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#### **Research article**

# The effects of partially replacing amine collectors by a commercial frother in a reverse cationic hematite flotation



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#### ABSTRACT

Iron ore froth flotation has recently been used to obtain pure minerals due to the depletion of high-grade deposits. Amine collectors have often been used in cationic reverse iron ore flotation to achieve the separation with quartz impurities. The main problem associated with these collectors is related to the control of frothing properties and the high cost of amine reagents. In this paper, we report the benefits of partial replacement of amine collectors with a frother in reverse cationic flotation of hematite. Surface tension and batch flotation experimental results indicated for the first time that replacing Flotigam EDA, an etheramine collector by more than 10% improved the flotation performance in both grade and recovery of hematite. It was also noted mild hematite entrainments for lower frother dosages whilst reagents cost estimation indicated profitability per ton of iron recovered. At higher frother dosage however, higher quartz recoveries were observed as a result of entrainment which led to satisfactory results in terms of iron grade in the sink, but results showed higher costs of reagents than when collector was used alone. From observed results, it was concluded that partially replacing amine collector by a frother does improve the performance of the flotation globally regardless of pronounced entrainment that was mainly due the mineralogy of the ore with notable amounts of locked particles in the lower sizes. Finally, it would be useful to investigate these findings with alternative reagents and on minerals with different mineralogical properties.

#### 1. Introduction

Hematite is one of the most abundant and economically exploitable iron minerals and contains up to 69% metal iron [1, 2]. The main gangue associated with most iron ores is quartz [3]. The primary separation method of hematite from its gangue materials has generally been magnetic separation process since iron minerals are both ferromagnetic and paramagnetic [4]. The depletion of high-grade iron ore necessitates an upgrade. Froth flotation has been proven to be one of the most suitable separation methods used to this end [3, 5, 6]. The increased use of iron ore flotation has resulted in the reduction of gangue impurities [7]. Flotation has thus produced iron "super-concentrates" with limited contents of silica, aluminium oxide and other impurities prior to subsequent metallurgical processes [8, 9]. Based on the collector used, iron-bearing metallic mineral flotation operations are of two main types: anionic and cationic. The difference between the two methods is related to which material (valuable mineral or gangue) is floated and recovered to the concentrate. The approach to be used is determined by preliminary

test results, weight relationships of the valuable mineral and gangue, and the type of reagents used. In anionic flotation, fine-sized crystalline iron oxides, such as hematite or siderite, are floated away from siliceous gangue material such as quartz or chert. In cationic flotation, silica or the gangue material is floated and the value-bearing minerals are removed as underflow, referred to as reverse flotation [3, 5].

Traditionally, cationic iron ore concentrators use amine collectors that act as both collector and frother [3, 6, 10, 11, 12]. There are two potential problems with this type of approach [6]. Firstly, control of flotation conditions is reduced when using one reagent with dual functions [13, 14, 15, 16]. Secondly, amines are expensive reagents, and it may be financially lucrative to replace them with cheaper frothers [17]. It would be desirable to have independent control over the collecting properties and frothing properties. Very few studies addressing this issue can be found in the literature though a handful of commercial and non-commercial frothers have been developed and tested on iron ore flotation. For instance, capryl alcohol was tested a few decades ago as a novel frother to partially replace amine collector and yielded satisfactory

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results that were patented [18]. Partial replacement of amine collectors has been investigated at laboratory scale [3]. Selected commercial frothers (synthetic polyglycol and pine oil types) have performed well when only with 10% replacement of amine collector dosage while linear alcohol frothers did not show any improvement. An increase beyond the 10% proportion resulted in lower performance in terms of both recovery and selectivity. Furthermore, several other groups have used many types of frothers for iron ore flotation [12, 19, 20, 21]. For instance, Nheta and Phasha have recently shown some improvement on the flotation performance with the use of Macadamia oil as an alternative frother [22]. Their study did not however show how the observed improvement would abate the dosage of collectors.

Thus, the aim of this project was to assess the flotation performance when partially (beyond 10%) replacing excess amine collector with a commercial frother. Frothers have been generally acknowledged to be very important in terms of their role with respect to bubble size, and the stability and mobility of the froth phase [25]. Though several types and classifications exist, common frothers have been intensively used in several mining plants. For instance, frother  $\alpha$ -terpineol, Methyl Isobutyl Carbinol, and DowFroth200 (DF200) together contribute to more than 90% of frother usage in froth flotation industry in the world [26]. Maturity in manufacturing technology, effectiveness (high performance) and mainly low cost are the main reasons these frothers are often employed. A frother of the same class, a mixture of alcohols and mainly polypropylene glycol methyl ether (Senfroth 200) was used in this study. Surface tension has been proven to be one of the most important factor in froth flotation [23, 24]. It follows that the existence of a sufficient difference between surface tensions of two hydrophobic surfaces, selective wetting can be reached by altering (lowering) the surface tension of the liquid. At a suitable value of liquid surface tension, particles with higher surface tension value will be wetted sufficiently while air bubbles would adhere onto the particles with lower surface tension value. Against this background, we hypothesized that there is a collector concentration (dosage) for which there was sufficient collector present to act as a collector, but not an excess so that it begins to act as a frother. Hence, surface tension measurements of solutions were assessed. Flotation performance was assessed using valuable mineral recovery and grade after froth flotation. Water recoveries and entrainment were measured to assess the frother dosages response.

#### 2. Materials and methods

#### 2.1. Materials

A sample of iron ore used in this study was obtained from a South African mine. The main iron-bearing mineral was hematite, with quartz as the main gangue mineral. The initial bulk sample of the iron ore approximately weighing 200 kg was screened on a 3.35 mm aperture size screen. The retained material (+3.35 mm) was crushed using a cone crusher and the material was then blended with the passing sample (-3.35 mm). The bulk sample was riffle split into buckets and further split and stored into representative portions (around 1 kg) using a rotary sample splitter. An Eriez stainless steel rod mill with a diameter of 200 mm, charged with 20 rods of varying diameter with the following ratio: 6

 $\times$  25 mm, 8  $\times$  20 mm, 6  $\times$  16 mm was used to mill the ore. For all the flotation tests, 1 kg bags of ore were milled using tap water (60 wt% solid). A milling curve was generated to obtain the required milling time to achieve a flotation feed of 80% passing 150 µm. A particle size distribution of the milled product was determined using both wet and dry screening and a chemical analysis by size was undertaken using X-ray fluorescence spectroscopy (Table 1) and QEMSCAN (Quantitative Evaluation of Minerals by SCANning electron microscopy).

Chemical analysis of the feed illustrated in Table 1 shows that more than 80% of hematite reside in the smaller size classes (sub 53  $\mu$ m), and the gangue material is mainly quartz (SiO<sub>2</sub>) and aluminium oxides (Al<sub>2</sub>O<sub>3</sub>). Quartz, amounting up to 17% lies on all size classes whereas aluminium oxides represent around 2% of the ore. The remaining fraction making up to 4% is made of traces of more than ten minerals including TiO<sub>2</sub>, NiO, CaO.

#### 2.2. Reagents

Gelatinised corn starch (Sigma-Aldrich, South Africa) was used as depressant throughout this study. A 2 wt% solution of starch was prepared by adding an accurately measured mass of starch to a known volume of distilled water and about 20 vol% of 10% sodium hydroxide solution. The mixture was stirred until gelatinized. All flotation tests were performed at 1500 g/t soluble starch. Scoping experiments were performed at three depressant dosages and the highest dosage was chosen to depress all floatable hematite minerals.

Flotigam EDA (Clariant, 98% purity), an etheramine was used as collector (Analytical grade) and polypropylene glycol methyl ether (Senfroth 200, Betachem) was used as frother. It is worth noting that with the knowledge of the purity of both frother and collector, the dosage was determined based on the mass of the dry sample to be floated. Thus, the corresponding volume of these reagents was added to the slurry.

#### 2.3. Instrumentation

Dynamic surface tension was measured using the Kruss tensiometer employing the Du Nouy ring method. The measurements were performed with a platinum ring which was submerged in the liquid (solution). As the ring was pulled out of the liquid, the force required to detach it from the liquid surface was precisely measured. This force is related to the liquid surface tension. The platinum ring was maintained continuously cleaned with ethanol after each measurement to avoid blemishes or scratches and to ensure greater results accuracy.

Surface tensions of solutions of increasing collector dosage at constant depressant dosage were measured in triplicates. To know at what dosage there was enough collector present to act as a collector, but not an excess so that it begins to act as a frother, surface tension measurements were assessed. The other reagent conditions such as pH and depressant (soluble corn starch) were monitored and held constant.

#### 2.4. Methods

Bench Flotation experiments in duplicate were performed in a 3 L Barker flotation machine. Agitation time with starch depressant was 5

<b>Fable 1.</b> Chemical analysis showing weight percentages of the feed per size class.													
Size (µm)	SiO <sub>2</sub>	TiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	MnO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	$P_2O_5$	SO <sub>3</sub>	$Cr_2O_3$	NiO	H <sub>2</sub> O
-25	15,04	0,17	3,52	77,19	0,06	0,33	0,56	0,62	0,19	0,03	0,08	0,04	0,13
+25 to-38	14,44	0,14	1,92	80,75	0,08	0,20	0,58	0,33	0,14	0,02	0,11	0,06	0,02
+38 to-53	17,01	0,13	2,08	78,48	0,07	0,17	0,57	0,36	0,14	0,02	0,10	0,05	0,06
+53 to-75	17,27	0,12	2,05	77,81	0,07	0,14	0,59	0,37	0,12	-	0,07	0,04	0,04
+75 to -150	18,56	0,12	2,16	76,29	0,08	0,12	0,57	0,38	0,12	0,01	0,12	0,03	0,04
+150 to -212	18,02	0,12	2,26	77,86	0,07	0,11	0,59	0,40	0,11	-	0,11	0,03	0,00
+212	17,88	0,12	2,29	76,76	0,11	0,09	0,58	0,39	0,11	-	0,08	0,04	0,07



Figure 1. Schematic diagram of bench flotation procedure.

min. Conditioning time with the collector was 2 min. Stirring with reagents was conducted directly in the flotation cell, with a pulp density of 33 wt% and the pH was maintained at a value varying between 9.5 and 10.5 (adjusted using dilute HCl or NaOH solution). After conditioning these reagents (total conditioning time: eight minutes), air switched was opened and maintained at a constant flow rate of 7 L/min while the stopwatch was restarted and the froth scraped off every 15 s for two, four, six and eight minutes for concentrates 1,2,3 and 4, respectively. At the end of the flotation experiment, air supply was closed, and 100 ml of tailings sample collected in syringes. Feed and tail samples were vacuum filtered and dried in an oven (60 °C) for 6 h. Chemical assay for iron of the concentrates, feed and tail samples was performed using X-ray fluorescence. Figure 1 illustrates the flotation procedure used adapted from Wiese [27].

#### 3. Results and discussion

#### 3.1. Surface tension of solutions

Water-collector solutions in proportions corresponding to the dosages 0, 25, 50, 75, 100 g/t were made up and are referred to as solution 1. Secondly, slurry samples (33% solids) were prepared and the reagents at the same concentration as above were added and the mixtures stirred for conditioning time of 5 min while the pH was adjusted to 10 by adding either 5% sodium hydroxide or 32%

hydrochloric acid. The slurries were then centrifuged at 4000 rpm for 10 min, the surface tensions of the clear liquid (referred to as solution 2) after having been in contact with iron ore, were measured. The discrepancy in surface tensions between solution 1 and solution 2 was a mean to assess the amount of collector that had adsorbed on the surface of the minerals.



Figure 2. Surface tension vs collector EDA concentration.



Figure 3. Surface tension vs collector concentration.





As expected, the surface tension decreased with an increase in collector concentration at a constant depressant dosage, for both solutions. Surface tensions of solution 1 were lower than that of solution 2 as clearly illustrated on Figure 2. This observation indicates that a considerable amount of collector ions had adsorbed on the surface solids, thus reducing the number of free ions in solution that were available to act as surfactants at the air-water interface and thus lower the surface tension for solution 2 [28]. The same finding may also be the evidence that some of the EDA collector adsorbed onto the mineral surfaces since it was no longer available to act as a surfactant.

Furthermore, surface tension measurements were performed with solutions made up with several collector concentrations. Figure 3 shows a steep drop of surface tension between 10 g/t and 25 g/t collector dosages and a constant run from 25 to 75 g/t, which shows that there is enough collector to form a layer on the surface of the minerals slightly below 25 g/t. As more collector was added, a substantial amount remains in solution, thus decreasing the surface tension. As more collector was added, multiple layers are expected to form on the minerals' surfaces and the surplus amount in solution is maintained constant, therefore the surface tension only slightly decrease. Two collector dosages in this region, representing 50% and 75% collector replacement were employed when replacing collector with a frother.

#### 3.2. Bench flotation results

Scoping experiments were performed at three depressant dosages and the highest dosage was chosen to depress all floatable hematite minerals. Any hematite that would be reporting to the concentrate in this case would be assumed to be so by entrainment. This allowed the separation of results into entrained and floatable hematite at different frother conditions. Two collector dosages (30 g/t and 60 g/t), representing 50% and 75% collector replacement by a frother were employed as guided by surface tension measurements. Figure 4 shows grades and recoveries of quartz at collector dosages of 30 g/t and 60 g/t, with frother dosages increasing from 25, to 50, to 100 g/t. Recoveries at 60 g/t collector dosage were higher than the recoveries at 30 g/t. This observation indicated that at low collector dosage the hydrophobicity of quartz particles is decreased and not necessarily a decrease in frothing effects of the collector [29, 30, 31]. Increasing frother dosage, at a constant 60 g/t

Notation: The first two digit represent collector dosage and the remaining ones the frother dosage. Example the following combination C30-F50 stands for 30 g/t of Amine (EDA) and 50 g/t of senfroth200.



Figure 5. The recovery rate of quartz using several frother dosage replacement.



Figure 6. Fe and water recoveries for <25 um particles.

collector dosage, resulted in an increase in quartz recovery and a decrease in quartz grade, as anticipated.

# Also, higher quartz grade observed for both C30-F50 and C60-F50 tests is induced by the faster solids kinetics as quartz is recovered at the start of the float when there's still high collector concentration in the pulp solution [29, 32]. Further increasing the frother dosage result in an increase in water recovery, decrease in both quartz recovery and its kinetics as shown on Figure 5. These trends were observed for all particle size classes.

Furthermore, quartz recovery was higher for tests performed with collector alone than all the tests where collector dosage was cut and replaced by the frother as illustrated with curve C100 on Figure 5.

#### 3.3. Effects of hematite entrainment

Chemical essays of concentrates clearly show that frother dosage is proportional to iron recovery and grade for sub 25  $\mu$ m particles (slimes) as shown in Figure 6.

Entrainment is very pronounced for finer particles than coarser as expected and promoted by higher water recoveries. It is assumed that larger particles were entrained through hydraulic by liquid convection and mechanical entrapment between consecutive bubbles. We also observed higher quartz recoveries at higher frother dosages (C60-F50 and C60-F100) due to both flotation and entrainment of liberated quartz particles. However, with sub 25  $\mu$ m particles of our ore comprising 80%



Figure 7. Water vs Fe recoveries.



Figure 8. Qemscan image of iron ore and mineralogical characterization





Fe, it was therefore expected to recover more hematite as well due to entrainment. Our observations are in agreement with previous experimental results [32].

Figure 7 further confirms entrainment of slimes with higher iron recoveries in the concentrates for sub 25 µm the particle class. Meanwhile, for particles sizes great than 25 µm, entrainment is reduced by one order of magnitude at the same reagent's conditions. Similar results were observed by Lima et al. [33] who determined an iron degree of entrainment of 0.53 for sub 20  $\mu m$  versus 0.05 for the particle class just above 40 µm. Frother dosages of runs C60-F50 and C60-F100 showed promising results as iron recoveries lies in the same range as C100 run, that is when higher collector dosage was used without a frother. Though, we expected to obtain higher quartz recoveries for smaller particle due to the higher specific area of this particle class [34], the mineralogy of the ore used (see below) in this study may be responsible of the opposite results. Furthermore, the variety in particle size distribution of our grinded ore would lead to more efficient separation. Higher grades of iron in the concentrate may not only be as results of the entrainment that is due to water recovery and thus, frother dosage. Some may be due to the mineral liberation of hematite particles. To this end QEMSCAN was used to characterize the mineralogy of the ore.

The minerals mapping of the iron sample used was determined in great details as the surface percentage calculated from the picture is illustrated on Figure 8 and are in agreement with XRF characterization results presented in Table 1. Though, several strategies have been developed to concentration iron ore, its mineralogical nature and thus mineral liberation remains a challenge for certain ores. Mhonde, Weise and Belinda have shown that South African iron ores iron have hematite particle entrap in silicate blocks for particles of all classes [35]. Their results showed that quartz particles are not well liberated and hence, higher starch dosages depress both hematite particles and locked particles. The same may be true for the collection aspect, that is the fraction of hematite particles that report to the concentrate are collected along with quartz particles due to non-liberation. It was also noticed that quatz would be fully liberated when the ore is grinded below 25 µm. However, doing so can only lead to several operational troubles with regards to froth pumping, over stabilising effect of slimes and eventually entrainment of fine hematite as shown in this study. Moreover, amine collectors cost nearly twice as much commercial frothers [36]. Thus, the cost advantage of partial replacing amine reagents by a frother is briefly illustrated by Figure 9.

We have computed the cost of reagents (both frother and collectors) that would be required to float our sample to recover a ton of metal iron. These values may differ based on iron concentration (grade) in the original ore to float: the higher the iron content in the ore, the more lucrative the estimation would be and vice-versa. From Figure 9, it is evident that all runs, but C30-F100 and C60-F100, result in lower reagents cost than C100 run (collector alone). From this rough assessment or a statistical analysis [37], it is clear that partial replacement of amine collectors by a commercial frother would be profitable. The use of higher frother dosage leads to higher cost of reagents per ton of iron recovered. Care should then be taken into account on the frother dosage as shown by both reagents cost estimation and hematite entrainment as mentioned in the previous paragraph. Though collector partial replacement by a frother has shown satisfactory results in terms of iron recovery and profitability, a more rigorous analysis needs to be conducted to generate a solid universal model [38, 39], independent from iron grade in the sample.

#### 4. Conclusions

In this study, a commercial frother was proven to successfully replace excess amine collector in a reverse cationic hematite flotation. With the use of laboratory batch flotation experiments and measurements of reagents mixture at different proportions, we were able to demonstrate for the first time that replacing the amine collectors by more than 10% improves the floatability of quartz and thus, improve the overall recovery of hematite floatation. While research on the use of frothers in iron ore concentrators on a pilot scale is in progress, the following conclusions currently be drawn:

- 1. Partial replacement of excess Flotigam EDA collector by the commercial frother Senfroth 200 up resulted in an overall improvement of hematite flotation recovery. When cutting collector dosage by half, true flotation recovery improved while 75% replacement showed a performance decline.
- 2. Higher frother dosage resulted in a decline in the overall grade due to water recovered that promoted entrainment of hematite minerals.
- 3. There was no clear difference observed between frothing characteristics of the amine collector and the frother used, except for higher water recovery when more frothers were used.
- Regardless of complex mineralogical characteristics of the South African ore, it would be financially lucrative to replace excess collectors by a frother at relatively low dosage.

#### Declarations

#### Author contribution statement

K. Fabrice Kapiamba Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Merveille Kimpiab: Analyzed and interpreted the data; Wrote the initial draft and reviewed the paper.

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#### Data availability statement

Data included in article/supplementary material/referenced in article.

#### Declaration of interests statement

The authors declare no conflict of interest.

#### Additional information

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