ore and concentrates for laboratory analyses increased for future work. It is also recommended that future studies continue to perform particle size analysis using multiple complementary techniques to ensure accuracy and reliability of the results. Employing a combination of methods such as laser diffraction, dynamic light scattering, and sieve analysis can provide a more comprehensive understanding of particle size distribution and help validate findings across different measurement scales. Additionally, the performance of anionic reverse flotation should be thoroughly examined by adjusting key parameters such as the solid-liquid ratio, reagent dosages, and flotation time. To enhance the quality of valuable minerals while minimizing waste, a series of steps, including cleaning and scavenging, should be incorporated into the process. Furthermore, a combination of gravity separation followed by flotation is suggested to effectively reduce impurities to acceptable levels in the concentrate, thereby enriching the desired iron minerals for efficient downstream extraction processes. To maximize beneficiation methods and support the growth of the mineral processing sector, further study and development in this area are crucial.

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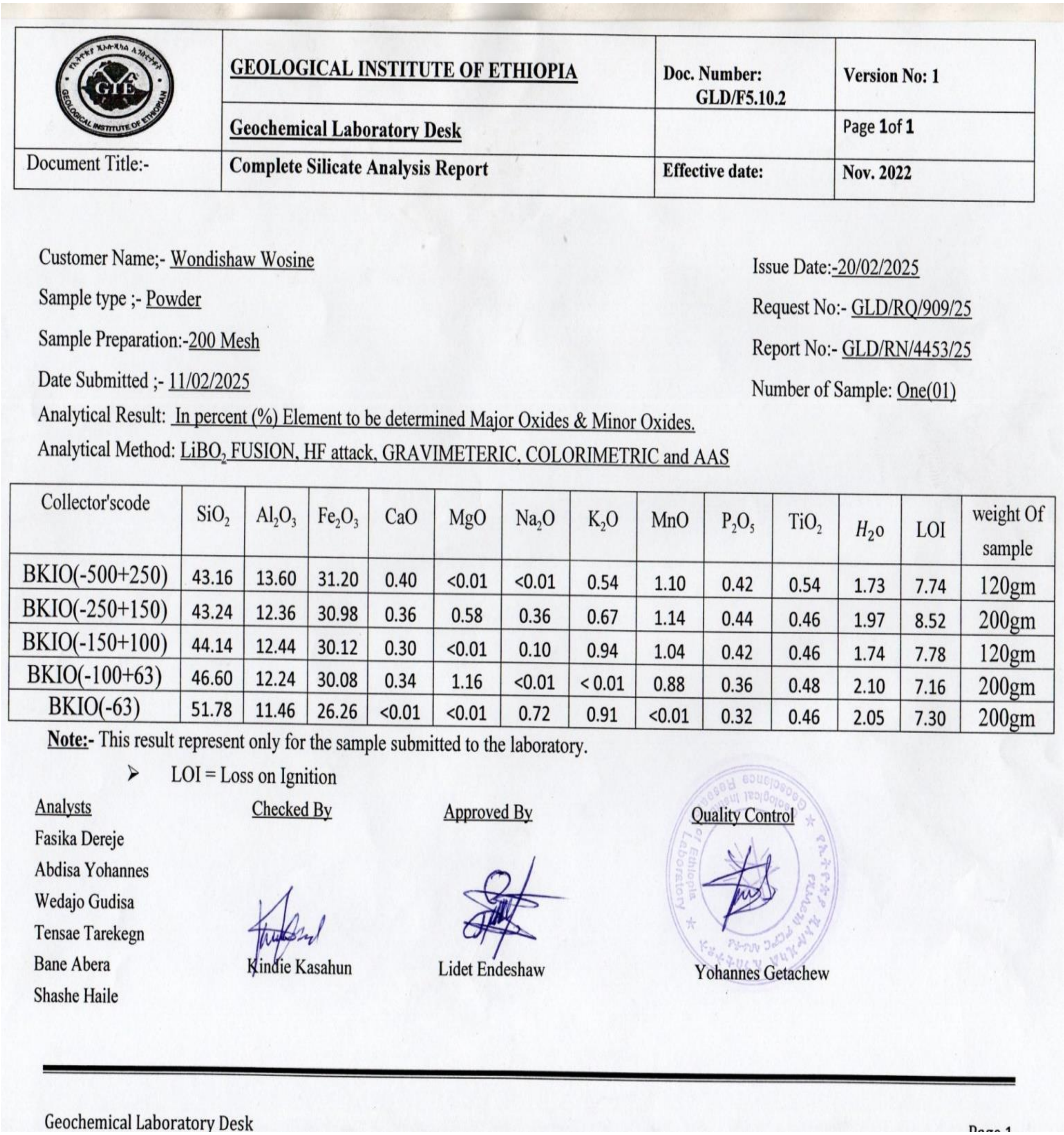
APPENDICES

Appendix A: Chemical analysis report for iron ores and concentrates



	GEOLOGICAL INSTITUTE OF ETHIOPIA	**Doc. Number:** GLD/F5.10.2	**Version No: 1**
	Geochemical Laboratory Desk		Page **1**of **1**
Document Title:-	**Complete Silicate Analysis Report**	**Effective date:**	**Nov. 2022**

Customer Name;- Wondishaw Wosine

Sample type ;- Powder

Sample Preparation:-200 Mesh

Date Submitted ;- 11/02/2025

Issue Date:-20/02/2025

Request No:- GLD/RQ/909/25

Report No:- GLD/RN/4453/25

Number of Sample: One(01)

Analytical Result: In percent (%) Element to be determined Major Oxides & Minor Oxides.

Analytical Method: $LiBO_2$ FUSION, HF attack, GRAVIMETERIC, COLORIMETRIC and AAS

Collector'scode	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	MnO	P_2O_5	TiO_2	H_2o	LOI	weight Of sample
BKIO(-500+250)	43.16	13.60	31.20	0.40	<0.01	<0.01	0.54	1.10	0.42	0.54	1.73	7.74	120gm
BKIO(-250+150)	43.24	12.36	30.98	0.36	0.58	0.36	0.67	1.14	0.44	0.46	1.97	8.52	200gm
BKIO(-150+100)	44.14	12.44	30.12	0.30	<0.01	0.10	0.94	1.04	0.42	0.46	1.74	7.78	120gm
BKIO(-100+63)	46.60	12.24	30.08	0.34	1.16	<0.01	< 0.01	0.88	0.36	0.48	2.10	7.16	200gm
BKIO(-63)	51.78	11.46	26.26	<0.01	<0.01	0.72	0.91	<0.01	0.32	0.46	2.05	7.30	200gm

Note:- This result represent only for the sample submitted to the laboratory.

- LOI = Loss on Ignition

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ቁጥር / Ref.No/Geo:
ቀን / date: March 12, 2025

The samples were analyzed in Arba Minch University College of Natural Sciences Department of Geology at XRF laboratory.

Issue Date: February 27, 2025
Sample preparation :200µm
Number of samples: 5

Customer/ Company Name: Wondishaw Wosine Wolka
Sample type: Rock powder
Date submitted: March 12, 2025
Analytical result in percentage (%) -major and trace oxides
Analytical method: XRF

Sample code	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Cr_2O_3	TiO_2	MnO	P_2O_5	Ta_2O_5
BKIC_005	15.16	2.41	33.50	0.08	0.00	0.93	0.05	0.39	1.00	0.81	0.00
BKIC_012	11.63	1.85	32.26	0.10	0.00	0.71	0.05	0.47	0.87	0.00	0.00
BKIC_001	13.25	2.29	34.22	0.07	0.00	0.87	0.06	0.38	1.24	0.77	0.00
BKIC_010	20.52	2.46	32.95	0.00	0.00	0.79	0.06	0.42	0.86	1.15	0.00
BKIC_013	17.60	2.23	32.80	0.07	0.00	0.83	0.06	0.37	1.19	0.00	0.00

Note: - This result represents only for five samples submitted to the laboratory

Analyst
Teklehaimanot Teklearegay (MSc)

Approved by
Leulalem Shano (PhD)

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When replying please indicate our reference No.

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Appendix B: Some photos of laboratory equipment's used


A. Jaw crusher


B. Cutting mill


C. Ball mill

D. Double cone mixer


E. Sieve shaker

F. Set of sieves

G. Air compressor

H. pH meter

I. Flotation cell

Appendix C: photo of samples prepared for flotation