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# ADDIS ABABA UNIVERSITY

## ADDIS ABABA INSTITUTE OF TECHNOLOGY

## CENTER FOR ETHIO-MINES DEVELOPMENT (CEMD)

# INVESTIGATION OF REVERSE FLOTATION RESPONSE FOR THE BENEFICIATION OF IRON ORE AROUND DAWA-MOYALE, SOMALI REGION, ETHIOPIA

A research project submitted to Addis Ababa university, Addis Ababa Institute of Technology, Center for Ethio-Mines development, in partial Fulfilment of the requirements for the Degree of Master of Engineering in Mineral Engineering.

**By**
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May, 2025
Addis Ababa, Ethiopia

# DECLARATION

I declare that this research project titled "Investigation of reverse flotation response for the beneficiation of iron ore around Dawa-Moyale, Somali region, Ethiopia" is my original work and has not been presented for a degree in any other university. All sources of materials used have been duly acknowledged.

Name: Abdifatah Maktel Abdullahi

Signature: ______________

Date: May 30, 2025

**ADDIS ABABA UNIVERSITY**
**ADDIS ABABA INSTITUTE OF TECHNOLOGY**
**CENTER FOR ETHIO-MINES DEVELOPMENT**

## Approval Sheet

This is to certify that the research project prepared by **Abdifatah Maktel Abdullahi**, entitled "Investigation of reverse flotation response for the beneficiation of iron ore around Dawa-Moyale, Somali region, Ethiopia" and submitted to Addis Ababa University, AAiT, in partial fulfillment of the requirement for the degree of Master of Engineering in Mineral Engineering, complies with the regulations of the university and meets the accepted standards concerning its originality and quality.

Signed by the Examining Committee: Signature Date

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# DEDICATION

To my beloved family, whose unwavering support made this journey possible.

# ABSTRACT

Iron (Fe) is the fourth most abundant element in the Earth's crust and constitutes approximately 6% of its composition, which is fundamental to the industrialization and civilization of any nation, including that of the Federal Democratic Republic of Ethiopia. The Dawa-Moyale area in the southern metamorphic terrain of Ethiopia has been recognized as a region rich in iron ore deposits. Therefore, this study aims to investigate the beneficiation potential of Dawa-Moyale iron ore in the Somali region of Ethiopia using the reverse flotation method. The study employed a two-level full factorial experimental design using Stat-Ease 360 software (Trial version) to model the influence of various experimental factors on flotation performance. The flotation experiments focused on feed particle size, pH value, and collector dosage as the independent variables, while iron grade and recovery were designed as process responses. The results of the experimental tests indicated that the models for iron grade and recovery were statistically significant, where the predicted values aligned well with the experimental results, with R-squared ($R^2$) values of 0.972 and 0.9962 for iron grade and recovery, respectively. The findings revealed that collector dosage (oleic acid), pH value, and the interaction between particle size and collector dosage significantly influence the Fe grade, whereas feed particle size is the sole experimental factor with a statistically significant effect on iron recovery. The maximum iron grade and recovery of 46% and 94.7%, respectively, were achieved under the flotation conditions determined as follows: a particle size of -150+63µm, a collector dosage of 125g/t, and a pH of 9.5. The results of this study provide valuable insights into the beneficiation potential of Dawa-Moyale iron ore and contribute to the development of the iron ore industry of Ethiopia.

**Keywords:** Collector Dosage, Dawa-Moyale, Iron Ore, Particle Size, Reverse Flotation

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# List of Abbreviations

| | |
|---|---|
| **AAiT** | Addis Ababa institute of technology |
| **AAS** | Atomic Absorption Spectroscopy |
| **BIF** | Bedded Iron Formations |
| **CMC** | Carboxymethyl Cellulose |
| **EGI** | Ethiopian Geological Institute |
| **g/t** | Gram per ton |
| **GSE** | Geological Survey of Ethiopia |
| **LOI** | Loss on Ignition |
| **MLA** | Mineral Liberation Analyzer |
| **pH** | Hydrogen Ion Concentration |
| **QEMSCAN** | Quantitative Evaluation of Minerals by Scanning Electron Microscopy |
| **rpm** | Revolution per minute |
| **USGS** | United States Geological Survey |
| **XRD** | X-Ray Diffraction |

# 1. Introduction

## 1.1 Background of the Study

The development of any nation, including that of the federal democratic republic of Ethiopia, lies in its industrialization, which in turn depends on the extraction of its mineral resources. Iron ranks as the most prevalent element in the Earth's crust, comprising approximately 6% of its composition (1–3). It is the primary raw material for producing steel, where its importance extends beyond just its use in steel production; it plays a crucial role in the global economy, industrial development, and modern civilization (3). A recent report published by the USGS in 2024 estimated that the global production of crude iron ore from mines is about 2,500 billion metric tonnes. The top three iron ore producers, in decreasing order, are Australia, Brazil, and China (4). Global demand for iron in steel production has increased significantly due to urbanization, infrastructure development, and industrialization (5).

The primary global iron deposits are associated with the banded iron formations from the Precambrian age, which are of sedimentary origin (6). These iron deposits typically occur in various forms: oxides, such as hematite ($Fe_2O_3$; 72.4% Fe), magnetite ($Fe_3O_4$; 70% Fe); hydroxides like goethite (FeO (OH); 62.9% Fe), limonite ($Fe_2O_3$.nH2O); carbonates such as siderite ($FeCO_3$; 48.2% Fe); and sulfides including pyrite ($FeS_2$; 46.6% Fe) (3,7,8). Among these, hematite, magnetite, and goethite are the most important due to their larger, economically minable quantities (9). Gravity, magnetic, and flotation methods are commonly employed to concentrate intermediate and low-grade iron-bearing minerals from the unwanted materials such as silica and alumina.

Flotation is a physicochemical concentration method that takes advantage of variations in the surface properties of valuable minerals and gangues (8). The efficiency of the flotation process is impacted by several critical chemical and physical parameters, including the mineral composition, particle size, degree of liberation, type and concentration of collectors, pH level, airflow rates, pulp density, and water wash rates (10). However, minerals are separated through flotation when minerals targeted for separation exhibit different affinities for air and water (11). According to Kawatra et. Al (2011) (12), flotation can be employed in various industries, including 1) The separating sulfide minerals from silica gangue (as well as other sulfide minerals), 2) The separating coal from ash-forming minerals, 3) The removal of silicate minerals from iron, or 4) The treatment of Wastewater. This method is especially effective for treating finely ground iron ores that cannot be effectively amenable to conventional gravity separation techniques (12). Although Magnetic separation can also effectively manage fine particle sizes, reliance solely on this method may result in the loss of non-magnetic components of iron ore (13).

Direct and reverse flotation routes have been practiced since the introduction of the flotation method in the United States in 1931; in direct flotation, the objective is to separate the iron ore by floating it to the froth product, whereas, in reverse flotation, unwanted materials are floated to the froth product and subsequently discarded (4, 9, 11). The reverse flotation method is typically categorized into two distinct types, based on the nature of the collector types employed: cationic reverse flotation and anionic reverse flotation. Reverse anionic flotation was first established in China, utilizing anionic collectors to float silica gangues activated with lime, followed by flotation with fatty acids as a collector at a high pH (11-12) while employing starch to depress the iron-bearing minerals (13). Hanna Mining and American Cyanamid also developed direct flotation and reverse anionic flotation during the 1930s and 1940s. By the 1950s, these methods were applied in Minnesota and Michigan concentrators (14). Around the same time, reverse cationic flotation was being developed by the U.S. Bureau of Mines, which ultimately became the preferred method for iron ore flotation in the United States and other Western countries (14,15).

Reverse cationic flotation employs amine collectors and starch depressants to selectively float silica-based unwanted materials, such as quartz, under alkaline conditions. However, a depressant is often necessary for hematite ores to prevent the flotation of hematite, ensuring a selective separation (13,16). The benefits of reverse anionic flotation compared to reverse cationic flotation include a lower sensitivity to slimes and decreased flotation reagent expenses. A comparative study by Ma et al. (2011) (17) indicates that effective reverse anionic flotation could only be achieved for fine or ultrafine particle sizes. However, it is a consensus that collector dosage, size fractions, and pH levels greatly influence the recovery of iron-bearing minerals and concentrate grade in both cationic and anionic reverse flotation methods (11). Finally, this study intends to conduct reverse anionic flotation experimental tests to determine the iron recovery and concentrate grade of the iron ore in the Dawa-Moyale area, along with mineralogical characterization. Although this detailed study may not represent the entire vast area of iron ore formations, it provides valuable insights into the characteristics and concentration behavior of the ore deposit, contributing to the ongoing strategic goals of developing Ethiopia's mining sector, specifically regarding the Dawa-Moyale iron ore.

## 1.2 Statement of the Problem

The Precambrian crystalline basement found in the northern, western, and southern parts of Ethiopia hosts both metallic and nonmetallic mineral resources (18–20). Particularly, the southern part of the country, including the study area, is rich in various mineral resources, including precious metals such as gold and platinum, rare earth metals, base metals like nickel, copper, lead and zinc, oxide metals such as iron and chromium, as well as industrial minerals including kaolin, feldspar, clay, asbestos, talc, marble, limestone, and granite (20). Concentration methods such as Magnetic separation, gravity separation, and flotation are typically employed to concentrate intermediate and low-grade iron ores from gangue materials like quartz and clay.

The Dawa-Moyale area, located in the southern metamorphic terrain of Ethiopia, has been identified as a region with promising iron ore deposits. Geologically, this area comprises poly-deformed and metamorphosed mafic and ultramafic rocks (19). Previous studies in the Dawa-Moyale area have primarily focused on providing a general overview of the geology and the overall geological setting. However, recent studies conducted by the GSE have concentrated on geological, geophysical, and geochemical research for gold exploration. Consequently, the iron deposits in the Dawa-Moyale area have not been thoroughly investigated in terms of their concentration response, particularly concerning the reverse flotation method, and have not been characterized in detail. This study aims to investigate the effects of particle size, pH values, and the dosage of anionic collectors, with two levels of each factor, as well as their interaction on iron recovery and concentrate iron grade.

## 1.3 Research Objective

### 1.3.1 General Objective

The primary objective of this research project is to investigate a reverse flotation response for the beneficiation of iron ore around Dawa-Moyale, Somali region, Ethiopia

### 1.3.2 Specific Objectives

To achieve the overall goal of this research project, the following specific objectives were pursued:

- Identify and quantify the major, minor, and accessory minerals in the Dawa-Moyale iron ores using Ore Petrography Analysis and X-ray diffraction techniques.
- Determine the elemental composition of the Dawa-Moyale iron ores through AAS technology, providing insights into the major and trace elements that make up the deposit.
- Identify the effects of key experimental factors, such as particle size, collector dosage, and pH levels, and their interaction on the iron grade and recovery using the reverse flotation method.

## 1.4 Research Questions

Applying this experimental approach along with a mineralogical characterization of the iron ore, this project research will contribute to the ongoing strategic goals of developing Ethiopia's mining sector, specifically regarding the Dawa-Moyale iron ore and try to answer the following questions:

1. What iron-bearing minerals and associated gangue materials are present in Dawa-Moyale iron ore?

2. What are the major and minor chemical constituents of the ore samples?

3. What effects do the experimental independent variables such as collector dosage, pH, and particle size, and their interaction have on the iron grade and recovery using the reverse anionic flotation method?

## 1.5 Description of The Study Area

Dawa-Moyale is located on the Ethiopia-Kenya border (Figure 1). In Ethiopia, it serves as the administrative Centre for two Ethiopian woredas, one in the Somali region and another in the Oromia region. The Dawa-Moyale of the Somali region, which constitutes the province where the present study was conducted, is the administrative city of the Dawa zone within the Somali regional state. It is situated between latitudes 3°30′00″N to 3°42′00″N and longitudes 38°58′00″E to 39°27′00″E, covering an area of 1200 $km^2$. It is connected to Addis Ababa, the capital city of the country, by a well-paved asphalt road that traverses through Shashemene, Dilla, and Yabelo before reaching Dawa-Moyale. The climate in the Dawa-Moyale area is subtropical and hot, characterized by a long dry season and a short rainy season from March to June (21).

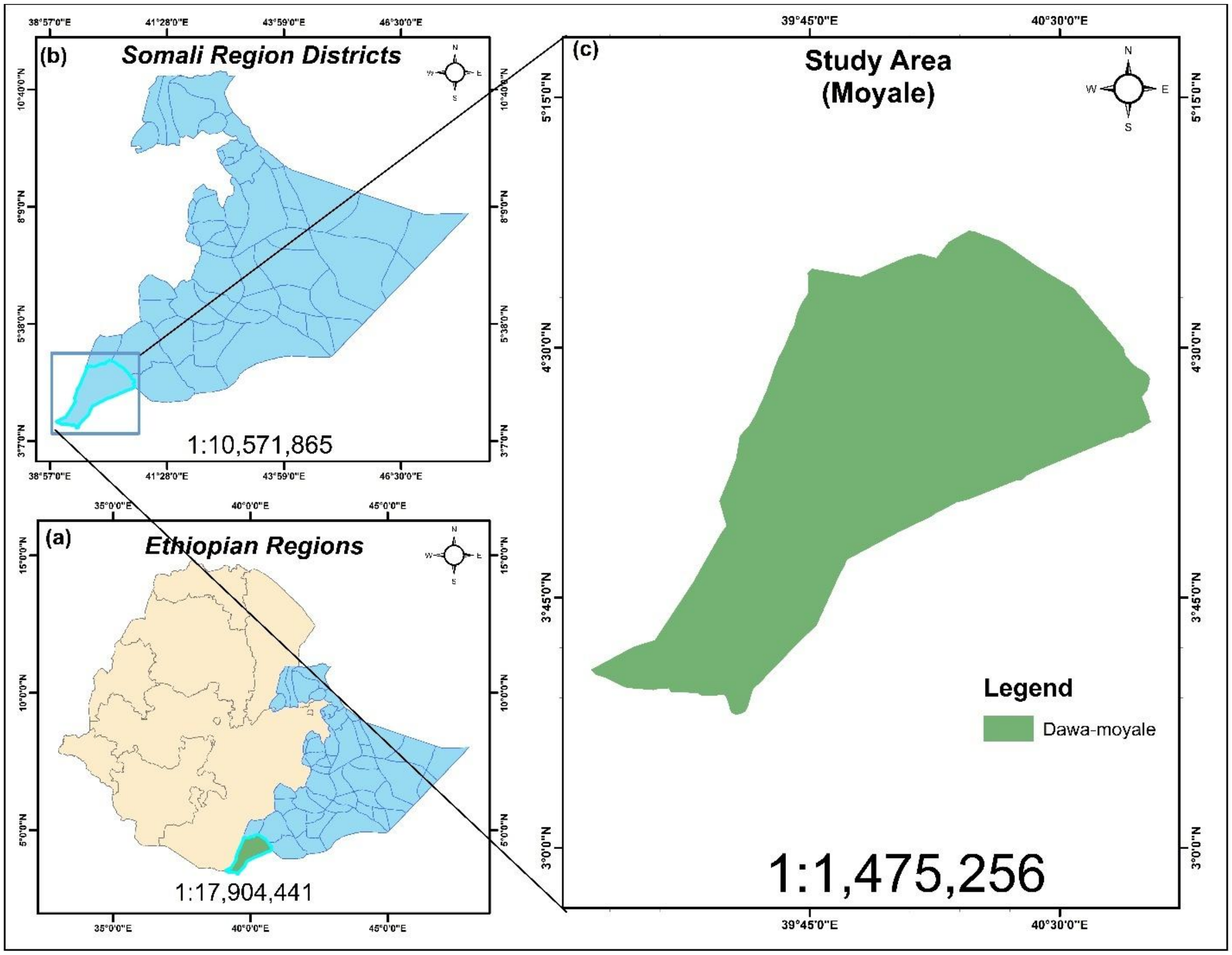



Figure 1. Location map of the study area

## 1.6 Scope of the Study

This study investigated the reverse anionic flotation of iron ores from the Dawa-Moyale area, Somali Region, Southern Ethiopia. The scope of the research includes a detailed literature review, iron ore characterization, and flotation experimental tests to better understand the ore's characteristics and potential for economic extraction. Specifically, this study encompasses the following key aspects:

- A comprehensive literature review of the lithological units and structural geology of the Dawa-Moyale area was conducted to understand the geological setting. Additionally, the review covers existing research on iron ore deposits, the mineralogical characterization of iron ores, and their beneficiation methods.
- Collection and description of rock samples from various locations within the study area.

- Mineralogical analyses using Ore Petrography Analysis and X-ray diffraction techniques to identify and quantify the primary and secondary mineral phases of the samples.
- Complete silicate analysis employing AAS to determine the concentrations of major, trace, and minor elements, offering insights into the overall composition of the ore.
- Laboratory reverse flotation tests were designed to study the effects of the particle size, pH level, and anionic collector dosage on the iron recovery and grade of iron-bearing minerals.
- A comprehensive report summarizing the findings and assessments made during the study.

## 1.7 Significance and limitations of the study

This research project holds significant academic importance and aims to provide valuable insights into the reverse flotation response of Dawa-Moyale iron ore. It seeks to fill a critical gap in the existing literature regarding the mineralogical characteristics and reverse flotation response of Dawa-Moyale iron ore. Additionally, the findings of this study could lead to substantial improvements in iron ore beneficiation during local mining at Dawa-Moyale. This could enhance the profitability of these activities and contribute to the economic development of the region. However, it is essential to acknowledge the limitations of the present study:

1. Time and resource constraints: Time and resource constraints restrict the ability to conduct thorough sampling across all deposit zones, comprehensive mineralogical and geochemical analyses, and to characterize all the flotation factors which could affect the response variables.
2. Laboratory-scale experiments: The reverse flotation experiments were conducted at the laboratory scale, which may not fully replicate industrial-scale conditions. Scaling up the process could introduce additional variables and challenges not observed in the laboratory process

# 2. Literature Review

## 2.1 Fundamentals of Iron

Iron (Fe) is the backbone of all industrial development and modern civilization (1). It is the primary raw material for steel production. In 1999, the global production of 780 million tons of raw steel required the mining of 992 million tons of iron ore (9). As of 2023, the global iron mine production is approximately 2.5 billion metric tons, with Australia being the world's leading iron ore producer. Australia contributes 960 million tons of crude iron ore, accounting for about 38% of global production (see Figure 1) (4). On the other hand, the estimated world crude iron ore reserves are around 87 billion metric tonnes in 2023 ( see Figure 2) (4).

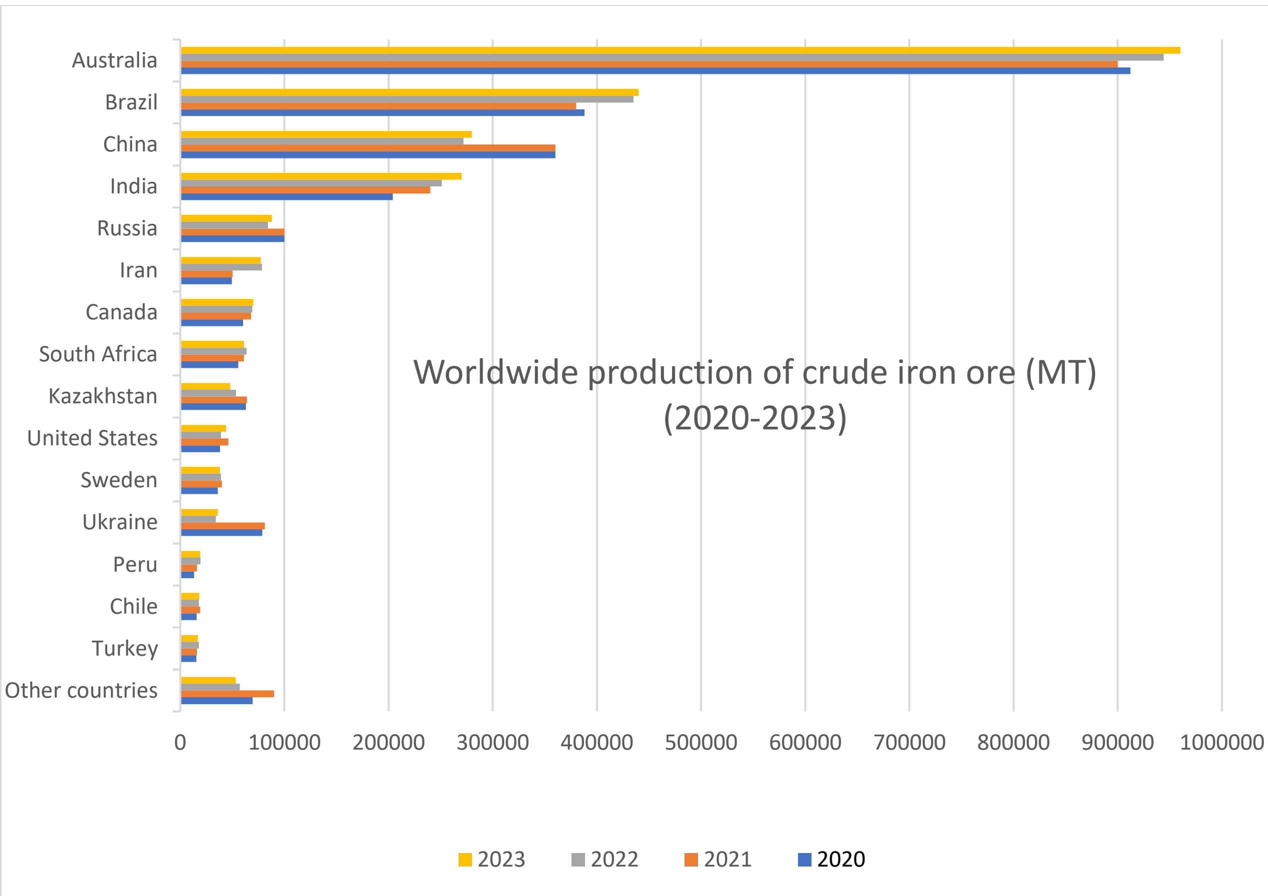



Figure 2. Worldwide crude iron ore production from 2020 to 2023 (4).

Iron is a common element in the Earth's crust, typically making up about 2% to 3% of sedimentary rocks and approximately 8.5% in mafic rock (9). Iron ore deposits are generally formed through three geologic processes: 1. Sedimentation process, which leads to the formation of BIF (Bedded

Iron Formations), 2. Magmatic activities that create segregation or replacement deposits, and 3. enrichment resulting from weathering at the surface and near-surface weathering (9). However, 90% of the iron-bearing minerals extracted globally originate from Precambrian BIF (Bedded Iron Formations), while the remainder comes from metasomatic and magmatic magnetite deposits (1). Additionally, larger quantities of low-grade resources can be found in oolitic ironstone and residual laterites.

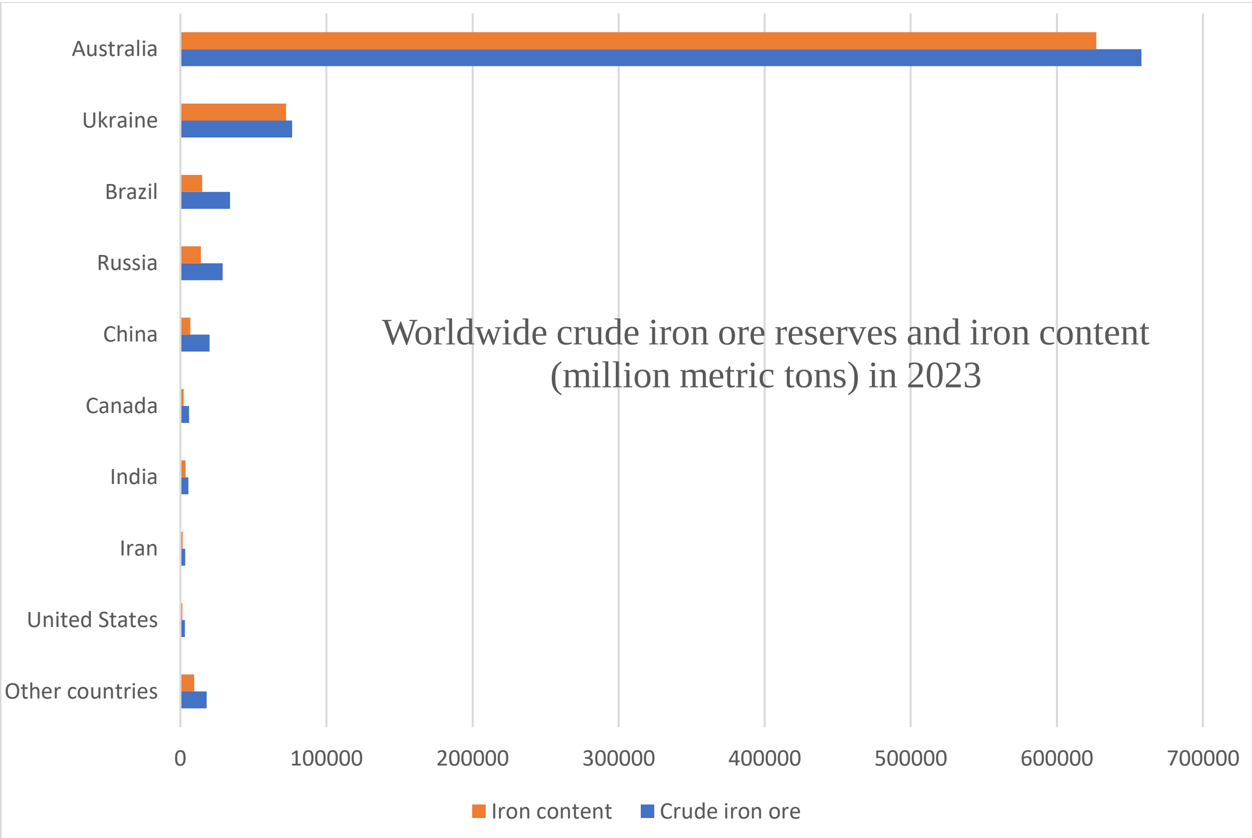



Figure 3. Distribution of crude iron ore reserves and iron content in various countries worldwide (4).

## 2.2 Iron Ore Characterization

More than 300 minerals have iron content, however, only five are recognized as the primary sources of iron extraction utilized in the steelmaking industry, as shown in Table 1 (22). Among these, hematite, magnetite, and goethite are the most economically viable iron-bearing minerals because of their larger quantities. These minerals are usually associated with gangue minerals and impurity elements, such as silicon, aluminium, phosphorus, sulfur, calcium, magnesium, sodium, potassium, titanium, and manganese, which are listed in Table 2. Previous studies have shown that the Dawa-Moyale iron ore deposit primarily consists of magnetite and hematite, with quartz, amphiboles, and other silicates serving as the main gangue minerals (23). The primary objective of mineral beneficiation technology is to produce a concentrate (containing valuable minerals) that meets satisfactory grades and recoveries, as well as tailings (gangue materials) that can be disposed of in an environmentally safe manner (24). Therefore, Mineralogical assessment is a paramount aspect that is required to be conducted before beneficiation (25–27). This stage involves using mineralogical data to address challenges encountered throughout the exploration, mining, and ore processing (28).

Table 1. The chemical and physical properties of essential iron-bearing minerals (22).

| Iron-Bearing Minerals | Chemical Name | Chemical Formula | %Fe (Iron, Wt.% | Colour | Crystal System | Specific Gravity | Mohs's Hardness | Melt Point(°C) |
|---|---|---|---|---|---|---|---|---|
| Hematite | Ferric Oxide | $Fe_2O_3$ | 69.94 | Steel grey to red | Hexagonal | 5.24 | 6.5 | 1565 |
| Magnetite | Ferrous–Ferric Oxide | $Fe_3O_4$ | 72.36 | Dark grey to black | Cubic | 5.18 | 6 | 1600 |
| Goethite | Hydrous Iron Oxide | $HFeO_2$ | 62.85 | Yellow or brown to nearly black | Orthorhombic | 3.3–4.3 | 5–5.5 | - |
| Siderite | Iron Carbonate | $FeCO_3$ | 48.2 | White to greenish grey to black | Hexagonal | 3.83–3.88 | 3.5–4 | - |
| Ilmenite | Iron–Titanium Oxide | $FeTiO_3$ | 36.8 | Iron-black | Hexagonal | 4.72 | 5–6 | 1370 |
| Pyrite | Iron Sulfide | $FeS_2$ | 46.55 | Pale brass-yellow | Cubic | 4.95–5.10 | 6–6.5 | - |

Understanding iron minerals in terms of mineral processing assists engineers in selecting an appropriate concentration process. Additionally, it is essential to identify the rock-forming minerals (gangue minerals) associated with iron carriers, as some may interfere with the chosen processing methods (27). Minerals are primarily identified based on their optical characteristics, which vary from one mineral to another due to differences in crystal structure and chemical composition (26). An optical microscope is the primary tool for identifying minerals. The study of iron ore minerals typically takes place under a reflected light microscope, whereas rock-forming (gangue) minerals are analyzed using a transmitted light microscope in polished and thin sections (26). However, given the vast diversity of the mineral kingdom, it can sometimes be difficult to identify all the minerals solely using an optical microscope. Additionally, the magnification capability of optical microscopes typically ranges from 50 to 1000 times. In such cases, advanced characterization techniques like X-ray diffraction, scanning electron microscope with micro-chemical analysis using energy-dispersive X-ray spectroscopy and wave-dispersive X-ray spectroscopy, and electron probe micro-analysis are employed. The latter techniques help in the determination of the mineral phases, which is very important.

The textural study, including particle or grain size distribution, mineral surface coatings, interlocking patterns, hardness or softness of the adjacent minerals, and mineral compositions, is crucial for determining size reduction and liberation in mineral processing (26). MLA is an advanced analytical technique that provides detailed liberation characteristics of ore samples, allowing for precise tailoring of recovery processes (29). However, this aspect is beyond the scope of the present study.

Table 2. Associated gangue minerals and their elemental forms (30)

| Elemental forms | Associated gangues |
|---|---|
| Alumina | • Gibbsite (hydrated aluminium oxide) |
| | • Kaolinite (a layered aluminosilicate mineral of general chemical formula $Al_2Si_2O_3(OH)_4$) |
| | • Other gangues like ferruginous clay in minor quantities |
| Silica | • Quartz, |
| | • Quartzite |
| | • Chert |
| | • Kaolinite |

| | |
|---|---|
| | Other than these minerals, sminnesotaite and greenalite are also silicate minerals which are found associated in very minor quantities with iron ore |
| Phosphorous | • Apatite |
| | • Hydroxylapatite |
| | • Fluorapatite |
| | • Chloropatite |
| | • Vollophane |
| | • Bromapatite |
| Sulphur | Found in the form of iron sulphide minerals.<br>iron sulfide minerals are |
| | • Pyrite |
| | • Marcasite |
| | • Pyrrohotite |

Research by Young et al. (2019) concluded that it is essential to identify trace elements that are harmful to the environment, such as arsenic (As), bismuth (Bi), antimony (Sb), mercury (Hg), Selenium (Se), tellurium (Te), etc.,

AAS is a widely recognized and accepted analytical technique for identifying and quantifying trace and ultra-trace levels of elements and metals (31). Its high accuracy and reliable precision have positioned it as one of the leading methods in elemental analysis. Utilizing AAS, one can assess the concentration of metals such as iron in rock samples to evaluate the feasibility of mining these rocks for iron extraction. X-ray fluorescence spectroscopy is another widely used analytical technique in ore characterization, recognized for its ability to provide rapid, non-destructive, and accurate quantitative and qualitative elemental analysis (32). In mineral processing, XRF is employed to determine the bulk chemical composition of ores. This information is critical for identifying a sample's major, minor, and trace elements and directly influences mineral processing methods.

Generally, integrating various analytical techniques in process mineralogy offers a comprehensive understanding of ore characteristics, mineral associations, and elemental distribution, which are critical for optimizing mineral processing. Concurrently, Christopher P. B. (2008) (29) suggested that the design and efficient management of complex ores have become more dependent on ore characterization. According to Young, Courtney A. (2019) (33), the ore beneficiation process must always be evaluated about the minerals makeup of the ore to accurately predict the liberation size of the particles and to determine the appropriate type of concentrator required. Understanding the mineralogical composition and physicochemical properties of iron-bearing minerals and associated gangue minerals is crucial for selecting and designing an efficient beneficiation process for the Dawa-Moyale iron ore deposit in southern Ethiopia.

## 2.3 Iron Ore Beneficiation

The general iron ore beneficiation methods include comminution, wet screening and desliming, gravity concentration, magnetic separation (low and high intensity, dry and wet), flotation, and selective flocculation to produce a concentrate rich in valuable minerals and a tailing predominantly composed of gangue materials (34).

Comminution represents the initial stage in the beneficiation of iron ores, comprising the crushing and grinding of run-of-mine ore to liberate the valuable mineral from the gangue constituents (29). Following the blasting of ore deposits, freshly excavated iron ore is transported via scrapers and conveyors to the mineral processing plant (29). The Crushing process, which is executed as a dry operation, involves the compression of the iron ore against a rigidly fixed surface, facilitating easier transportation and yielding material of controlled particle size (8). The wet grinding process is implemented upon achieving a sufficiently reduced particle size through primary, secondary, or tertiary crushing units. This process is characterized by the abrasion of ore using free-moving stainless steel media, including rods, balls, or pebbles, further liberating valuable minerals from unwanted gangue minerals (8).

Notably, size reduction is often the most significant energy consumer within mineral processing, accounting for up to 50% of total energy consumption (28). However, this process is essential for liberating the ore particles and increasing the surface area of the ore, thus enabling their effective recovery in subsequent beneficiation stages (8). The overarching goals of minerals liberation include 1) The Separation of target minerals from gangue, 2) The Separation of target minerals from one another, and 3) the Exposure of mineral surfaces for further reaction, such as flotation and leaching.

The effectiveness of comminution heavily influences the success of ore recovery, as inadequate liberation may result in the loss of iron ore within waste material. Baawuah et al. (2020) (35) demonstrated a consistent increase in the proportion of fully liberated magnetite particles in finer-size fractions. However, this trend was reversed for particles smaller than 38 µm. Ma et al. (2011) (17) reported a decrease in the efficiency of both cationic and anionic reverse flotation processes for particle sizes coarser than 75µm. The comminution stage generates a spectrum of particle fragments, which may range from fully liberated particles to binary and tertiary composites (see Figure 3) (36).

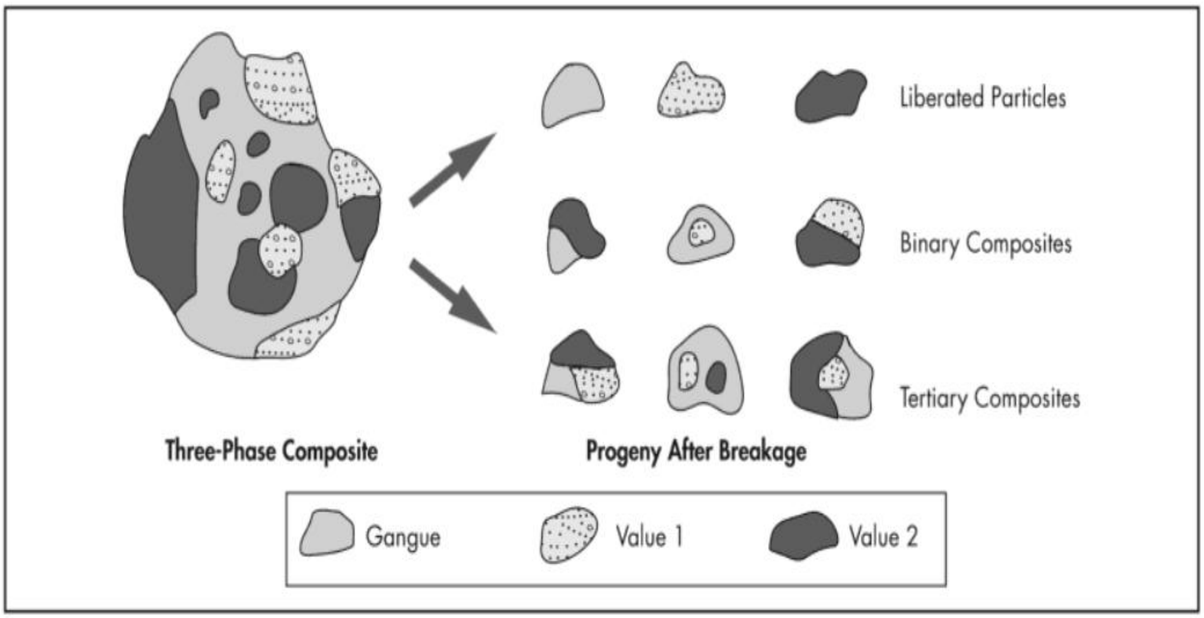



Figure 4. This illustrates the products of rock fragmentation and shows that the daughter fragments of a complex ore can be liberated particles, binary composites, or tertiary composites (33).

The Classification of crushed or ground products is an important step preceding any mineral processing operation, as all processing equipment is specifically designed to function optimally within designated material size ranges. Size separation can be effectively accomplished by screening or classifying coarse and fine particles (37). The primary objectives of individual screening include removing undersized materials unsuitable for reprocessing via crushers and eliminating oversized materials and debris that should not advance to subsequent processing stages. Industrial screening is predominantly employed for separations involving particles larger than 0.2 mm and is frequently integrated with crushing equipment, given that efficiency markedly declines for smaller particle sizes (37). Various types of screening equipment are utilized, including stationary and vibrating grizzlies, roll grizzlies, revolving screens, vibrating screens, shaking screens, reciprocating screens, sieve bends, and rotary sifters (38). Conversely, mineral particles, deemed too fine for effective sorting through screening, are typically processed using wet classifiers (8). Classifiers can be classified based on the equipment design into horizontal current and vertical current types or mechanical, non-mechanical, sedimentation, and hydraulic or fluidized-bed types, depending on the equipment design (37). Although size-based separation can sometimes produce a marketable mineral product, it often fails to separate valuable minerals from waste rock effectively. Mineral concentration can be achieved using physical and chemical properties (37).

The primary goal of mineral concentration regardless of the methods employed, is to separate run-of-mine materials into valuable minerals found in the concentrates, gangue materials found in the tailings, and locked particles in the middling (8). The most common techniques for concentrating iron-bearing minerals are dry and wet low-intensity magnetic separation, due to the ferro-paramagnetic properties of the iron ores. However, the processing of iron ores must be tailored to their mineralogy, textural relationships, and the degree of liberation of the iron-bearing minerals from the gangue (7). Gravity concentration methods such as jigs, shaking tables, spiral concentrators, and centrifugal gravity separators are rarely used for iron ore concentration. In contrast, flotation is employed to upgrade concentrates from magnetic separation by reducing the silicate content in the concentrate (22, 32, 36). Figure 4 illustrates a typical beneficiation flowsheet of magnet ore.

## 2.4 Froth Flotation

Froth flotation is a common technique for selectively concentrating hydrophobic particles from hydrophilic ones, thereby enriching valuable minerals with the help of chemical reagents (40). It is one of the most vital methods for concentrating sulfide ores, finely disseminated low-grade metallic ore bodies, or where the gravity difference between minerals is too small (8). Froth flotation effectively concentrates fine iron ores that are smaller than -100 mesh and has been shown to improve magnetite concentrates at four taconite operations in Minnesota (7). Additionally, it serves as the primary recovery method for hematite ores, as demonstrated at the Tilden mine in Michigan, United States (33). Since the introduction of iron ore flotation in the United States in

1931, direct flotation, and reverse flotation routes have been practiced; in direct flotation, the goal is to float the iron ore into the froth product, whereas in reverse flotation unwanted gangue materials are floated to the froth and subsequently discarded (7, 13, 15, 18). Depending on the type of collector applied in the process, reverse flotation is categorized into cationic and anionic types.

Reverse anionic flotation was first established in China, utilizing anionic collectors to float silica gangues activated with lime, followed by flotation with fatty acids as a collector at a high pH (11-12) while employing starch to depress the iron-bearing minerals (13). Hanna Mining and American Cyanamid also developed direct flotation and reverse anionic flotation during the 1930s and 1940s. By the 1950s, these methods were applied in Minnesota and Michigan concentrators (14). During the same period, reverse cationic flotation was developed by the United States of America Bureau of Mines, which ultimately became the most feasible route for intermediate and low-grade iron ore flotation in the United States and other Western countries (18, 19).

Reverse Cationic flotation employs amine collectors and starch depressants to selectively float silica-based gangue minerals, such as quartz, under alkaline conditions (9, 15). However, for hematite ores, a depressant is often necessary to prevent the flotation of hematite, ensuring a selective separation.

The benefits of reverse anionic flotation over reverse cationic flotation include its lower sensitivity to slimes and reduced reagent costs (17). However, a comparative study by Ma et al. (2011) (17) indicates that effective reverse anionic flotation can only be achieved for fine or ultrafine particle sizes. This presents a challenge as the necessity for processing these fine particles can lead to increased costs. Specifically, additional energy is required during the comminution stage for size reduction, along with higher reagent consumption.

An effective flotation process occurs when the valuable and gangue minerals have different affinities for air and water in the direct or reverse flotation (15, 35). However, Flotation performance is significantly influenced by various parameters (42). Klimpel (1984) categorized the primary variables into three principal groups (see Figure 5), indicating that careful experimentation is essential for thoroughly analyzing each variable.

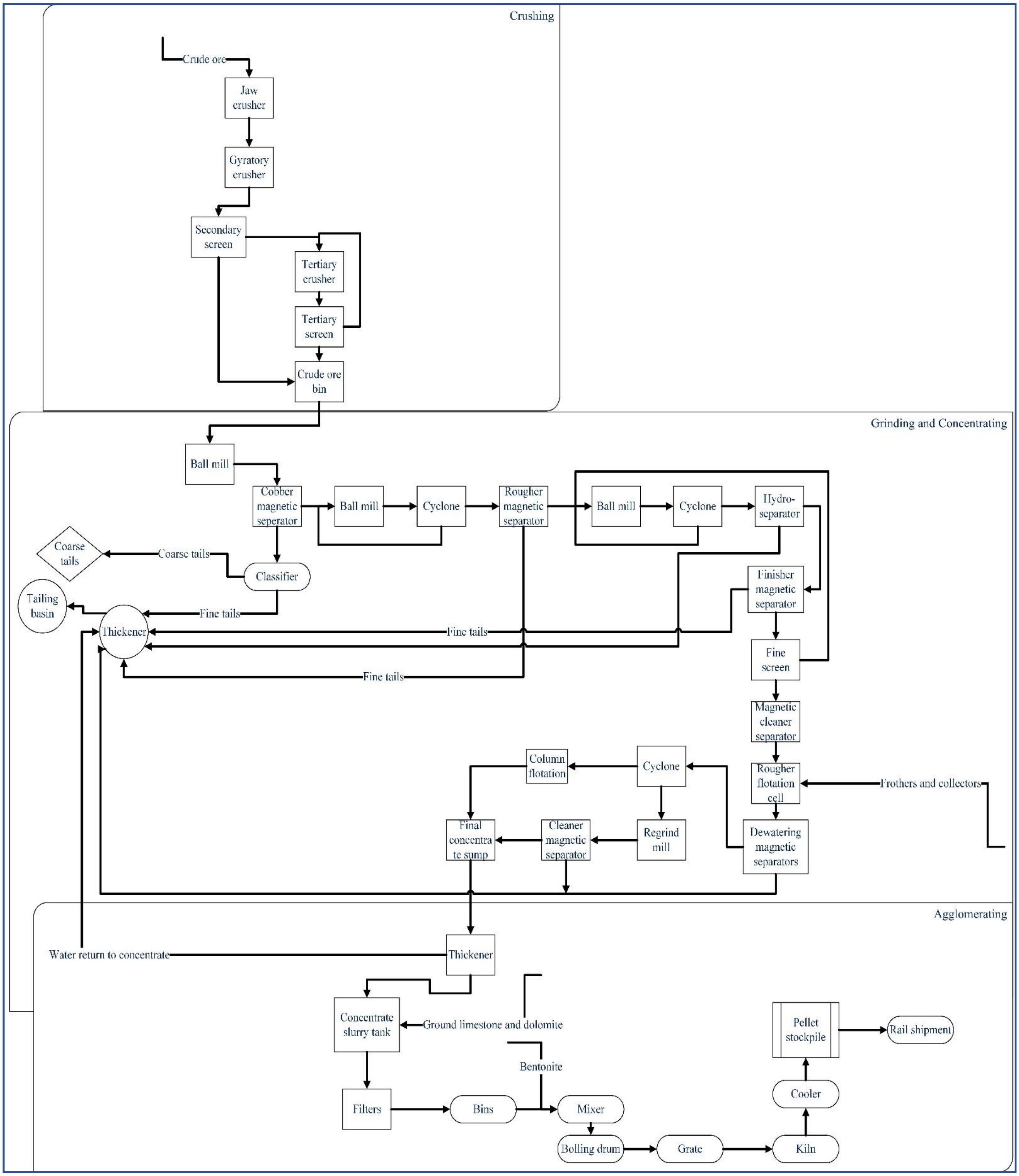



Figure 5. Minntac magnetite ore beneficiation flowsheet (33)

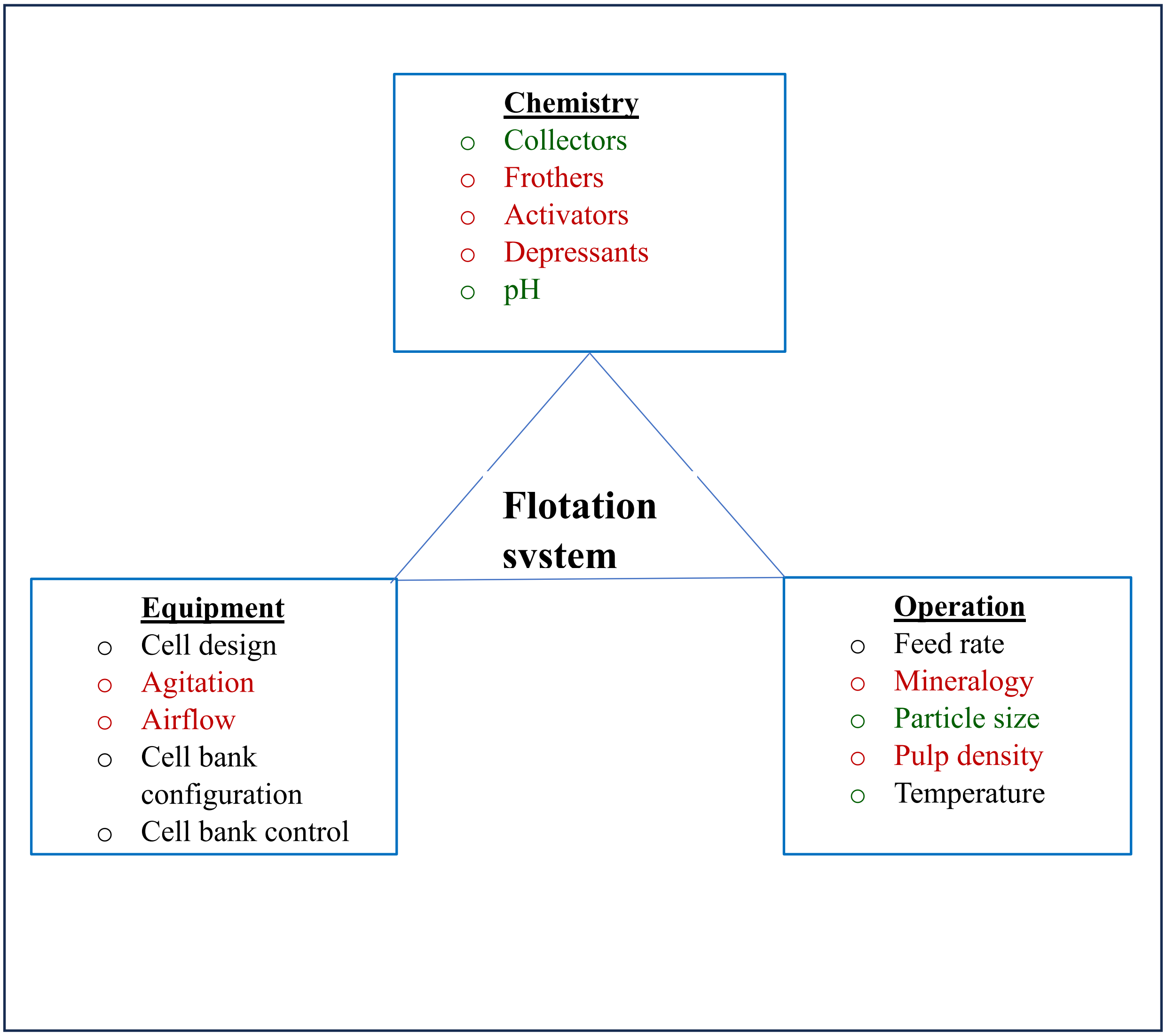



Figure 6. Summary of variables in the flotation system adapted from Klimpel, 1984 (29). Variables shown in green were the manipulated variables, while the red colored were the controlled variables of the present study.

### 2.4.1 Flotation reagents

Flotation reagents are introduced into the pulp to modify the surface chemistry of minerals by enhancing the hydrophobicity difference among them, which facilitates the separation of valuable minerals from gangue materials (40). These reagents are incorporated into the slurry and distributed using an impeller. Several chemical reagents are used in flotation to modify the surface properties of particles. Collectors, frothers, depressants, dispersants, activators, and pH regulators are the most common flotation reagent types used in the flotation process, where each has a specific purpose, although some reagents may have multiple purposes (43). The flotation process depends on the reagent introduced to the water suspension of the finely ground iron ores, which selectively causes either iron oxide minerals or gangue particles to develop an affinity for air. Minerals that exhibit this affinity adhere to the air bubbles, moving through the suspension, and are extracted as a froth product. Reagents that are introduced to boost the affinity for air are typically referred to as collectors. Those that aid in the creation of a stable bubble or froth are called frothers. Additionally, substances used for regulatory purposes, such as adjusting pH levels or improving dispersion or flocculation, are termed modifiers (33). In the mineral processing practice, the metallurgists must consider the flotation reagents' ultimate goal, which is to select the appropriate combination of chemicals that most economically and selectively separate the desired mineral phases with high recovery (38).

2.4.1.1 Collectors

In the flotation process of iron ore, collectors play a crucial role as reagents. Their main purpose is to increase the attraction between mineral particles and air bubbles by forming a hydrophobic layer on the surface of the mineral particles (44). These reagents are organic compounds that improve the hydrophobic properties of specific minerals by adsorbing molecules or ions onto their surfaces (7, 12). This adsorption decreases the stability of the hydrated layer that isolates the mineral surface from the air bubbles, making it easier for mineral particles to attach to the bubbles when they come into contact (8). To evaluate the flotation potential of iron oxides, researchers typically employ highly soluble collectors composed of organic molecules with carbon chains ranging from 10 to 18 units in length.

During the beneficiation of iron ore using the reverse flotation technique, unwanted minerals such as quartz, apatite, fluorapatite, kaolinite and gibbsite are removed by depressing the iron-bearing minerals (11). Anionic collectors dissolve in a solution, they break apart into negatively charged active components that attach to the positively charged surfaces of minerals. In the flotation process of minerals containing iron, common anionic collectors include Fatty acids, resin acids, soaps, alkyl sulphates, and sulphonates (11). In contrast, cationic reverse flotation employs amine collectors and starch depressants to selectively float silica-based gangue minerals, such as quartz, under alkaline conditions. Cationic collectors typically consist of primary aliphatic amines or diamines, beta-amine, or ether amines, usually in acetate form (33).

#### 2.4.1.2 Frothers

Frothers are surface-active compounds with distinct properties(8). They consist of polar and non-polar groups, which together help in bubble formation and stabilize froth. Frother lowers the surface tension of water, creating an environment conducive to froth formation (8). Various classifications of frothers exist in the literature, with one system proposed by Wills (2005) categorizing them as acidic, basic, or neutral. Frothing agents frequently used in the flotation of minerals containing iron include pine oils, aliphatic alcohols, polypropylene glycols, alkyl ethers of polypropylene glycol, and cresylic acids. These alcohols generally have five to eight carbon atoms, examples being mixed amyl alcohol, methyl isobutyl carbinol, and certain heptanols and octanols (11). However, Filippov, et al., (2014) (7) noted that in the reverse cationic flotation of iron ores, frothers are rarely used because the partially neutralized ether mono- and diamines that serve as collectors also act as frothers.

#### 2.4.1.3 Regulators

Regulators, also known as modifiers, are commonly employed in the flotation process to adjust the work of the collector by enhancing or reducing its water-repelling effect on mineral surfaces (8). This adjustment improves the selectivity of the collector towards specific minerals. Regulators can be divided into three categories: activators, depressants, and pH modifiers. Activators are used to enhance collector adsorption under certain conditions (10, 32). The careful selection of activators is essential to avoid activating gangue minerals, as their adsorption negatively impacts the overall flotation performance.

Depressants improve flotation selectivity by rendering specific minerals hydrophilic, thus inhibiting their flotation. Corn starch is the most commonly utilized depressant in iron ore flotation due to its widespread availability and effective iron oxide depressing capabilities. However, research by Filippov, et al., 2014 (7) emphasized the importance of the amylose to amylopectin ratio in iron oxide depression, demonstrating that superior results were achieved using starch with an amylopectin to amylose ratio of 75%/25% compared to pure amylopectin. Dextrin, CMC, and humic acid are also common depressants utilized in iron oxide flotation. Research by Filippov, et al., (2019) (45) indicated that dextrin outperforms corn starch at a pH of 10.5. The study also revealed that pH significantly impacts iron recovery when using CMC as a depressant, with recovery rates improving as pH levels rise.

A crucial factor in selecting flotation reagents for a specific ore is determining the appropriate pH value and corresponding pH modifier (24). The alkalinity of the slurry plays a crucial role in the selective flotation of iron ore. It can be modified by introducing reagents such as lime, sodium carbonate (soda ash), and, to a lesser extent, sodium hydroxide or ammonia. Typically, the pH range for iron ore flotation falls between 8.5 and 11.5 (7, 10, 13, 15, 38).

**2.4.2 Effect of particle size**

Numerous studies have examined the influence of particle size on flotation efficiency (37, 38). Reverse flotation has demonstrated efficacy in the iron ore industry for particles smaller than 150µm (46). The flotation rate constant is dependent on particle size, with floatability increasing as particle size decreases until reaching a critical point, after which it begins to decline (38). Coarse particle sizes may result in poor concentrate grade and recovery due to insufficient mineral liberation, while fine particle sizes can lead to increased costs in flotation because of their extensive surface areas, which can cause excessive reagent absorption (47).

Research conducted by Ma, et al., (2011) comparing two flotation methods reveals that reverse cationic flotation is more effective for larger particles (>210 µm), whilst reverse anionic flotation yields better results for smaller particle sizes. In contrast, Iwasaki (48) reported that the effectiveness of quartz flotation diminishes as particle size increases (>75µm), regardless of whether cationic or anionic flotation is employed.

A study conducted by Lima et al.(46), in which a reverse flotation experiment was performed to examine how different particle size ranges affected the flotation process, investigated three size fractions: -150 (global), -150+45 (coarse), and -45 (fine) µm. Their findings revealed that for the coarser fraction (−150+45µm) at pH 9.5, the silica content in the concentrates increased as amine dosages were raised. However, this increase in silica content was not observed in the -150µm (global) and -45µm (fine) fractions under the same conditions.

Generally, the relationship between particle size and flotation processes is interconnected. Therefore, it is recommended to identify the optimal mean particle size at which both recovery and grade are maximized.

# 3. Materials and Methods

This section outlines the methods and procedures used to investigate the reverse flotation response of Dawa-Moyale iron ore in the Dawa zone, Somali region, southern Ethiopia. It details the research design, instruments and apparatus, reagents, sample collection and preparation, ore characterization, reverse flotation experimental tests, and data analysis techniques employed in the present study.

## 3.1 Research Design

This research project employed a quantitative experimental design to assess the reverse flotation response of Dawa-Moyale iron ore under a 2-level full factorial design. The experimental procedure conducted in the present study and the subsequent data analysis were summarized in Figure 7. The process included collecting iron Samples from various localities in the Dawa-Moyale area, preparing the samples, particle size distribution analysis, conducting Ore Petrography Analysis, performing X-ray diffraction analysis, and carrying out chemical analysis using AAS. Additionally, reverse flotation experimental tests were conducted, followed by data analysis.

## 3.2 Materials

### 3.2.1 Instruments and Apparatus

The experimental apparatus employed in the present study comprises various instruments and devices. These include a Jaw-crusher (RoHs53743), Centrifugal miller (RETCH-56402), ball mill and Sieve-Shaker (RETCH-A200) all manufactured in Germany at AAiT laboratory. Additionally, an Ore Petrography Analysis and AAS were utilized at the EGI central laboratories. Further equipment used at the AAiT industrial chemistry laboratory encompassed a Denver flotation cell, a universal hot-oven, and an electronic balance.

### 3.2.2 Reagents

The reverse flotation technique is an effective way to concentrate iron ore to float impurities such as silica, alumina, and phosphorous away from the iron-bearing minerals (17). In the present study, corn starch, oleic acid, and pine oil were used as a depressant, collector, and frother, respectively. These reagents were purchased from FINEBAZ Chemical General Trading PLC, Addis Ababa, Ethiopia.

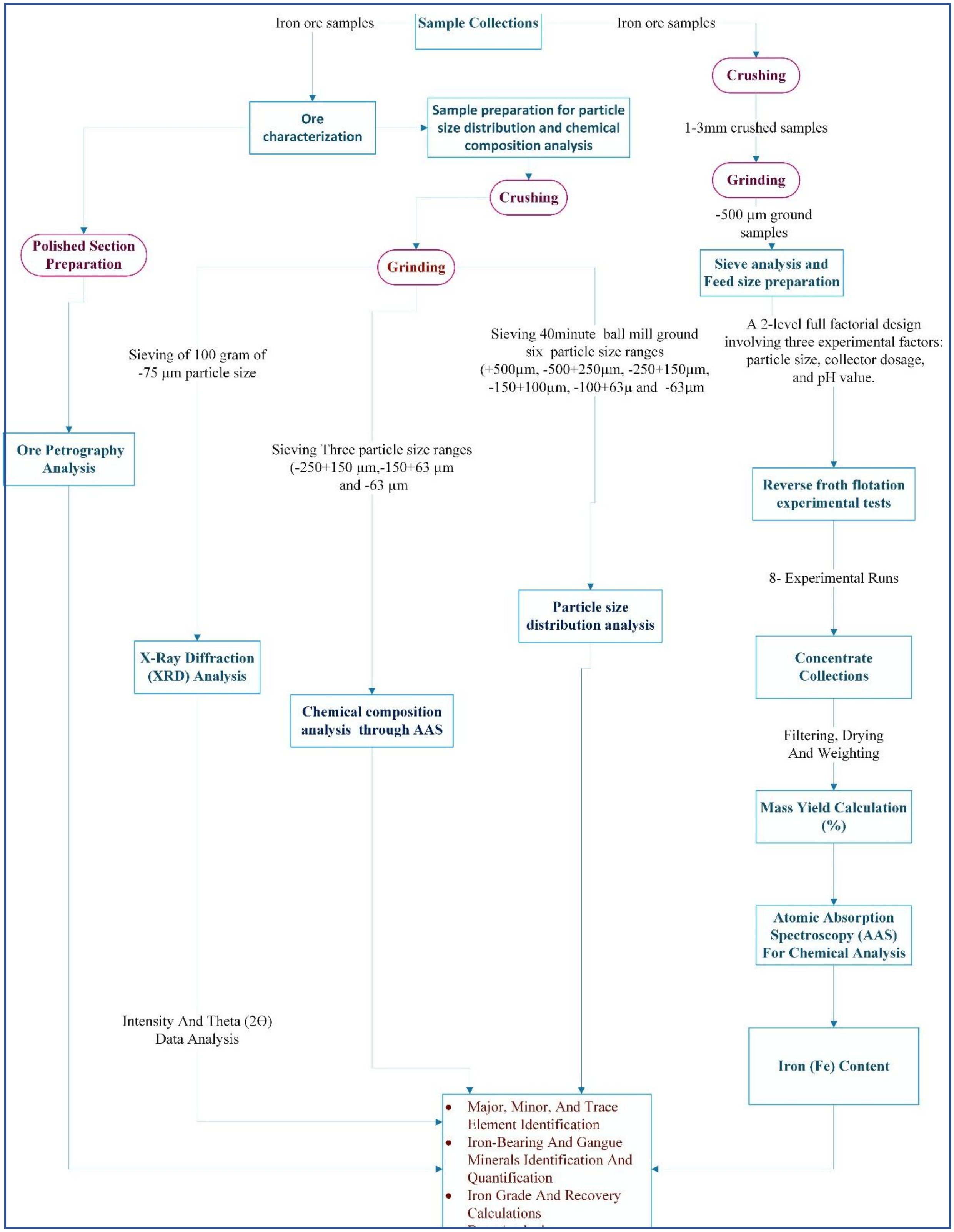



Figure 7.The overall research framework of iron ore characterization and sequential beneficiation using reverse flotation techniques.

Sodium hydroxide (NaOH) and calcium chloride ($CaCl_2$) utilized as a pH regulator and silica activator, respectively, were obtained from the Laboratory of AAiT. Fresh solutions of 2.5 % corn starch, 5% sodium hydroxide, and 5% calcium chloride were prepared daily to ensure consistency and reliability during the laboratory experiment.

## 3.3 Methods

### 3.3.1 Raw Material Collection and Preparation

Fifteen representative samples of iron ore, were collected from different localities of small-scale mining boreholes and outcrops in the Dawa-Moyale area of southern Ethiopia. These samples were stored in clothes and plastic-made sacks labelled with sample codes and numbers. The samples were then transported to the AAiT laboratory, where they were subjected to oven drying at $100^0$C for 24 hours. After drying, the samples were crushed using a laboratory jaw crusher within the closed set of a 3mm sieve.

A grinding test was conducted in a cylindrical laboratory ball mill to obtain a fine powder product of approximately 500 µm or less. After comminution, iron ore samples underwent particle size distribution utilizing a sieve shaker (RETCH A200, Germany) at the AAiT for the reserve flotation experiment. Accordingly, the mesh sieve was arranged in descending order of size as i. 500 µm ii. 250 µm, iii. 150 µm, iv. 100µm, v. 63, and vi. pan. Finally, two level particle sizes (-150+63µm and -63µm) were prepared to perform laboratory-scale reverse flotation tests. This allowed for an examination of the effect of particle size on the performance of reverse flotation.

### 3.3.2 Ore Characterization

3.3.2.1 Chemical Analysis

AAS was employed to determine the whole-rock chemistry and elemental distribution in the different particle size intervals and the chemical analysis of the reverse flotation concentrates at the EGI central laboratories. This information is essential for identifying a sample's major, minor, and trace elements, as well as the iron content in the concentrate, which directly influences mineral processing methodologies.

3.3.2.2 Ore Mineralogy

**Ore Petrography Analysis**

Among the rock samples collected from the field, one sample was prepared as a polished section for Ore Petrography Analysis at the EGI central laboratories. The petrographical analysis of this ore sample aimed to identify the mineralogy of the polished section quantitatively and qualitatively.

**X-ray diffraction**

A sample of 100 grams of homogenized ground material with a particle size of -75 µm was analyzed using X-ray diffraction to identify the crystal phases present in the ore, both qualitatively and semi-quantitatively, at the inorganic chemistry laboratory, school of chemistry, faculty of science, Addis Ababa University. XRD provides valuable information about the crystalline structure of minerals, which is essential for understanding their flotation behavior.

### 3.3.3 Reverse Flotation Experimental Design

Previous researchers have conducted experiments on iron ore beneficiation from various locations and determined that reverse flotation is the most widely used method for treating fine-grained, lower-quality iron ores like those found in Dawa-Moyale. The reverse flotation experiments were conducted at AAiT industrial chemistry laboratory to determine the flotation variables: feed particle size, pH value, and collector dosage, which have statistically significant effects and their interaction with the response variables, namely iron grade and recovery. The experimental factors and their levels of investigation are shown in Table 3. This study selected a 2-level Full factorial design over the other design methods as this type of design investigates the effect of multiple process parameters and their interaction effects on the response variables (49).

Table 3. Experimental factors and their levels

| Factor type | Experimental Factors | Units | Coded factor | Low | High |
|---|---|---|---|---|---|
| [Numeric] | Particle Size | µm | A | -63 | -150+63 |
| [Numeric] | Collector Dosage | g/t | B | 83 | 125 |
| [Numeric] | pH | | C | 9.5 | 11.5 |

All the experiments (8 numbers) were carried out based on the randomized experimental run order generated by the Stat-Ease 360 software. Accordingly, the optimum parameters of particle size, collector dosage, and pH value were determined through preliminary tests, after which comprehensive experimental tests were conducted utilizing these established optimal parameters. Throughout the experiment, several variables remained constant to ensure that any observed change in the dependent variables could be attributed to the parameters under investigation. These constants included 25 g/t of pine oil frother, 208g/t of 2.5% corn starch Depressant, 240g of raw materials, 208g/t of 5% calcium chloride ($CaCl_2$) activator, and an agitation speed of 1500 rpm for both conditioning and flotation, as well as 10 minutes flotation.

3.3.3.1 Experimental Procedures

Using a Denver laboratory flotation machine with a 3-litre capacity, the following procedures were applied to conduct reverse froth flotation experiments.

1. The cell was filled with 240 grams of iron ore samples having different particle size ranges based on the randomized order of the experimental runs and 2 litres of water and agitated for 3 min with an impeller speed of 1500 rpm.
2. A 5% NaOH solution was used to adjust the pulp pH if necessary, varying to 9.5 and 11.5 pH values.
3. Corn starch, calcium chloride (CaCl), and oleic acid were introduced into the cell at 3-minute intervals to depress the iron-bearing minerals, activate silica, and remove silica, respectively.
4. A frothing agent, pine oil, was added at a dosage of 25g/t during all the experimental runs and conditioned for two minutes.
5. After conditioning the slurry, compressive air was injected into the pulp to create bubbles, followed by manually collecting the froth product.
6. At the end of each test, the products were filtered and dried in a hot oven at 100 $^0$C for 24 hours.
7. The concentration yield (%) was calculated according to equation (1) (35).

$$\text{yied } (\%) = \frac{M_c}{M_f} x \ 100 \qquad \text{Eq. (1)}$$

Where $M_c$ is the weight of the flotation concentrate, and $M_f$ is the weight of the sample (weight of the feed) in grams.

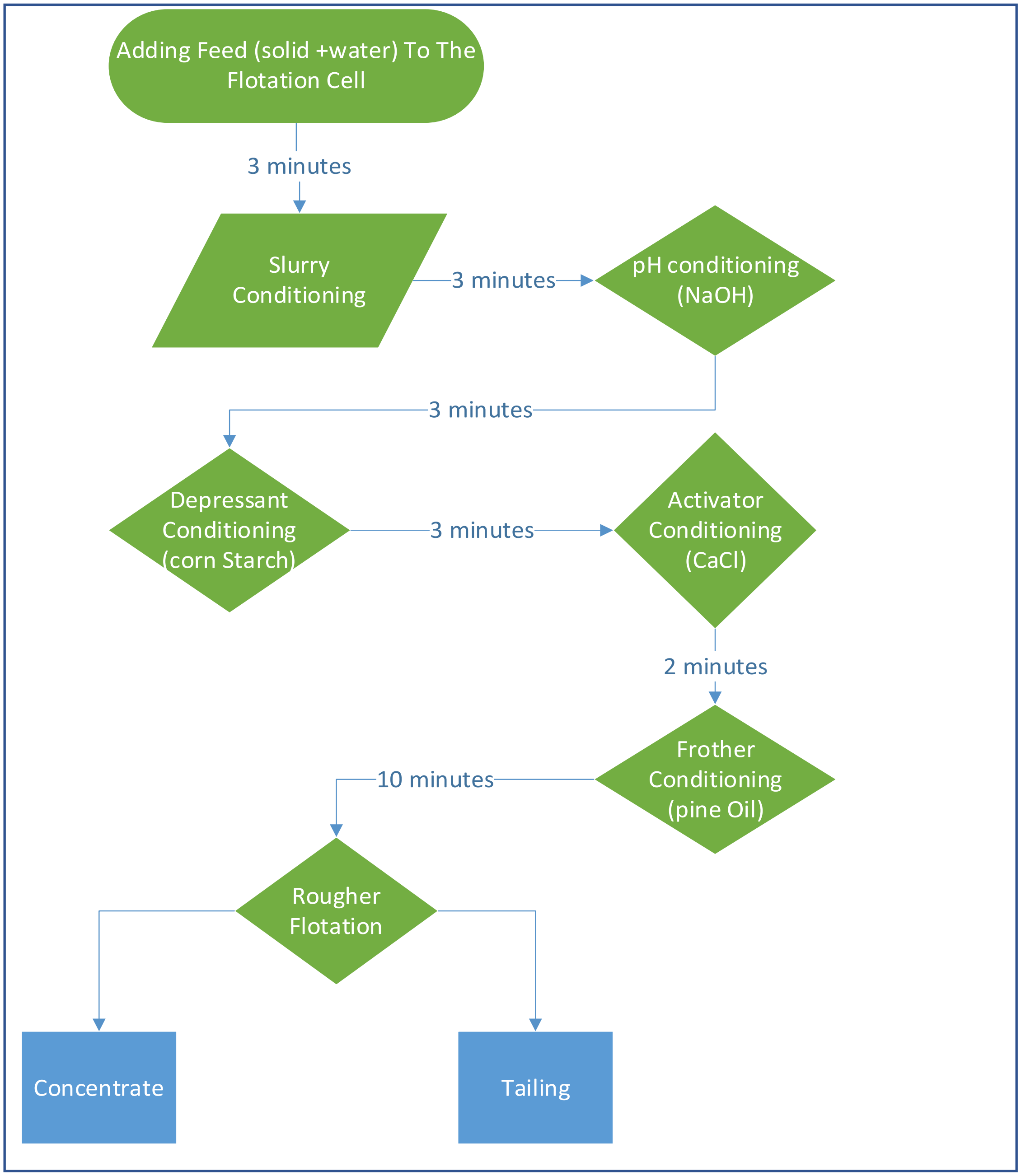



Figure 8.The schematic representation of the conditioning process employed in the experimental reverse flotation methodology of the present study.

8. Using AAS, complete silicate analyses were conducted on all flotation concentrates to determine the iron (%Fe) content in the concentrate. The concentrate grade (% Fe) and iron recovery(% Fe) of each experiment were calculated using equations (2) and (3), respectively (50).

$$Iron\ (Fe)\ Grade\ (\%) = \frac{m\ x\ assay\ of\ the\ valuable\ mineral\ (magnetite\ (Fe3O4))}{100} \quad \text{Eq. (2)}$$

$$Iron\ (Fe)\ Recovery\ (\%) = \frac{Yc}{f} \quad \text{Eq. ( 3)}$$

Where **Y** is the yield of concentration in %, **c** is the grade of iron (Fe) in the concentrate, **f** is the grade of iron (Fe) in the feed, and **m** is the maximum (theoretical) iron % in the mineral being concentrated (~72.36 % for magnetite). The flowsheet of the conditioning reverse flotation experiment is presented in Figure 8.

3.3.3.2 Experimental Data Analysis

Using statistical software (Stat-Ease 360 Trail version), all the experimental data was analyzed. This software facilitated modeling the predictive equations of concentrate grade (% Fe) and recovery (% Fe), which are the functions of the particle size of the feed, collector dosage, and pH values. The study examined the statistical significance of the independent variables, as well as their interaction effects on grade and recovery through an analysis of variance (ANOVA).

To evaluate the adequacy and reliability of the model, this study analyzed half-normal plot to identify the significant effects of the flotation variables on the iron (Fe) grade (%) and recovery (%) and ANOVA results, focusing on the p-value and model f-value to confirm that the model could effectively predict responses within the specified variable ranges. Additionally, the model's performance was further validated by analyzing the coefficient of determination ($R^2$) and comparing the adjusted $R^2$ and predicted $R^2$ values to evaluate its predictive capability. The actual and predicted values were computed and compared.

# 4. Results and Discussion

## 4.1 Iron Ore Characterization

Understanding the elemental and mineralogical composition, mineral association within the ore, mineral liberation, and interlocking characteristics of the ore is important in comminution circuit design and beneficiation method selection and optimization. Therefore, the characterization of Dawa-Moyale iron ore samples underwent particle size distribution, mineral phases identification through Ore Petrography Analysis and X-ray diffraction (XRD) analyses, and the assessment of chemical composition before laboratory-scale flotation tests.

### 4.1.1 Particle size analysis

Examining the particle size distribution of geological materials can be used to assess the efficiency of grinding processes on rock samples (51). Particle size analysis was performed on the Dawa-Moyale iron ore through sieving, utilizing a standard series of sieve sizes ranging from 500 to 63 µm. The results of the particle size analysis of 40-minute grinding are presented in Table 4. The particle size distribution curve shown in Figure 9 illustrates the data from 40-minute ball mill grinding, which does not achieve the desired feed particle size range, with at least 80% of particles retained at 150 µm.

Table 4. Particle size analysis of 40-minute ball mill grinding of Dawa-Moyale iron ore.

| Sieve size range (microns) | Aperture size (microns) | Weight retained (g) | % Weight retained | Cumulative wt.% Retained | Cumulative wt.% Passing |
|---|---|---|---|---|---|
| +500 | 500 | 71.5 | 71.5 | 71.50 | 28.50 |
| -500+250 | 250 | 13.9 | 13.9 | 85.40 | 14.60 |
| -250+150 | 150 | 1 | 1 | 86.40 | 13.60 |
| -150+100 | 100 | 5.6 | 5.6 | 92.00 | 8.00 |
| -100+63 | 63 | 7.1 | 7.1 | 99.10 | 0.90 |
| -63 | pan | 0.9 | 0.9 | 100.00 | 0.00 |
| $\Sigma$ | | 100 | 100 | | |

### 4.1.2 Chemical Composition Analysis

An AAS analysis was conducted on representative feed samples collected from the Dawa-Moyale region in southern Ethiopia, examining varying particle sizes at the EGI central laboratories.

The major elements present in the ore expressed in oxide form are summarized in Table 5. The chemical analysis results indicated that the feed contains an average of 37.45 % iron (Fe), along with silicon (Si), aluminium (Al), calcium (Ca), magnesium (Mg), manganese (Mn), titanium (Ti), phosphorous (P) and LOI (loss on ignition) are the major impurity elements. Traces of sodium (Na) and potassium(K) were also detected.

The ore sample contains an average of 51.76 % magnetite ($Fe_3O_4$), constituting the dominant iron ore mineral present in the ore, while titanium, aluminium, and silica represent the main gangue minerals. Table 5 shows that the distribution of iron decreases as the particle size decreases. The maximum iron content is found in the +150 µm size fraction, which contains lesser gangue

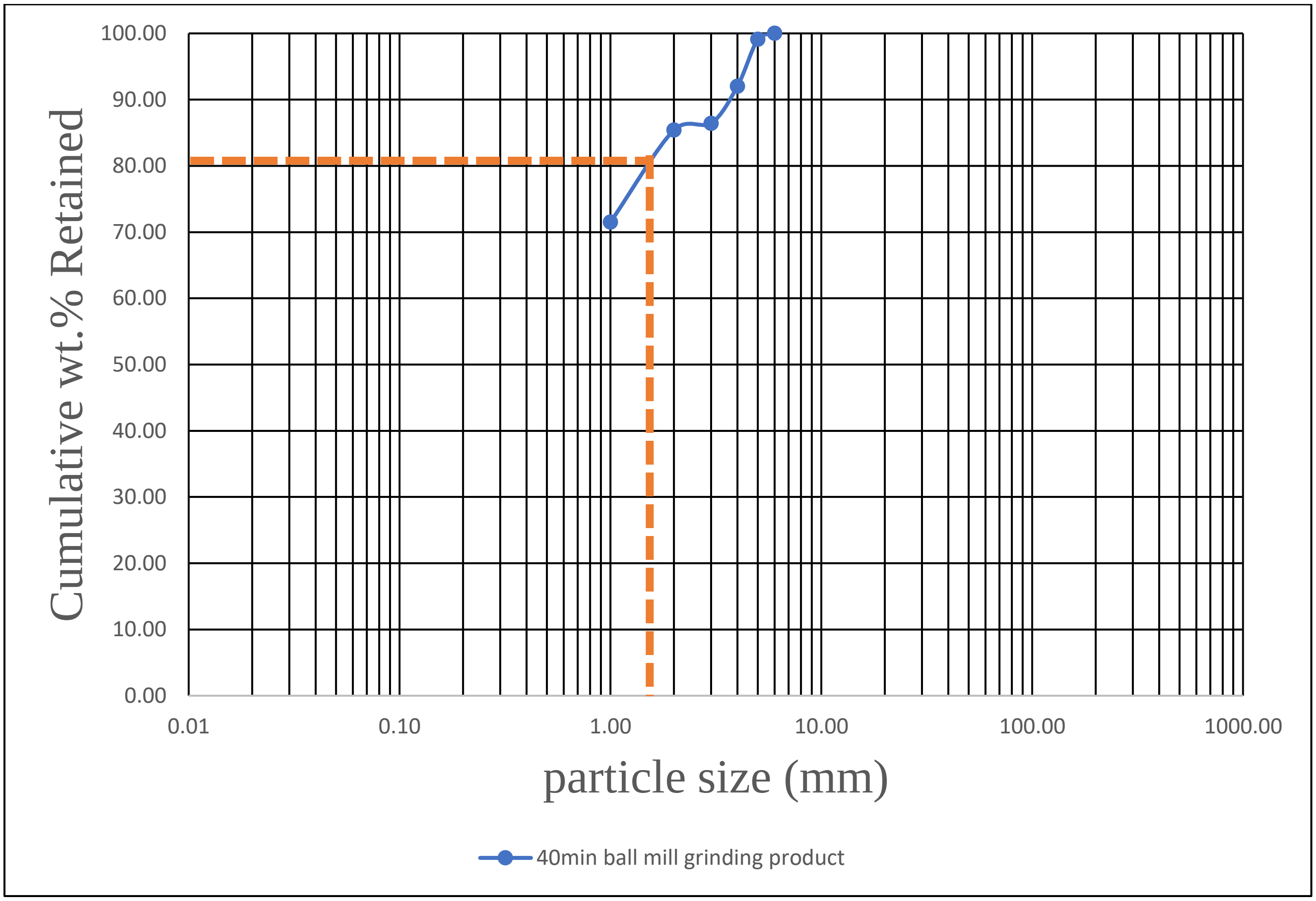



Figure 9. The Particle size distribution curve for Dawa-Moyale iron ore

minerals, while the lowest iron content is found in the -63μm particle size fraction. Based on these results, reverse flotation was conducted on the -150+63μm and -63μm particle size fractions.

Table 5. AAS analysis of different particle size ranges

| Sieve size (μm) | Chemical composition (wt.%) | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_3O_4$ | Fe (Total) | CaO | MgO | $Na_2O$ | $K_2O$ | MnO | $P_2O_5$ | $TiO_2$ | $H_2O$ | LOI |
| -250+150 | 2.70 | 10.12 | 59.36 | 42.95 | 0.54 | 3.20 | 0.40 | <0.01 | 0.58 | <0.01 | 22.46 | 0.43 | <0.01 |
| -150+63 | 3.84 | 13.66 | 54.48 | 39.42 | 0.90 | 4.30 | <0.01 | <0.01 | 0.90 | 0.58 | 22.44 | 0.28 | 0.02 |
| -63 | 4.86 | 15.80 | 41.44 | 29.99 | 8.32 | 5.54 | <0.01 | <0.01 | 0.66 | 0.60 | 17.94 | 0.49 | 3.08 |
| Average | 3.80 | 13.19 | 51.76 | 37.45 | 3.25 | 4.35 | 0.40 | <0.01 | 0.71 | 0.59 | 20.95 | 0.40 | 1.55 |

### 4.1.3 Ore Mineralogy

**Ore Petrography Analysis**

The ore petrography analysis is presented in Table 6. Based on the results, the Dawa-Moyale iron sample is mainly composed of magnetite (55%) and ilmenite (25%), with a minor amount of hematite (3%), pyrite (2%), and other gangue minerals (15%). The magnetite ore exists in granoblastic texture. The significant presence of magnetite suggests that this deposit could be considered a high-grade source of iron ore. Furthermore, the inclusion of ilmenite ($FeTiO_3$) indicates potential for titanium recovery, thereby enhancing the economic value of the deposits.

Table 6. Modal mineralogy (Vol.%) of the Dawa-Moyale iron ore

| Sample ID | Magnetite | Ilmenite | Hematite | Pyrite | Other gangue minerals |
|---|---|---|---|---|---|
| MM-01 | 55 | 25 | 3 | 2 | 15 |

**X-ray diffraction**

The X-ray diffraction analysis of Dawa-Moyale iron ore, illustrated in Figure 10, indicates the presence of magnetite, hematite, ilmenite, bornite, quartz, calcite, and chromite as the primary mineral phases present in the iron ore. Besides the presence of magnetite and hematite as the iron bearing minerals, the presence of ilmenite, bornite, and chromium suggests the potential for valuable by-products, including ilmenite, bornite and chromite, which are the mineral ores of titanium, copper and chromium, respectively.

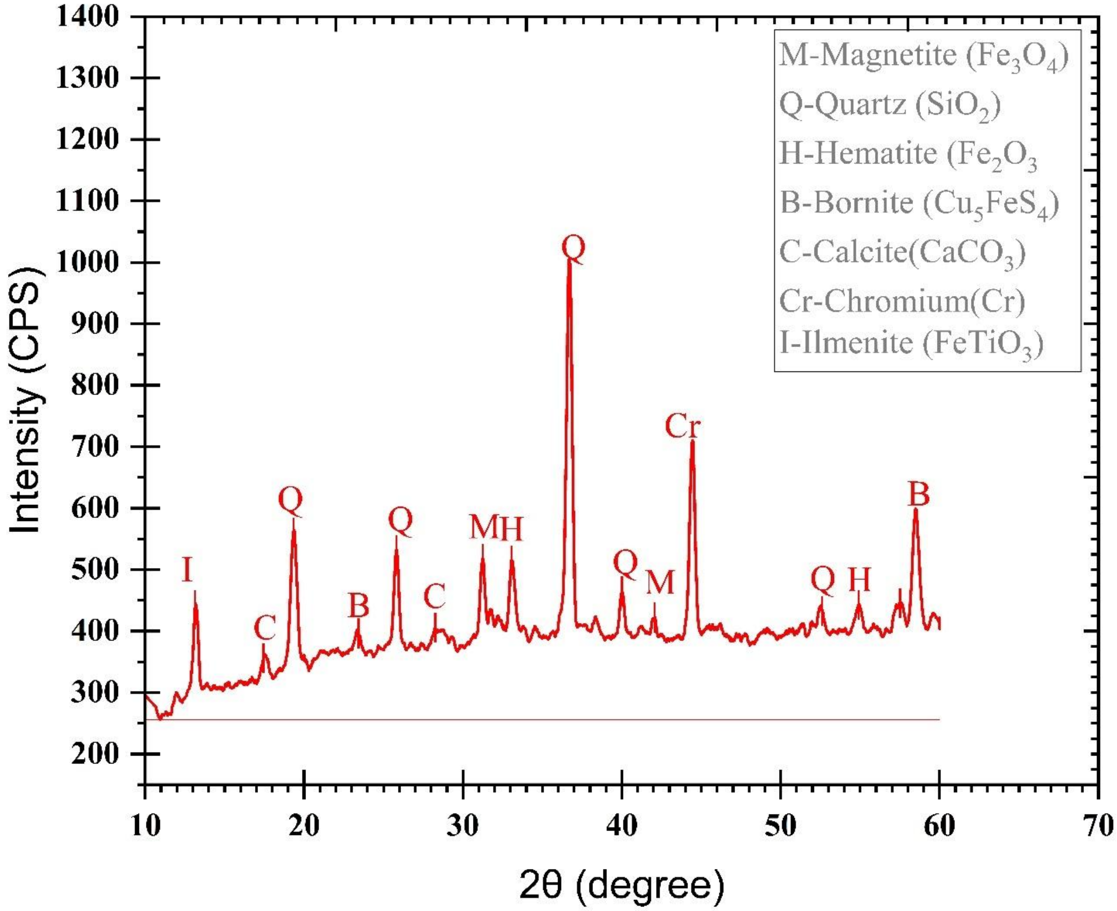



Figure 10. X-ray diffraction patterns showing the main phases present in Dawa-Moyale iron ore.

## 4.2 Reverse Flotation Tests

The results of two-level, full factorial-designed experiments are presented in Table 7. This study examined the effect of feed particle size, collector dosage, and pH values or their interactions on iron grade and recovery. A half-normal plot was utilized to identify significant effects, and ANOVA analysis was conducted on the experimental results to assess the significance of the model.

Table 7. Experimental data showing observed and predicted values with coded variables.

| Experimental No | Feed particle size (µm) | Collector Dosage (g/t) | pH | Concentrate yield % | Fe grade (%) | | Fe Recovery (%) | |
|---|---|---|---|---|---|---|---|---|
| | A | B | C | Y | Observed | Predicted | Observed | Predicted |
| 1 | -63 | 125.0 | 11.5 | 49.0 | 38.3 | 38.1 | 60.2 | 65.2 |
| 2 | -63 | 83.0 | 9.5 | 65.4 | 41.0 | 41.5 | 64.7 | 65.2 |
| 3 | -150+63 | 83.0 | 11.5 | 84.6 | 33.3 | 33.9 | 94.2 | 93.9 |
| 4 | -150+63 | 83.0 | 9.5 | 95.0 | 37.6 | 36.5 | 94.7 | 93.9 |
| 5 | -150+63 | 125.0 | 11.5 | 87.9 | 43.5 | 43.1 | 93.4 | 93.9 |
| 6 | -63 | 125.0 | 9.5 | 71.0 | 39.8 | 40.7 | 66.7 | 65.2 |
| 7 | -150+63 | 125.0 | 9.5 | 87.8 | 45.9 | 45.7 | 93.2 | 93.9 |
| 8 | -63 | 83.0 | 11.5 | 58.6 | 38.8 | 38.9 | 65.7 | 65.2 |

### 4.2.1 Adequacy of the Model

4.2.1.1 Iron (Fe) Grade

The half-normal plot displayed in Figure 11 reveals that the factors B (collector dosage), C (pH value), and AB (the interaction between particle size and collector dosage) significantly influence the Fe-grade. Among these factors, B and AB positively affect the response, whereas factor C negatively impacts the iron grade.

The analysis also conducted a Shapiro-Wilk test to assess the normality of the unselected terms on the effects plot. Ideally, after the selection of statistically significant terms is finished, the Shapiro-Wilk p-value should exceed 0.10, suggesting that the unselected terms are normally distributed (52). The Shapiro-Wilk p-value of 0.108 was obtained and presented in Figure 11, which provides support for the selection of effects. Figure 12 presents a Pareto chart that depicts the t-values associated with the effects of various factors and their interactions on the Fe-grade. It highlights the comparative significance of these factors and interactions in influencing the Fe grade.

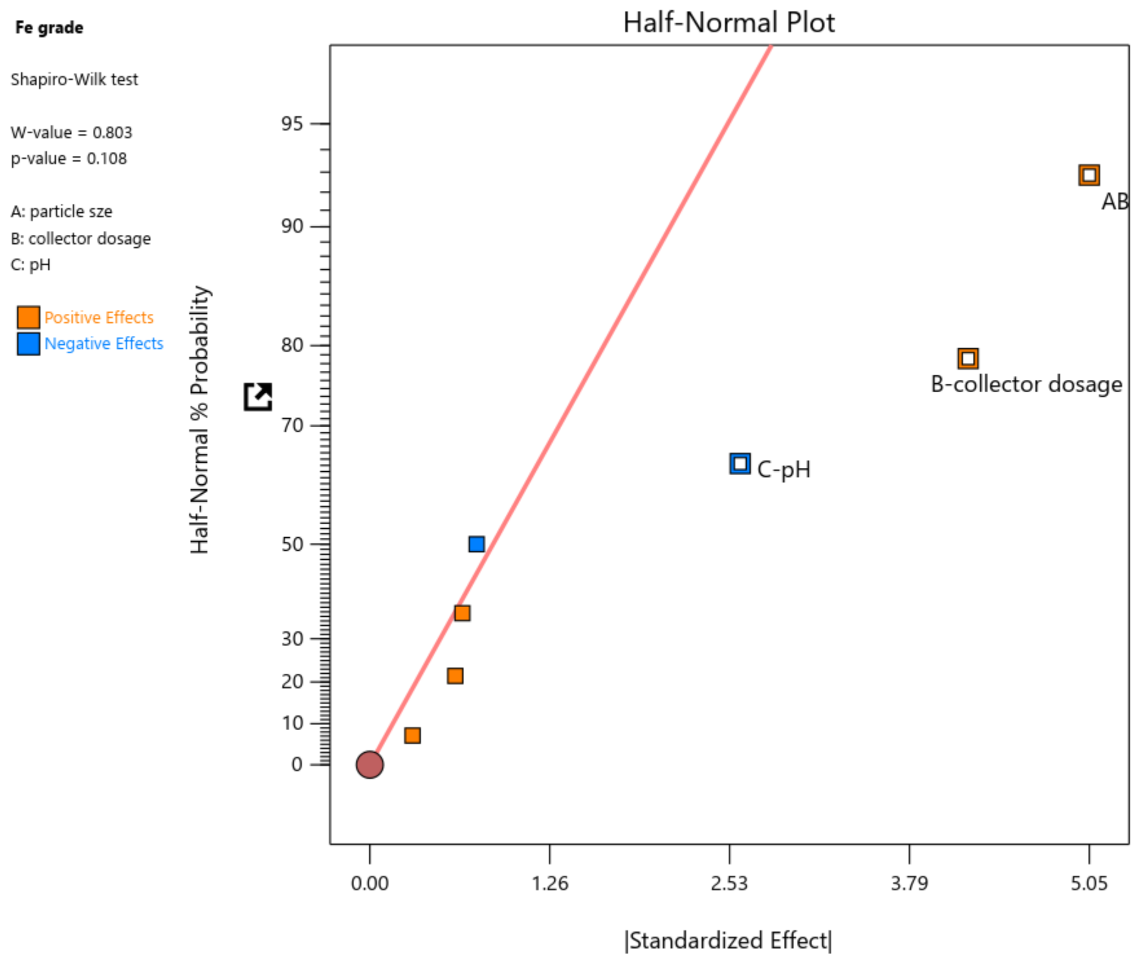



Figure 11. A Half-normal plot for Fe grade, showing significant factors and interactions.

The terms AB (interaction of particle size and collector dosage) and B (collector dosage) exceed the Bonferroni limit, clearly indicating their significance for the Fe grade. Additionally, the term C, representing the pH value, was identified as a significant factor even though its t-value falls below the Bonferroni limit.

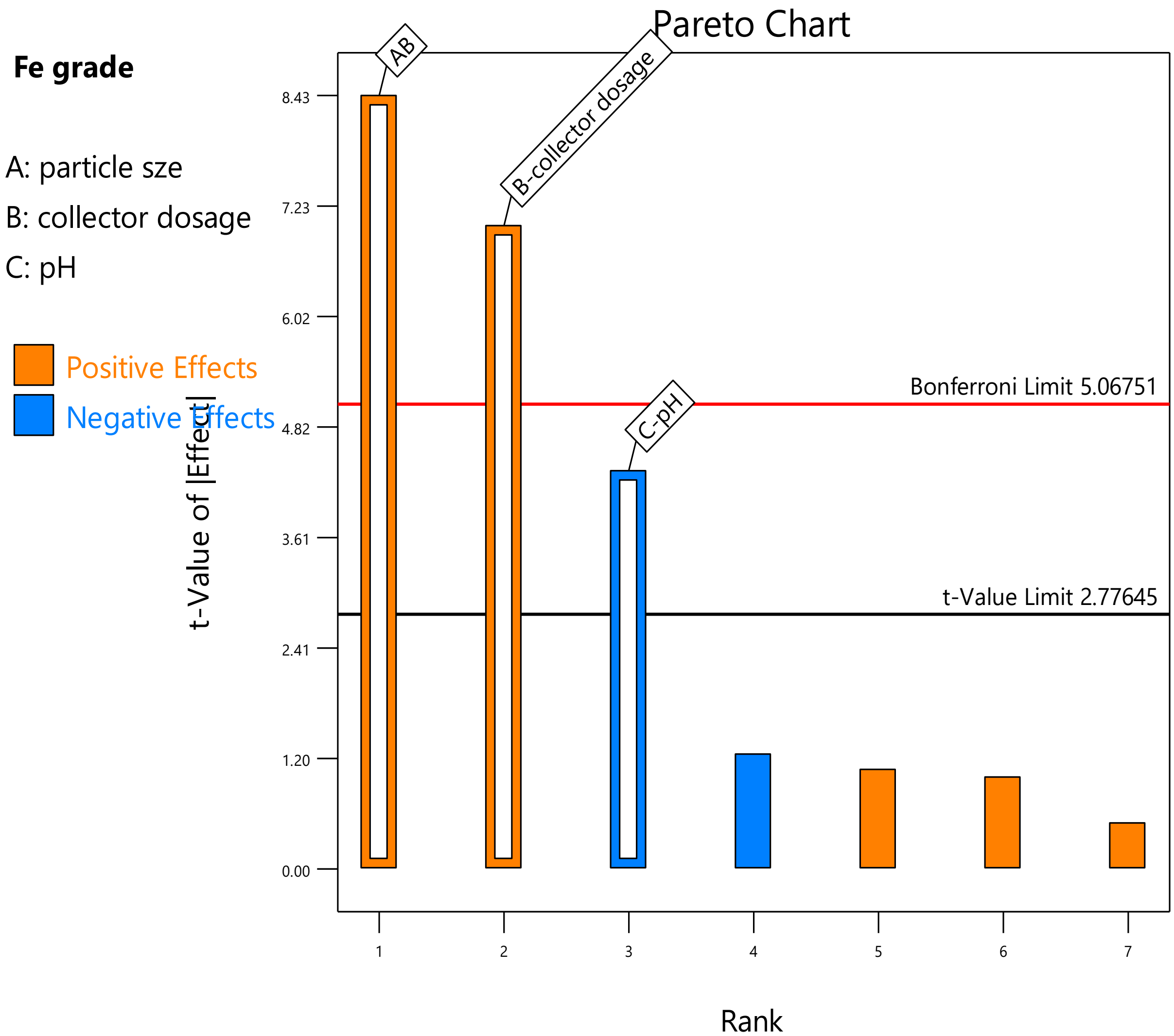



Figure 12. Pareto chart for Fe grade showing t-values of different effects and interactions

The adequacy of the model for Fe grade was further checked by analysis of variance (ANOVA) as summarized in Table 8. The model F-value of 46.37 indicates that the model is significant at a confidence level of 95%, with only a 0.15% probability that a "model F-value" of this magnitude could result from random noise. P-values less than 0.05 suggest that certain model terms are statistically significant; In this case, the terms B, C, and AB are identified as significant. Values greater than 0.1 indicate that the model terms are non-significant. The findings indicate that the Fe grade depends more on the interaction between particle size and the collector dosage.

Table 8. Analysis of variance (ANOVA) for Fe grade

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| **Model** | 99.81 | 3 | 33.27 | 46.37 | 0.0015 | significant |
| **B-collector dosage** | 35.28 | 1 | 35.28 | 49.17 | 0.0022 | |
| **C-pH** | 13.52 | 1 | 13.52 | 18.84 | 0.0122 | |
| **AB** | 51.01 | 1 | 51.01 | 71.09 | 0.0011 | |
| **Residual** | 2.87 | 4 | 0.7175 | | | |
| **Cor Total** | 102.68 | 7 | | | | |

The model summary statistics presented in Table 9 indicate that the determination coefficient ($R^2$) value for the Fe grade is 0.972, demonstrating the significance of the model. Additionally, the predicted $R^2$ of 0.888 is in reasonable alignment with the adjusted $R^2$ of 0.951. The adequate precision, which measures the signal-to-noise ratio, should be greater than 4. For the Fe grade model, the Adequate Precision was calculated to be 19.784, indicating a sufficient signal. The lower coefficient of variation (C.V. %) for the response at 2.130 also validates the reproductivity of the model.

Table 9. Results of model summary statistics for Fe grade

| | | | |
|---|---|---|---|
| **Std. Dev.** | 0.847 | **$R^2$** | 0.972 |
| **Mean** | 39.770 | **Adjusted $R^2$** | 0.951 |
| **C.V. %** | 2.130 | **Predicted $R^2$** | 0.888 |
| **Press** | 11.480 | **Adeq Precision** | 19.784 |

The model equation of iron (Fe) grade (%) response is presented as follows:

$$Fe\ grade\ (\%) = 43.025 + O.1B - 1.3C + 0.002AB \qquad \text{Eq. (4)}$$

Where B is the collector dosage, C is the pH value, and AB is the interaction between particle size and collector dosage. Figure 13 presents the predicted versus actual Fe grade values, indicating a strong correlation between the experimental and predicted data. The study predicted a maximum Fe grade of 46%, with the flotation conditions determined as follows: a particle size of -150+63µm, a collector dosage of 125g/t, and a pH of 9.5.

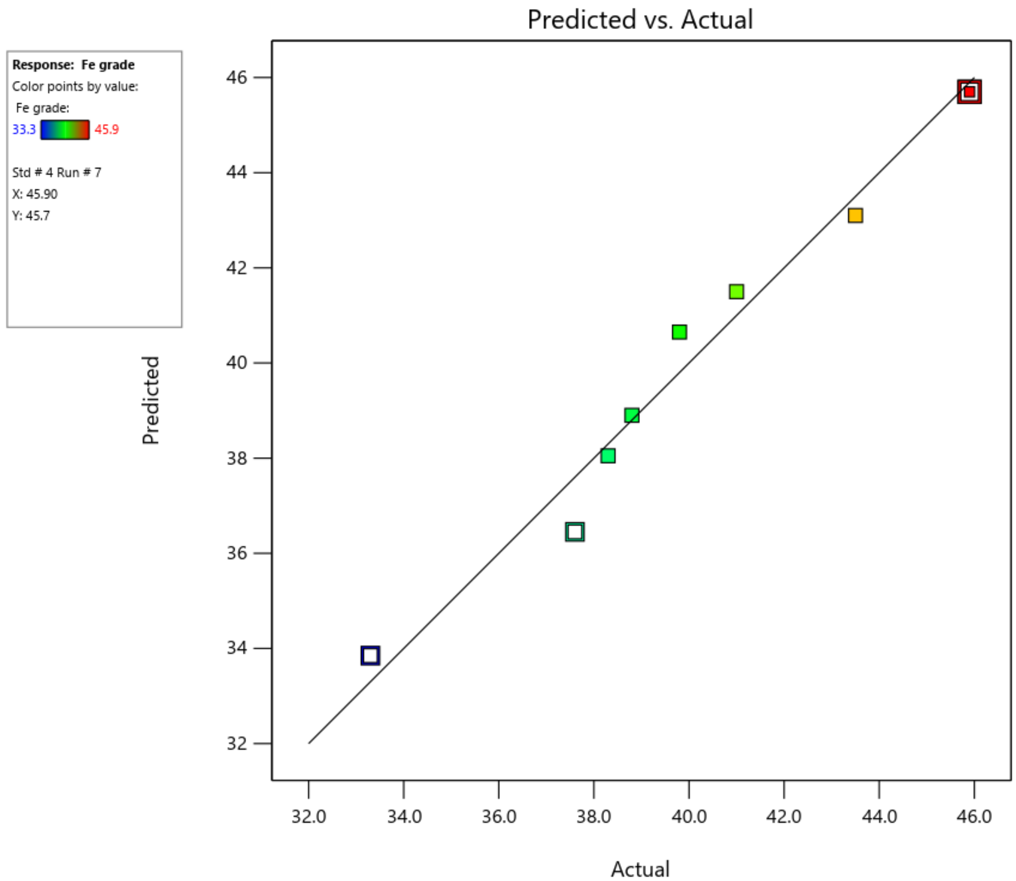



Figure 13. Relationship between actual and predicted values of Fe grade

4.2.1.2 Iron (Fe) Recovery

A half-normal plot was similarly utilized to identify the significant factors affecting iron (Fe) recovery. Figure 14a shows the half-normal plot for the response of iron (Fe) recovery (%). The term *A*, representing the particle size of the feed, appears in the upper right-hand section of the graph, indicating it has the largest absolute values and is thus the most significant factor, where it has a positive effect on the response. A Shapiro-Wilk p-value of 0.198 was obtained, supporting the normality distribution of the unselected terms on the effect plot. The Pareto chart shown in Figure 14b indicates that term A (particle size of the feed) exceeds the Bonferroni limit, underscoring its significance for iron (Fe) recovery (%).

An analysis of variance (ANOVA) for iron (Fe) recovery (%) was conducted to evaluate the adequacy of the model, as shown in Table 10. The model F-value of 1592.37 implies the model is significant at a confidence level of 95%. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.05 indicate model terms are significant. In this case, the term A-particle size is a significant model term. The lower P-values (< 0.0001) show that the variable plays a paramount role in influencing the response. The result indicates that the iron (Fe) recovery depends more on the particle size of the ore feed.

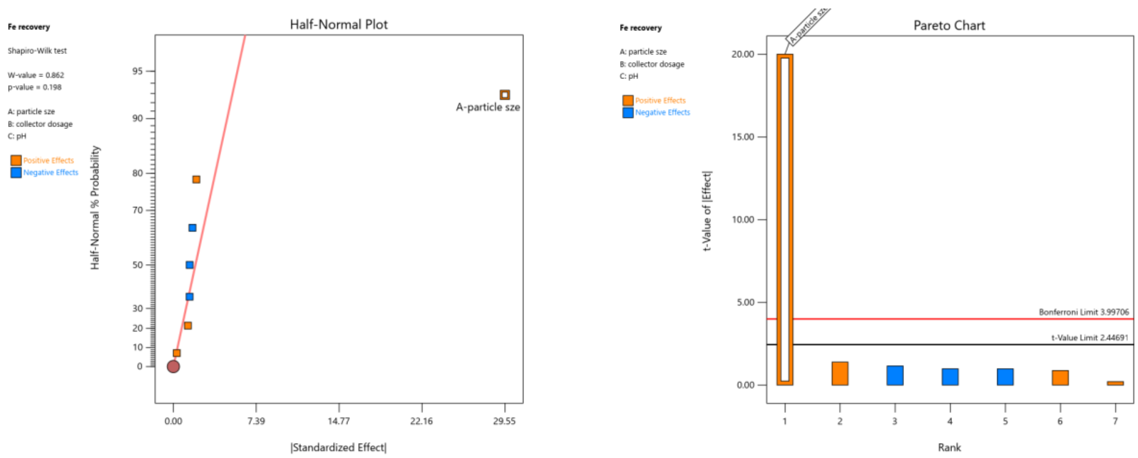



Figure 14. Significant effects of flotation variables on the iron (Fe) recovery (%). (a) A half-normal plot for iron (Fe) recovery, showing significant effects. (b) A Pareto chart for iron (Fe) recovery showing t-values of different effects.

Table 10. Analysis of variance (ANOVA) for Fe recovery (%)

| Source | Sum of Squares | df | Mean Square | F-value | p-value | |
|---|---|---|---|---|---|---|
| **Model** | 1641.36 | 1 | 1641.36 | 1592.37 | < 0.0001 | Significant |
| **A-particle size** | 1641.36 | 1 | 1641.36 | 1592.37 | < 0.0001 | |
| **Residual** | 6.18 | 6 | 1.03 | | | |
| **Cor Total** | 1647.54 | 7 | | | | |

The fit summary statistics for the model shown in Table 11 reveal that the R-squared ($R^2$) value for the iron recovery (%) is 0.9962, underscoring the significance of the model. Furthermore, the predicted $R^2$ of 0.9933 strongly aligns with the adjusted $R^2$ of 0.9956. The adequacy precision was determined to be 56.4336, indicating a strong signal. The model equation of iron (Fe) recovery (%) response is presented as follows:

$$Fe\ recovery\ (\%) = 79.10 + 0.237A \qquad \text{Eq. (5)}$$

Where A is the particle size of the ore feed. The current study predicted a maximum Fe recovery of 94.7%, with the flotation conditions determined as follows: a particle size of -150+63µm, a collector dosage of 83g/t, and a pH of 9.5.

Table 11. Results of model summary statistics for Fe recovery

| | | | |
|---|---|---|---|
| **Std. Dev.** | 1.02 | **$R^2$** | 0.9962 |
| **Mean** | 79.55 | **Adjusted $R^2$** | 0.9956 |
| **C.V. %** | 1.28 | **Predicted $R^2$** | 0.9933 |
| **Press** | 10.99 | **Adeq Precision** | 56.4336 |

### 4.2.2 Effect of Flotation Variables on Concentrate Iron (Fe) Grade (%)

The study identified collector dosage, pH values and the interaction between particle size and collector dosage as significant effects influencing the Fe grade in flotation processes. Figure 15 presents a two-dimensional one-factor analysis of Fe grade, with collector dosage represented on the x-axis and the Fe grade (%) on the y-axis. In Figure 15a, factors A (particle size) and C (pH value) are held constant at 63µm and 11.5, respectively, and the result indicates that the Fe grade (%) increases with an increase in collector dosage. However, when the pH value is decreased from 11.5 to 9.5 while maintaining a constant particle size, as shown in Figure 15b, the result indicates the same trend as observed in Figure 15a. Additionally, when both actual factors, A (particle size) and C (pH value), are decreased from +63µm to -63µm and 11.5 to 9.5, respectively, as shown in Figure 15c, the results indicate an opposite trend to that of Figure 15a.

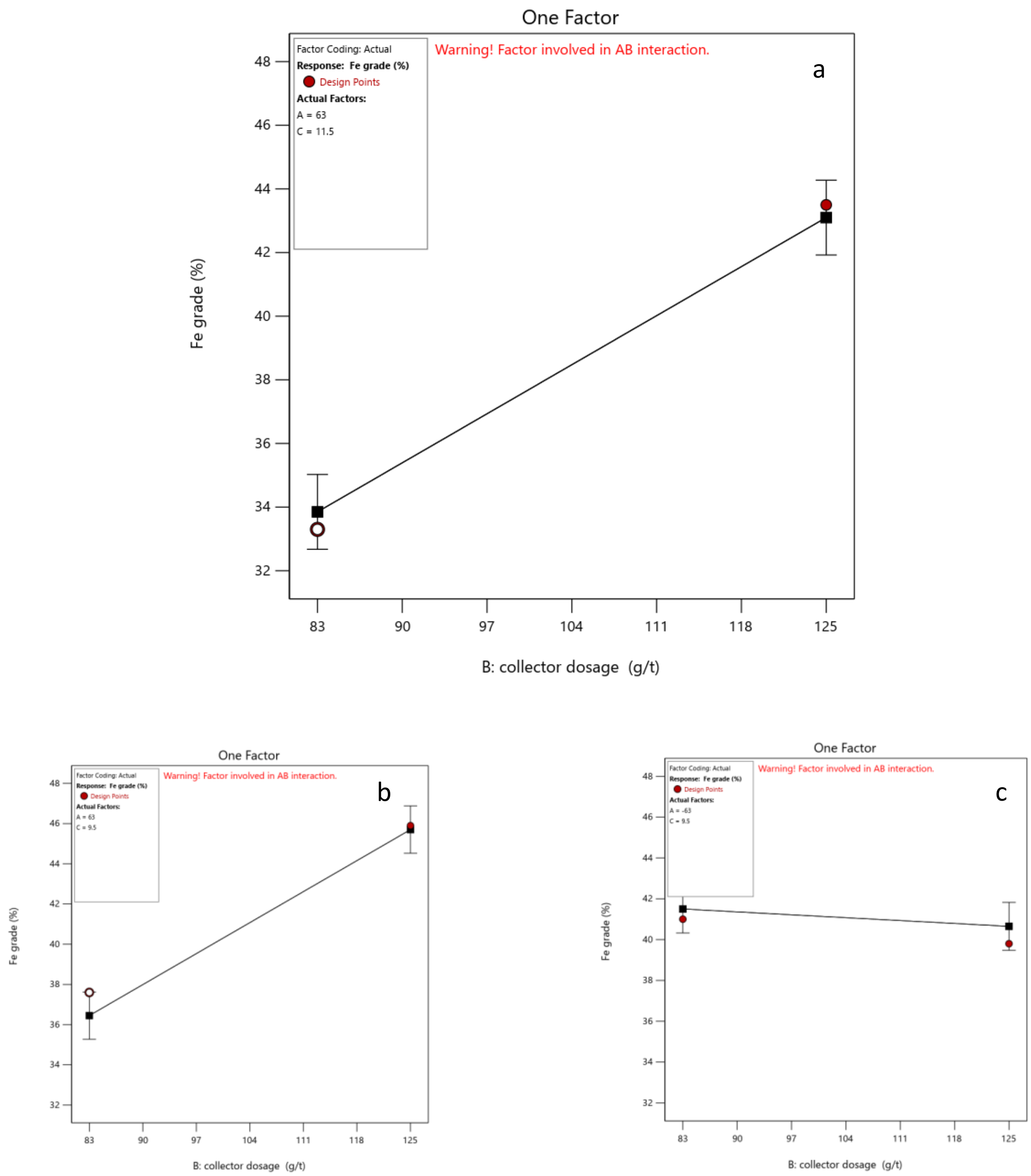



Figure 15. (a) Effect of collector dosage on Fe grade (%) at a higher level of particle size (+ 63µm) and a higher level of pH 11.5. (b) Effect of collector dosage on Fe grade (%) at a higher particle size (+63µm) and a lower level of pH 9.5. (c) Effect of collector dosage on Fe grade (%) at a lower level of particle size (-63µm) and a lower level of pH 9.5.

The second significant factor influencing the Fe grade is the pH value. As shown in Figure 16, the x-axis represents the C(pH) factor, and the y-axis represents the Fe grade response, where A (particle size) and B (collector dosage) are the actual factors which remain constant. The figure indicates that the Fe grade (%) decreases with an increase in pH level and increases as the pH level decreases.

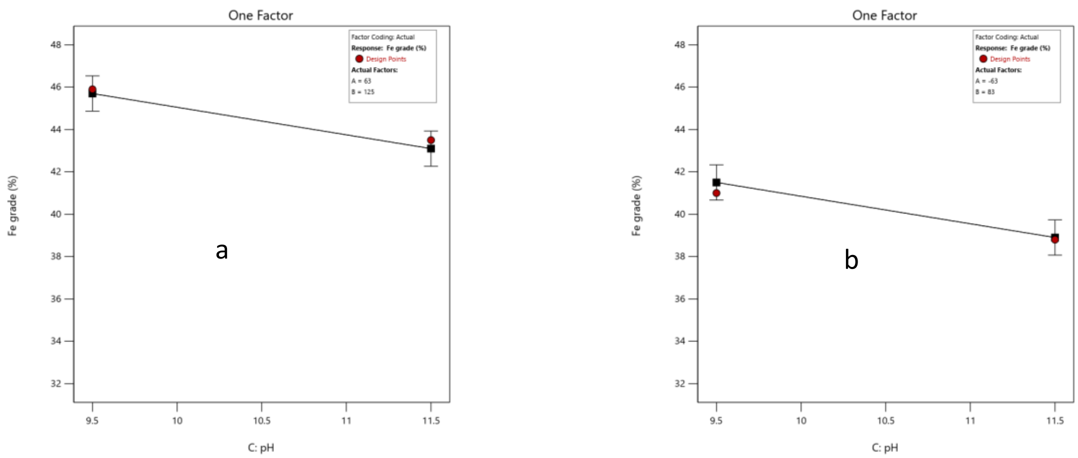



Figure 16. (a) Effect of pH on the Fe grade (%) at a high particle size (+ 63µm) and a high collector dosage (125g/t). (b) Effect of pH on the Fe grade (%) at a low particle size (-63µm) and a low collector dosage (83g/t).

Figure 17 depicts the interaction between collector dosage and particle size, illustrating that the effect of collector dosage varies with particle size. The figure indicates that no interaction effect occurs at smaller particle sizes, while a significant effect is observed at larger particle sizes. The optimal iron (Fe) grade (%) is achieved at a particle size and collector dosage of +63µm and 125g/t, respectively, with an actual factor of C(pH) at 9.5.

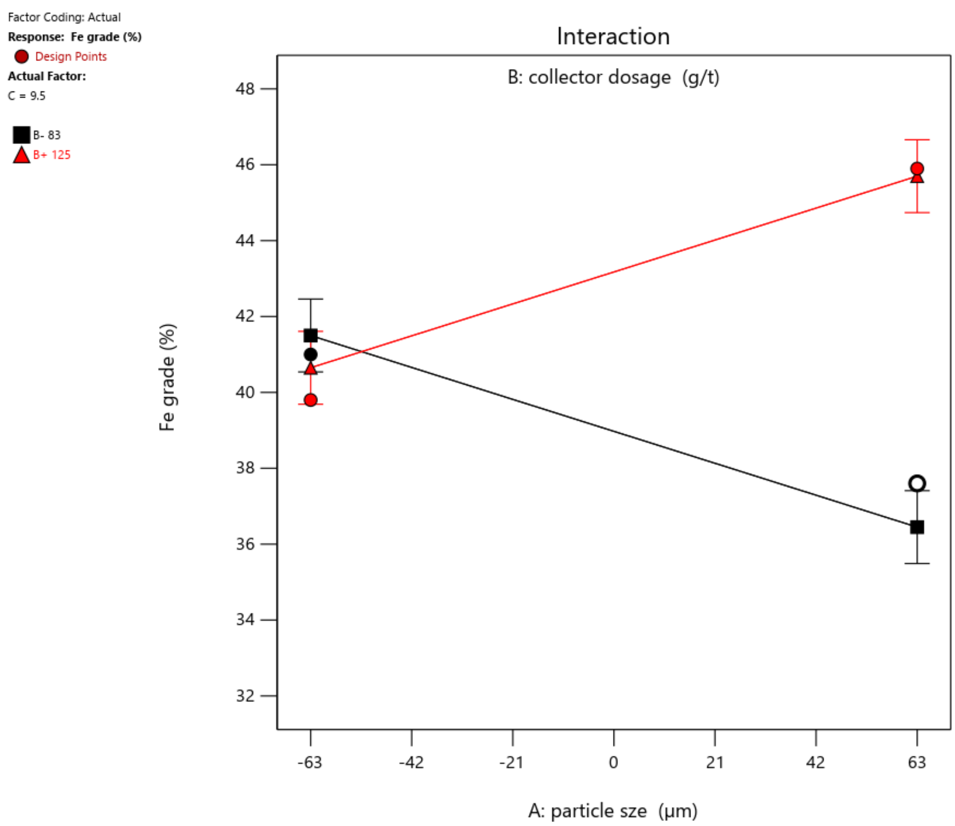



Figure 17. Interaction effects between particle size and collector dosage on iron (Fe) grade (%)

To gain deeper insights into the interaction effects of these variables, a three-dimensional response surface plot is illustrated in Figure 18. This plot features the iron (Fe) grade (%) on the y-axis, with particle size and collector dosage represented as $X_1$ and $X_2$ axes, respectively. Additionally, the pH value has been taken as the actual factor in Figure 18. It can be observed that a high iron (Fe) grade (%) can be achieved at high levels of particle size and collector dosage.

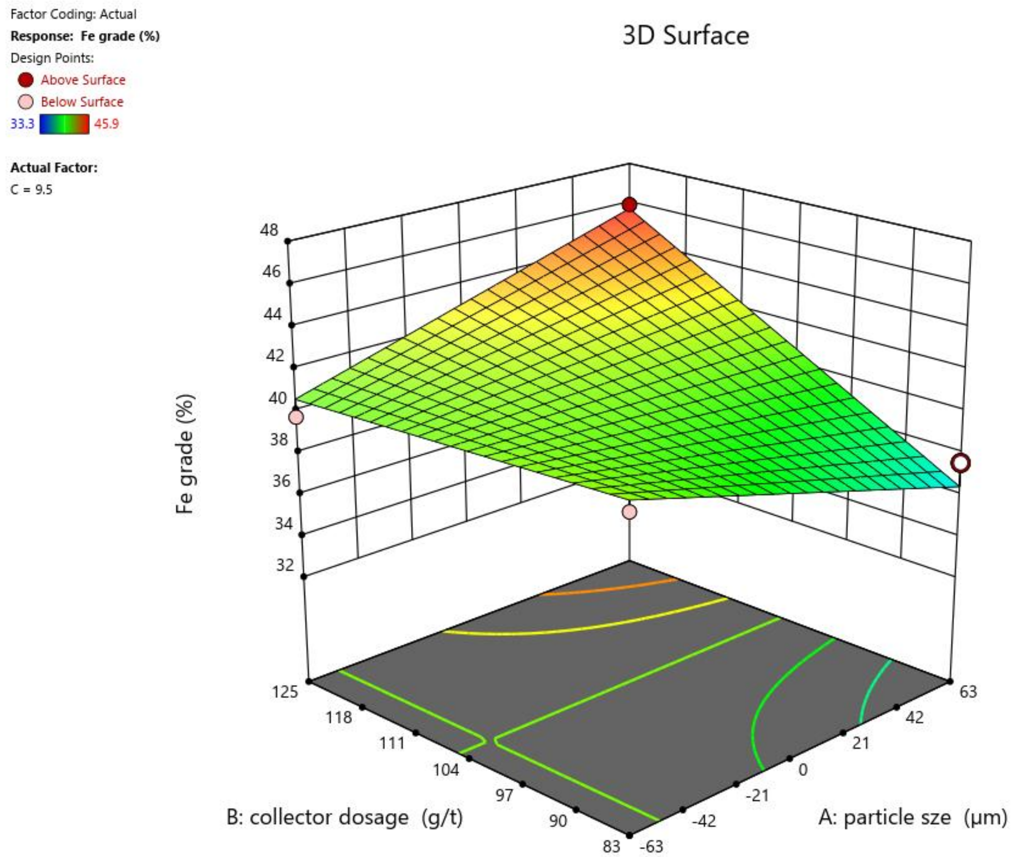


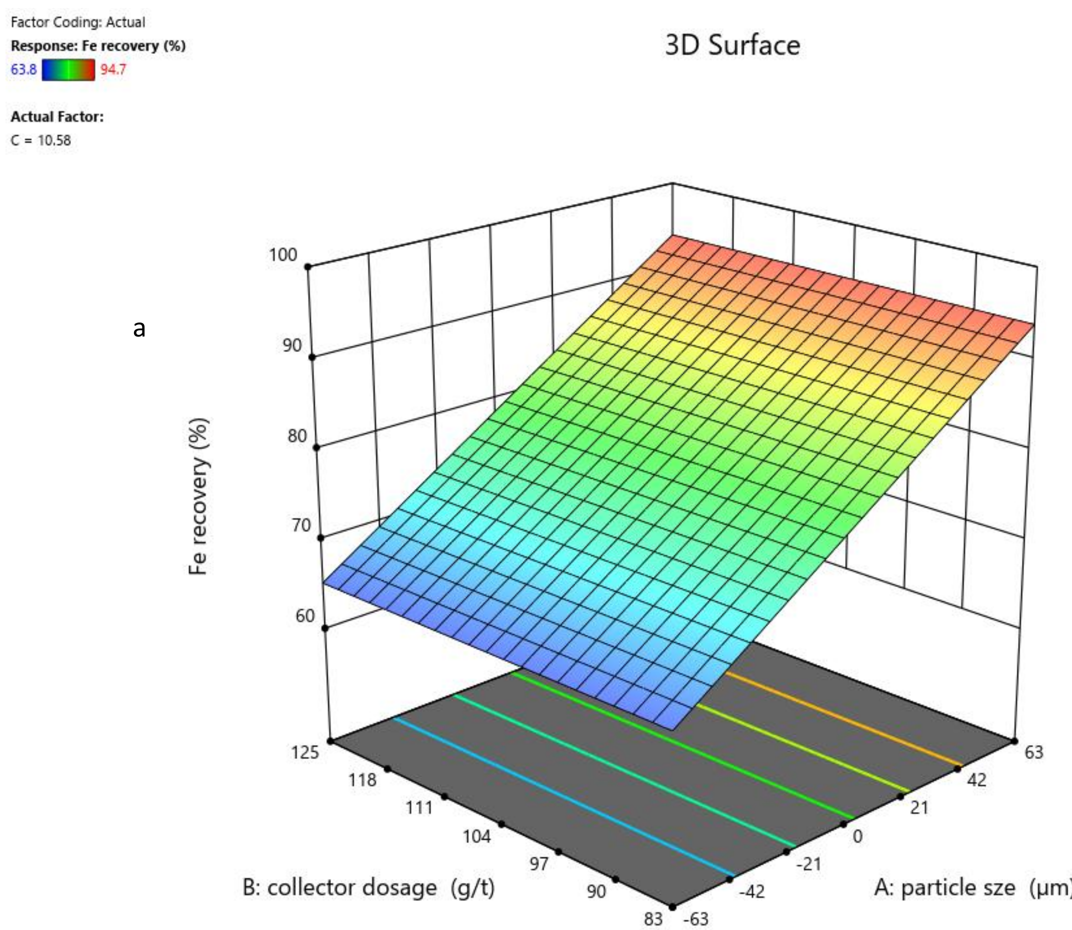

Figure 18. Three-dimensional diagram showing the effect of collector dosage and particle size on iron (Fe) grade (%), with pH value at the central level.

### 4.2.3 Effect of Flotation Variables on Iron (Fe) Recovery (%)

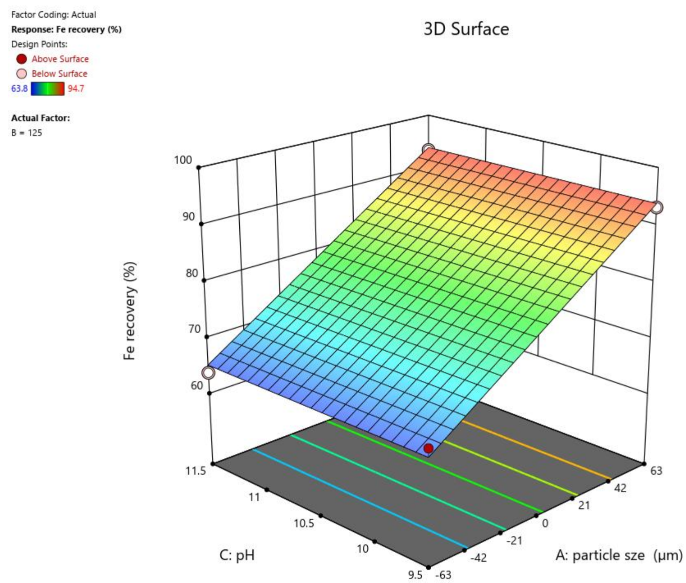
The 3D plot of the response surface was used to examine the relation between experimental factors and the iron (Fe) recovery (%) response. Figure 19a shows the effect of collector dosage and particle size on the iron recovery (%). It can be seen that iron recovery increases with larger particle sizes of the ore feed, while variations in collector dosage do not seem to influence iron recovery (%). With the pH maintained at the central level (10.58), optimal iron recovery (%) is achieved at higher particle sizes, indicating that the iron recovery is predominantly dependent on the particle size of the ore feed.

Figure 19b illustrates the effects of particle size and the pH value on iron recovery, with collector dosage held at the central level of 125g/t. Here, the percentage of iron recovery rises as the particle size increases, while changes in pH value do not influence the iron recovery. Consequently, iron recovery is significantly influenced by the particle size of the ore feed.

Figure 19c shows the effect of collector dosage and the pH value, with particle size at the central level on the iron recovery (%). The results indicate that varying these two parameters does not yield any considerable effects on the percentage of iron recovery. Therefore, neither collector dosage nor pH values significantly impact the iron recovery in the current study. Overall, the analysis of the effect of these experimental factors on iron recovery (%) reveals that there are no interaction effects, while the particle size of the ore feed plays a crucial role in determining the iron (Fe) recovery (%) in this study.



b




c

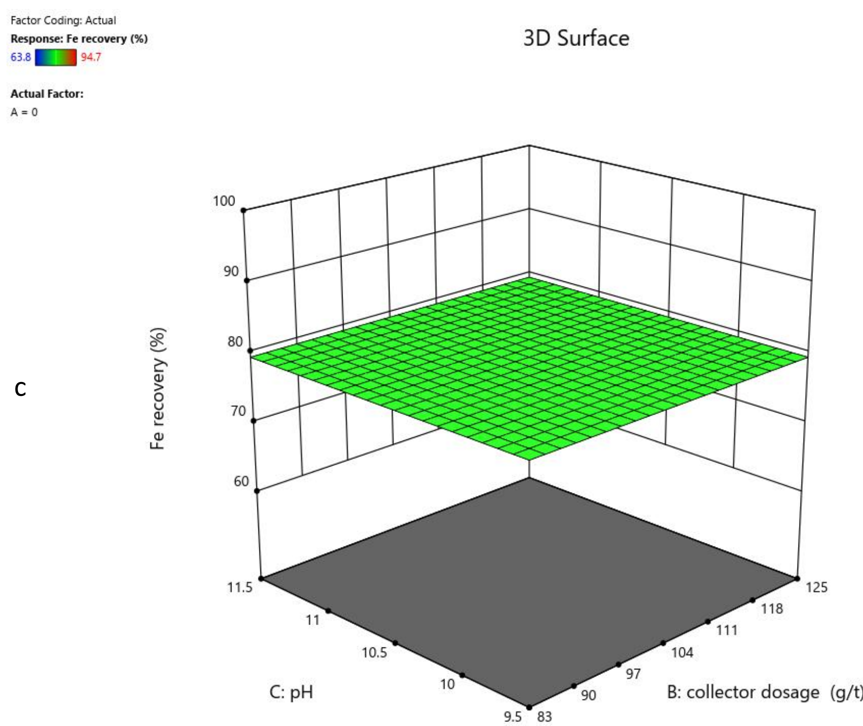



Figure 19. Three-dimensional diagram showing the effect of (a) collector dosage and particle size on iron (Fe) recovery (%), with pH value at the central level; (b) particle size and pH value, with collector dosage at the central level; and (c) collector dosage and pH value with particle size at the central level on iron (Fe) recovery (%).

# 5. Conclusions and Recommendations

## 5.1 Conclusions

Reverse flotation has proven an effective method for upgrading iron ores, particularly for fine-grained ores where conventional gravity and magnetic separation techniques may be less efficient. Factors influencing flotation performance include particle size, pH, and reagent dosage. According to the Literature, the optimal particle size is typically below 150 μm, with pH levels ranging from 8.5 to 11.5 for anionic collectors, and from 11.5 to 12 for cationic collectors. Additionally, careful optimization of collector and depressant dosages is essential. These parameters formed the basis for the experimental design of this study.

This study employed ore characterization and reverse flotation experiments on Dawa-Moyale iron ore. The process of reverse flotation experiments involved the sequential removal of undesirable materials and the depression of iron-bearing minerals to enhance the quality of Dawa-Moyale iron ore, located in the Dawa zone of the Somali region in Ethiopia. The ore characterization of the Dawa-Moyale iron deposit revealed important insights into the mineralogical and chemical composition. X-ray diffraction and Bulk model analyses identified magnetite as the dominant iron-bearing mineral. Comprising approximately 55% of the ore, with a minor amount of hematite and ilmenite also present. Chemical analysis showed an average iron content of 37.45%, along with silica, alumina, and titanium as the main gangue components. Particle size analysis indicated that 40 minutes of ball mill grinding was insufficient to achieve the desired feed size distribution for flotation. The presence of valuable by-products like ilmenite and chromite was also noted.

Iron ore particles from the Dawa-Moyale region can be recovered by manipulating the flotation parameters, including the particle size of the ore feed, collector dosage, and pH levels. Analysis of Experimental results using half-normal plots and Pareto charts revealed that collector dosage and pH levels are pivotal in determining the iron grade (%), while particle size is the most significant factor affecting iron (Fe) recovery (%). Additionally, interactions between particle size and collector dosage were found to have substantial impacts on the Fe grade response. However, it is important that no interaction effects among the various experimental variables were observed for the Fe recovery response.

The equations modeling iron grade and recovery were determined to be significant with a high confidence level. The comparison between the experimental results and the predictions from the model equations showed satisfactory agreement. Under the optimal conditions, identified by the model, it was predicted that an iron grade of 46% with an iron recovery of 93.2% could be achieved. Consequently, this study concludes that a particle size of -150+63 µm exerts a significant effect on both iron grade and recovery, while the use of oleic acid at 125g/t as a collector has a statistically significant effect on the iron (Fe) grade (%).

## 5.2 Recommendations

Based on the investigations conducted in this project, several recommendations for future research can be proposed.

- In the extension of ore characterization, such as conducting a liberation study to assess the degree of liberation of iron minerals utilizing point counting and MLA techniques and performing quantitative mineralogical analysis using the QEMSCAN technique may be employed. This advanced mineralogical investigation facilitates the examination of varying processing performances, particularly in terms of grinding and flotation efficiency.
- Performing further tests on depressant types and dosages is required to identify the conditions under which particle size, collector dosage, and pH values are most effective.
- As the current study employed a two-level full factorial design, the future and interested researchers should conduct response surface methodology (RSM), which is the best statistical technique for the optimization of the iron grade and recovery.
- In light of the development of the Dawa-Moyale iron ore, it is imperative for policymakers and stakeholders to give special attention to strategies that will attract and facilitate potential investors in domestic business activities. This proactive engagement is essential to optimize the economic benefits of the resource and ensure sustainable development within the country.

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# Appendices

## Appendix A- Report for Ore Petrography Analysis

**Geological Institute of Ethiopia**
**Mineralogy and Geotechnical Laboratory Research**
**Desk Result Form**

Case Team:- Mineralogical: Lab section:- Mineralogy ✓ Physical ☐

Client /Originator Name:- Abdifatah Maktal

Client Category: - Survey ☐ Gov. ☐ Pvt. ✓ Date Submitted:- 22/01/2025

File name:- 230/2025 PVT Area Ref:- Moyale No of Samples:- 1 Sample No:- MM-01

Sample Type: - Ore/ Rock Lab No:- 230/2025 Type of Analysis:- Ore petrography

Preparation required:- Polished section

I) **Hand specimen Description:-**

II) **Mineral composition**

| Mineral | Modal (%) | Texture |
|---|---|---|
| Magnetite | 55 | Xenoblastic |
| Ilmenite | 25 | Xenoblastic |
| Gangue | 15 | - |
| Hematite | 3 | Xenoblastic |
| Pyrite | 2 | Xenoblastic |

**III) Textural Descriptions / Notes:** Granoblastic Texture

Magnetite and Ilmenite grains are dominantly visible on the section.

**Described By / Analysts** Girma Asemu & Asamnew Besufikad

**Checked by** Abdu Ebrahim

**Date completed:-** 14/02/2025

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# Appendix B- Report for Complete Silicate Analysis

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

Customer Name;- Abdifata Maktel Abdulahi /Addis Abeba University

Issue Date:-06/02/2025

Sample type ;- Powder

Request No:- GLD/RQ/872/25

Sample Preparation:-200 Mesh

Report No:- GLD/RN/4390/25

Date Submitted ; 22/01/2025

Number of Sample:- Fiftyn(15)

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

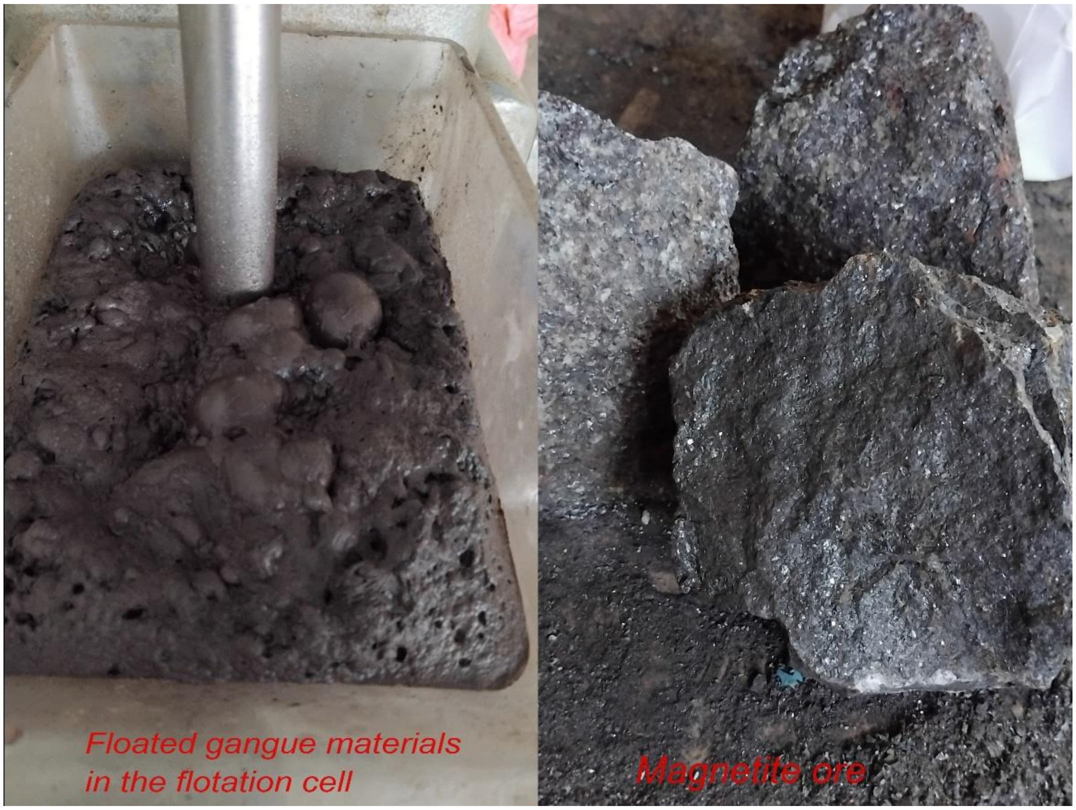
Analytical Method: $LiBO_2$ FUSION, HF attack, GRAVIMETERIC, COLORIMETRIC and AAS

| 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 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| BT-001 | 3.02 | 8.24 | 56.70 | 0.90 | 3.86 | 4.72 | < 0.01 | 0.88 | 0.58 | 19.50 | 0.41 | <0.01 | 200gm |
| BT-002 | 3.70 | 10.66 | 53.68 | 0.80 | 4.44 | 5.04 | < 0.01 | 0.80 | 0.58 | 19.84 | 0.59 | 1.08 | 210gm |
| BT-003 | 4.12 | 15.32 | 49.22 | 1.10 | 6.08 | 1.68 | < 0.01 | 0.76 | 0.54 | 19.72 | 0.51 | 1.11 | 250gm |
| BT-004 | 4.22 | 11.40 | 52.00 | 1.00 | 7.44 | <0.01 | < 0.01 | 0.76 | 0.52 | 19.30 | 0.45 | 1.61 | 110gm |
| BT-005 | 4.22 | 11.10 | 52.90 | 1.12 | 3.88 | <0.01 | < 0.01 | 0.92 | <0.01 | 24.28 | 0.09 | <0.01 | 215gm |
| BT-006 | 3.80 | 13.38 | 56.40 | 1.12 | 4.50 | <0.01 | < 0.01 | 0.88 | 0.60 | 19.36 | 0.45 | <0.01 | 220gm |
| BT-007 | 3.42 | 8.00 | 54.96 | 0.98 | 3.40 | 5.60 | < 0.01 | 0.70 | 0.58 | 22.25 | 0.42 | <0.01 | 225gm |
| BT-008 | 4.14 | 16.72 | 46.08 | 1.06 | 5.00 | 0.80 | 1.76 | 0.78 | 0.50 | 19.48 | 0.63 | 1.71 | 225gm |
| BT-009 | 2.82 | 8.50 | 61.96 | 0.58 | 3.96 | 5.84 | < 0.01 | 0.68 | 0.58 | 15.54 | 0.50 | <0.01 | 200gm |
| BT-010 | 3.00 | 11.70 | 63.42 | 0.58 | 3.84 | 0.80 | < 0.01 | 0.66 | 0.66 | 15.30 | 0.21 | <0.01 | 200gm |
| BT-011 | 3.08 | 11.80 | 61.60 | 0.54 | 3.74 | 0.32 | < 0.01 | 0.72 | 0.58 | 16.84 | 0.28 | <0.01 | 215gm |
| BT-012 | 3.24 | 11.90 | 60.08 | 0.68 | 3.60 | 1.28 | < 0.01 | 0.76 | 0.62 | 17.64 | 0.44 | <0.01 | 215gm |
| BF-63 | 4.86 | 15.80 | 41.44 | 8.32 | 5.54 | <0.01 | < 0.01 | 0.66 | 0.60 | 17.94 | 0.49 | 3.08 | 250gm |
| BF150 | 3.84 | 13.66 | 54.48 | 0.90 | 4.30 | <0.01 | < 0.01 | 0.90 | 0.58 | 22.44 | 0.28 | 0.02 | 220gm |
| BF-250 | 2.70 | 10.12 | 59.36 | 0.54 | 3.20 | 0.40 | < 0.01 | 0.58 | <0.01 | 22.46 | 0.43 | <0.01 | 200gm |

Geochemical Laboratory Desk Page 1

# Appendix C -Materials and reagents used in the experiment

(a). Raw materials and their flotation response


Floated gangue materials
in the flotation cell
Magnetite ore

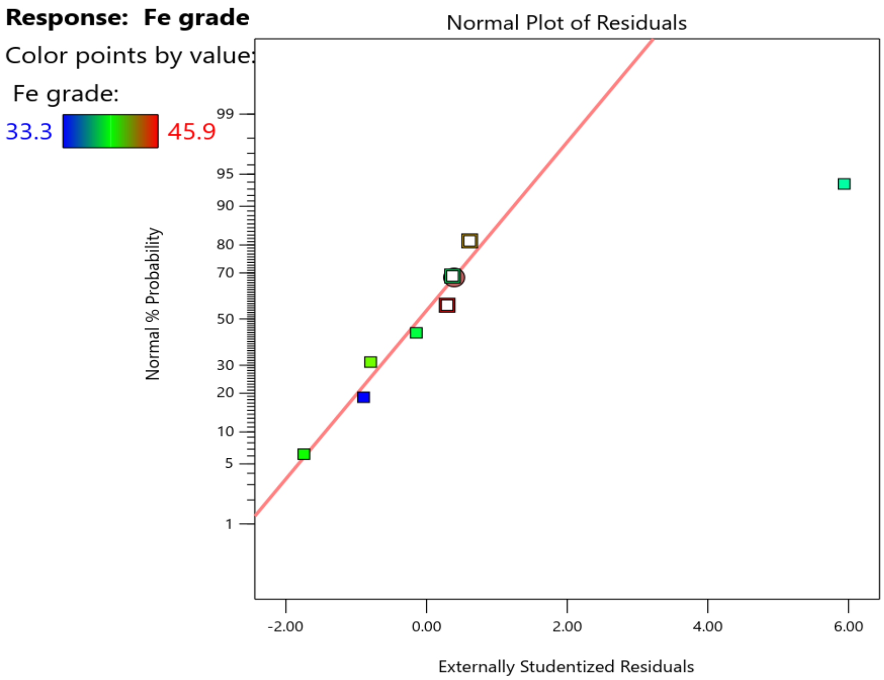
(b). Reagents


Sodium Hydroxide
AR
Pellet
Assay: 99.8%
Size: 500 gm
pH regulator
STARCH MAIZE (CORN)
Extra Pure
LOBA CHEMIE PVT. LTD.
Depressant
Calcium chloride
CaCl2
Activator
Oleic Acid
Extra Pure
collector
PINE OIL
Frothing reagent

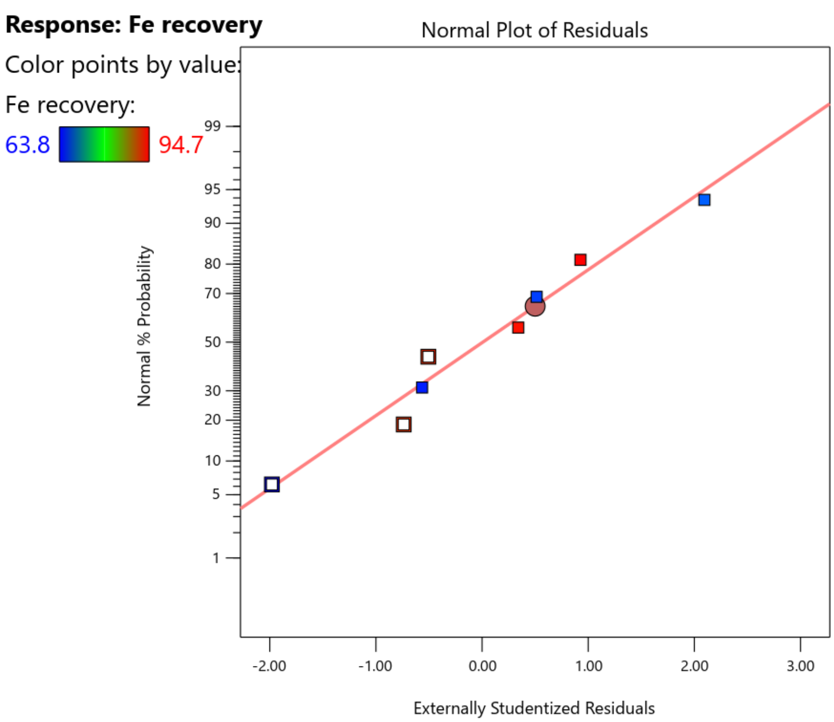
(c). Feed preparation equipment

## Appendix D- Normal Plots for Residuals

(a). Fe grade (%)

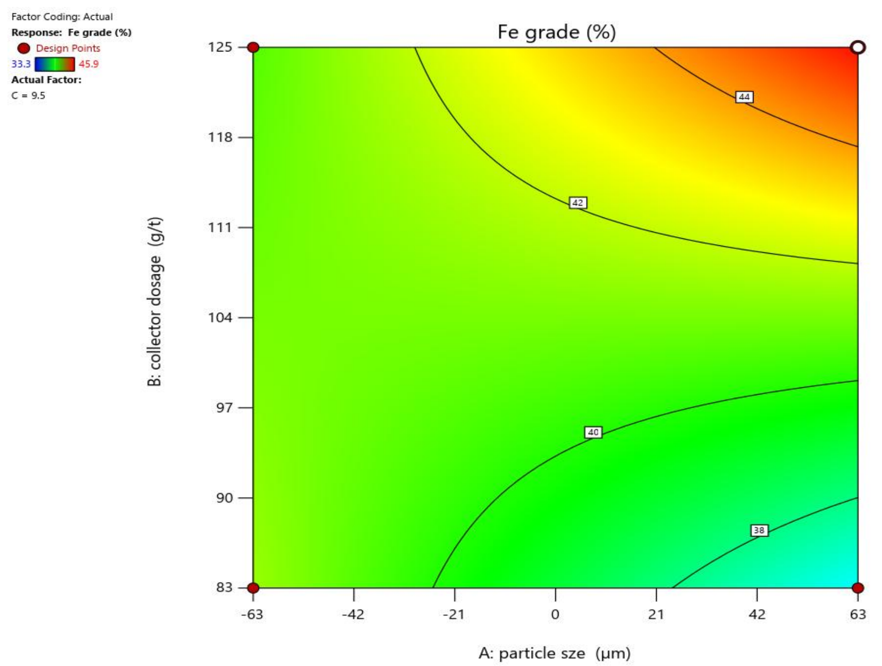



(b). Fe recovery (%)



## Appendix E- Contour maps displaying experimental responses

(a). Fe grade (%)




(b). Fe recovery (%)