Reverse Flotation of Hematite from Low-Grade Iron Plant Tailings: Optimization of Flotation Reagents

Promise Msinga, Willie Nheta

***Abstract*— High-grade iron ore feed is essential for efficient furnace operation, as it reduces coke consumption, improves energy efficiency, and lowers greenhouse gas emissions. However, many mineral processing plants produce tailings with low Fe grade, which is below the required +65% Fe grade for blast furnace feed. Disposing of these tailings not only represents a loss of valuable iron but also contributes to growing tailings facilities that pose environmental and community risks. This research aimed to upgrade the iron content of plant tailings to meet furnace specifications and reduce overall waste generation. Iron ore plant tailings were characterized using XRF, ZRD and SEM to evaluate their suitability for concentration using the flotation process. The expected outcome from flotation is a final iron-rich product exceeding 65% Fe, leading to improved process efficiency, reduced tailings generation, and a smaller environmental footprint. A concentrate containing 92% Fe_2O_3 was obtained using a combination of sodium oleate and Lupromin FP 18AS as co-collectors.**

***Keywords*— Reverse flotation, hematite recovery, quartz flotation, gibbsite removal, iron depression.**

I. Introduction

Iron and steel industries rely on iron ores that meet specific grade and mineralogical standards. Many iron plant tailings contain substantial amounts of quartz (SiO_2) and alumina-bearing gangue minerals. These gangue phases lower the overall iron content. They also interfere with beneficiation by increasing the mass of low-value material that must be removed. For example, [1] showed that quartz in iron ore increases flux consumption and reduces furnace efficiency.

To produce a concentrate fit for furnace feed, iron grades of about 65% Fe or higher are commonly required. Achieving this helps lower slag volume, reduce coke use, and improve thermal performance. The consequence of not reaching the Fe grade is that energy consumption, emissions, and operational costs can rise significantly. [2] found that reverse flotation of a South African banded iron formation achieved only approximately 63% Fe, emphasizing the challenge of beneficiating iron plant tailings.

Reverse flotation is a promising route for tailings with siliceous and aluminous gangue. Instead of floating the iron minerals, reverse flotation focuses on removing the gangue and leaving the iron-rich fraction behind. [3] noted that mixed anionic/cationic collector systems are emerging for such applications. However, few studies have systematically explored collector dosage, pH, and their interactions specifically for iron plant tailings with both quartz and alumina gangue.

This study focuses on the characterization of iron ore plant tailings with the aim of recovering hematite by reverse flotation. A cationic collector (Lupromin FP 18AS), typically used for sulfides, was tested for quartz flotation. It was paired with sodium oleate as an anionic collector targeting the alumina-rich gangue. The collector combination was evaluated via a Box–Behnken experimental design varying pH and collector dosages. The aim was to produce a hematite concentrate with an Fe grade greater than or equal to 65% while maintaining good recovery. The findings contribute to resource efficiency, improve furnace-feed quality, enhance sustainability, and support Sustainable Development Goal 12 (Responsible Consumption and Production).

II. Materials and methods

The experimental work was conducted to evaluate the reverse flotation response of hematite from iron plant tailings under controlled laboratory conditions. The section outlines the materials, reagents, and equipment used, as well as the experimental design and procedures followed. Characterization techniques such as XRF, XRD, and SEM-EDS were applied to determine the chemical composition, mineral phases, and textural features of the feed material. The flotation tests were performed according to a Box–Behnken design to assess the influence of collector dosage and pH on hematite grade and recovery.

A. Materials

The feed material consisted of iron plant tailings collected from Kumba Iron Ore, South Africa. The sample was riffled, milled, and prepared for flotation testing. Reagents used included methyl isobutyl carbinol (MIBC) as a frother (supplier Betachem (Pty) Limited), sodium oleate as an anionic collector, Lupromin FP 18AS as the cationic collector, corn starch as a depressant from Sigma-Aldrich (600 g/t constant dosage), and CaO (from Sigma-Aldrich) and HCl for pH adjustment. Laboratory-grade tap water was used to prepare slurries and reagent solutions

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B. Equipment

Flotation tests were performed using a laboratory flotation cell D12 (3L capacity). A PHS-3BW benchtop pH meter was used to monitor and adjust pulp pH. A spinning riffler from Eriez Magnetics model 10 Way with feeder model 15A and a Jones riffler were employed for sample splitting and subsampling. Chemical composition of feed and products was analyzed with X-ray fluorescence (XRF) (Rigaku; instrument model ZSX Primus II). Mineralogy and phase identification were carried out using X-ray diffraction (XRD) (Rigaku; instrument model Ultima IV X-RAY DIFFRACTOMETER) and scanning electron microscopy (SEM) with energy dispersive spectroscopy (TESCAN model). These instruments provided grade and particle association data supporting the flotation results.

C. Design of flotation experiments

A Box–Behnken response-surface design was selected to investigate three main factors: (1) pH (range 4 to 10), (2) sodium oleate dosage 750 – 1000g/t, and (3) Lupromin FP 18AS dosage 45 – 90g/t. A total of 15 flotation runs were conducted, including center-point replicates. The depressant (corn starch at 600 g/t) and the frother (one pipette drop of MIBC) were held constant. Operating conditions such as pulp density (~15 wt% % solids), impeller speed (1100 rpm), and airflow rate (0.10 m³/min) were maintained constant. The goal was to identify the combination of factors that maximized hematite grade while achieving acceptable recovery of iron.

D. Experimental procedure

The feed material was first characterized to assess its chemical composition and mineral phases. XRF was used to determine the Fe grade and quantify major gangue elements such as Si and Al. XRD identified the dominant mineral phases, including hematite, quartz, and alumina-bearing species. Scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS) was used to examine particle morphology and mineral associations relevant to flotation behavior.

The bulk tailings sample was crushed using a jaw crusher to approximately 2mm and homogenized. Representative subsamples were obtained using a Jones riffler, followed by further splitting with a spinning riffler to produce 500g portions for milling. A milling curve was developed using 500g samples in a laboratory ball mill containing 9.43kg of mixed-size steel balls (11-40 mm) and 500mL of tap water. The optimal milling time was determined to be 28 minutes, giving 80% passing 75 microns. About 10% of the milled product was coarser than 212 microns; this fraction was screened out and recycled to the milling to ensure a consistent feed size for flotation. Based on these results, 450g portions were prepared for each flotation run.

Flotation reagents included sodium oleate as an anionic collector for alumina-bearing minerals, Lupromin FP 18AS as a cationic collector for quartz, corn starch as an iron depressant (600g/t constant), and methyl isobutyl carbinol (MIBC) as the frother. Stock solutions were prepared by dissolving 1 g of reagent in 100mL of tap water. The required volumes were withdrawn by syringe according to each run's conditions.

For each test, 450g of milled feed was transferred to a 3L flotation cell containing tap water at approximately 1 wt% solids. The pulp pH was adjusted with CaO or HCl to the desired level and agitated at 1100 rpm. Conditioning was carried out sequentially: Lupromin FP 18AS (2 min), sodium oleate (2 min), and corn starch (5 min). A single drop of MIBC was then added, and air was introduced at 0.10 m³/min to initiate flotation. Concentrates and tailings were filtered, dried, weighed, and analyzed by XRF to determine Fe grades and evaluate flotation performance.

III. Results and Discussion

A. Milling curve of the as-received sample

Fig. 1 shows the milling curve obtained from batch grinding tests (9.44 kg ball-mill), by plotting the percentage of material passing 75 microns versus milling time. A linear regression of Y = 2.4214x + 11.72604 (R^2=0.99876) was fitted, where Y is the percent passing 75 microns and X is the milling time in minutes. By setting Y = 80% one obtains X = 28.2 min, and by setting Y = 90% the corresponding time is X = 32.3 min. This shows that to reach an 80 % passing 75 microns cut size, the required time is approximately 28 min, whereas pushing to 90 % passing would require approximately 32 min.

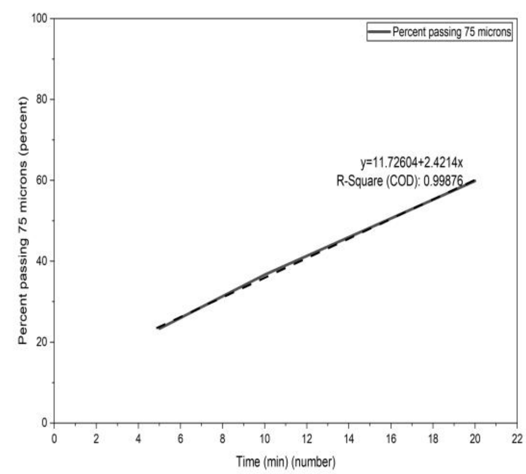

Fig. 1 Iron plant tailings milling curve

Selecting 28 minutes for the flotation feed in the reverse flotation tests, therefore, represents a compromise. On one hand, it ensures reasonable liberation of the hematite from quartz silicate gangue, enabling effective reagent–mineral interaction and flotation of silica (gangue) in the concentrate fraction. On the other hand, extending grinding to 32 minutes (for 90 % passing) risks over-grinding. A study by [4] found that ultrafine magnetite and hematite particles agglomerate and reduce flotation performance by forming slimes and surface films. Another study investigating quartz/hematite systems [5] demonstrated that very fine particles and high solid concentrations lead to elevated entrainment of gangue and lower selectivity.

Grinding for less than 28 minutes would leave a larger proportion of particles above 75 microns and insufficiently liberated. In the context of the project, incomplete liberation means hematite is still locked with quartz, thereby lowering hematite recovery or increasing quartz contamination in tailings. For example, [6] showed that coarser size fractions (>125 microns) achieved worse recovery in industrial-scale flotation due to insufficient liberation. Accordingly, 28 minutes was chosen as the optimum

B. Chemical composition of the as-received sample

Table 1 presents the major bulk chemical composition of the head sample as determined by XRF. The values reported are Fe = 58.41%, Si = 4.57% and Al = 2.49%. These numbers represent the principal metal of the feed to the beneficiation circuit and serve as a baseline indicator for the material's suitability for downstream processing. XRF is widely used for rapid chemical screening in iron ore beneficiation studies [7].

Components	Fe	Si	Al
Grade (%) wt	58.41	4.57	2.49

The Fe content of 58.41 % suggests the material is moderately rich in iron, though not at the premium levels typically targeted for direct sinter or pellet feed. The combined Si and Al content (7.06 %) signals a significant gangue load, likely associated with quartz (SiO_2) and alumina‑bearing minerals (Al_2O_3‑bearing phases). Literature on iron ore tailings and low‑ grade resources shows that elevated silica and alumina contents often necessitate removal via flotation, magnetic separation, or hybrid methods [8]. Reverse flotation is considered effective for removing quartz gangue from hematite/iron‑oxide systems when silica is a dominant impurity [7]. While the XRF data do not identify mineral phases, they do support the choice of a beneficiation route focused on gangue removal.

C. Mineral phases of the as-received sample

Fig. 2 shows the XRD pattern of the head sample from the iron-plant tailings. The dominant crystalline phases identified include hematite (Fe_2O_3), magnetite (Fe_3O_4), and quartz (SiO2), with minor peaks corresponding to iron-silicate minerals (possibly fayalite, Fe_2SiO_4).

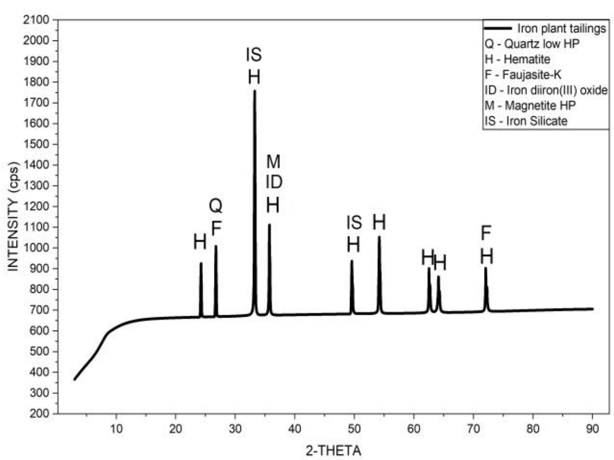

Fig. 2 XRD pattern for the as-received iron plant tailings.

The absence of peaks associated with gibbsite (Al(OH)3) or kaolinite (Al2Si2O5(OH)4) indicates that alumina is not present as discrete hydrous phases but rather occurs within silicate structures. The co-existence of hematite and quartz is typical of low-grade banded iron formations, and the strong quartz peak confirms that silica is the dominant gangue mineral. The minor presence of iron-silicate phases is problematic for reverse flotation, as such phases tend to have intermediate surface properties, partially hydrophobic and iron-bearing, which can lead to poor selectivity between hematite and silicate gangue. Iron-silicate minerals can consume reagents, especially cationic collectors and depressants, due to their mixed surface charge behavior at neutral to slightly alkaline pH [1].

The absence of discrete alumina phases but the detection of iron-silicate peaks also suggests that a portion of Al may be structurally bound within quartz or feldspathic matrices. This structural alumina is difficult to remove by flotation and may increase alumina levels in the concentrate, lowering its metallurgical quality [2]. Therefore, optimizing collector type and pH control is essential to enhance hematite–quartz separation and minimize entrainment of silicate-bound alumina.

D. Surface morphology and mineral associations of the as-received tailings sample

Fig. 3 presents a back-scatter SEM image at 500x magnification with three marked EDS spectra of the as-received sample. The elemental compositions from the EDS analyses show that Spectrum 1 has O = 56.86 wt%, Fe = 33.53 wt%, Si = 7.38 wt%, and Al = 2.25 wt%; Spectrum 2 has O = 58.67 wt%, Si = 26.47 wt% %, Fe = 12.46 wt%, and Al = 2.18 wt%; and Spectrum 3 has O = 52.58 wt%, Fe = 35.24 wt%, Al = 5.15 wt%, and Si = 6.18 wt%. The bulk XRF composition (Fe = 58.4 wt%, Si = 4.6 wt%, Al = 2.5 wt%) and XRD phases (hematite, quartz, magnetite, iron silicate, and faujasite-K) confirm an iron-oxide-dominated sample with minor silicate gangue. Based on the EDS compositions, Spectrum 1 represents a hematite grain, Spectrum 2 corresponds to a silicate-rich particle, and Spectrum 3 indicates an intergrown hematite–aluminosilicate particle. These mixed textures imply partial mineral locking and variable degrees of liberation.

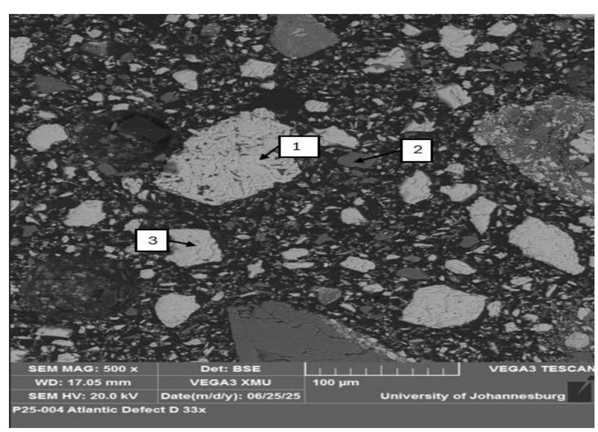

Fig. 3 SEM image of the as-received iron tailings sample

The 100 micron scale bar in the micrograph represents the actual field size of the image and allows estimation of individual grain dimensions. Most hematite grains observed are between 40 and 80 microns, while the silicate fragments are finer and more angular. These textural observations guided the selection of a target grind size of 75 µm, ensuring that approximately 80 % of the material passes this size while limiting fines below 10 µm to less than 10 %. Grinding to this degree promotes sufficient liberation of the iron oxides from the silicate gangue, as indicated by the separation between bright hematite and grey silicate regions, without generating excessive ultrafine slimes that would increase entrainment losses or depress recovery. The SEM–EDS evidence thus confirms that a 75 micron product provides an optimal balance between liberation and flotation selectivity for the reverse flotation of hematite.

E. Flotation experiments

Flotation experiments revealed that using a combination of sodium oleate and Lupromin FP 18AS, together with cornstarch as a depressant, Fe_2O_3 was increased to 92%.

IV. Conclusion

The results indicate that the reverse flotation route applied to the hematite-rich tailings achieved a balance between adequate liberation and effective separation of silicate gangue. The optimized milling condition, corresponding to 80 % passing 75 microns, produced sufficient mineral liberation without generating excessive fines that reduce selectivity. The XRF, XRD, and SEM-EDS analyses confirmed hematite as the dominant phase with quartz and aluminosilicates as the principal impurities, validating the chosen reagent scheme. The use of sodium oleate and Lupromin FP 18AS in combination, together with cornstarch as a depressant, enhanced the selective removal of quartz and alumina-bearing phases. Further studies are required for the optimization of the flotation process.

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