

# Evaluation of a mixed collector made of dodecylamine and fatty acids in the direct flotation of iron ore tailings

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## Abstract

Mining tailings have become a major problem for society, especially after the tailings dam collapses in the cities of Mariana - MG and Brumadinho - MG, in Brazil. This study aims to investigate the possibility of using a mixed collector composed of dodecylamine (DDA) and a fatty acid (GA) reagent in the direct flotation of iron ore tailings. The sample used in this study (10.8% Fe) consists of quartz and hematite, the particles of which are released. The direct flotation tests revealed that the mixed collector at a molar ratio of DDA:AG of 1:7.0 produced better results than AG alone. Increasing the dosage of the mixed collector DDA:AG 1:7.0 did not produce satisfactory results in terms of Fe content and recovery. However, the tests carried out on the -150  $\mu\text{m}$  iron ore tailings sample using the DDA:AG 1:7.0 mixed collector produced a final concentrate with 64.04% Fe. These results can be explained by the greater collection efficiency and selectivity promoted by the mixed collector, due to the greater degree of compaction of the monolayer formed by the reagent, which results from the lower electrostatic repulsion between ions of the same species.

**Keywords:** Iron ore tailings; Direct flotation; Mixed collector; Dodecylamine; Fatty acids.

## Avaliação de um coletor misto constituído por dodecilamina e ácidos graxos na flotação direta de rejeitos de minério de ferro

## Resumo

Os rejeitos de mineração têm se tornado um grande problema para a sociedade, principalmente após os rompimentos das barragens de rejeitos nas cidades de Mariana - MG e Brumadinho - MG, no Brasil. Este estudo tem como objetivo investigar a possibilidade de utilização de um coletor misto composto por dodecilamina (DDA) e um reagente de ácido graxo (AG) na flotação direta de rejeitos de minério de ferro. A amostra utilizada neste estudo (10,8% Fe) é constituída por quartzo e hematita, cujas partículas são libertadas. Os testes de flotação direta revelaram que o coletor misto numa razão molar de DDA:AG de 1:7,0 produziu melhores resultados do que o AG sozinho. O aumento da dosagem do coletor misto DDA:AG 1:7,0 não produziu resultados satisfatórios em termos de teor de Fe e recuperação. No entanto, os ensaios realizados com a amostra de rejeitos de minério de ferro na granulometria -150  $\mu\text{m}$  utilizando o coletor misto DDA:AG 1:7,0 apresentaram um concentrado final com 64,04% Fe. Esses resultados podem ser explicados pela maior eficiência de coleta e seletividade promovida pelo coletor misto, devido ao maior grau de compactação da monocamada formada pelo reagente, que decorre da menor repulsão eletrostática entre íons da mesma espécie.

**Palavras-chave:** Rejeitos de minério de ferro; Flotação direta de minério de ferro; Coletor misto; Dodecilamina; Ácido graxo.

## 1 Introduction

Iron ore is an important global commodity, with the steel industry—a major sector of the global economy—being its main consumer. Approximately 58% of the world's iron ore production is concentrated in Australia and Brazil [1]. The decreasing average Fe content in deposits, combined with the

increasing consumption of iron by the steel industry, makes the use of low-grade iron ore fines and tailings inevitable [2].

However, the failure of the iron ore tailings dams in the Brazilian cities of Mariana, MG (2015), and Brumadinho, MG (2019), which caused irreparable environmental damage

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and the loss of hundreds of human lives, has brought attention to the management and disposal of tailings in the mining industry [3-5]. Since then, alternatives have been sought to reduce the risks of tailings deposition. One alternative is to develop co-products using iron ore tailings—mainly through geopolymers—in the civil construction sector [6-9]. Another alternative is to reprocess iron ore tailings through direct flotation of iron ore to obtain a concentrate with high Fe content and reduce the production of tailings.

The traditional system for direct flotation of iron ore consists of using fatty acids as collectors and silicate as depressants, such as sodium silicate and sodium hexametaphosphate [10-12]. In this system, the fatty acid is chemically adsorbed on the surface of the iron oxide through the formation of covalent chemical bonds between the surface of hematite and the carboxylate ion [13,14]. To obtain satisfactory results in terms of iron content and metallurgical recovery, a sufficiently high dosage of the collector must be used, due to the lower iron content in the flotation feed [14,15].

Several authors [16-24] have studied the effects of mixed collectors to increase selectivity in the flotation process and reduce the total consumption of reagents. When a mixed collector is exposed to a surface which, by definition, must have a heterogeneous distribution of energetically different sites, the weaker collector present on the mixed collector will be adsorbed preferentially on the sites with higher surface free energy, and the strongly collector will be adsorbed preferentially on the sites with lower surface free energy. Therefore, mixed collectors have a higher adsorption efficiency compared with a single collector reagent, which is mainly adsorbed on sites with higher surface free energy [16].

The mixture of sodium oleate and dodecylamine yields positive results on the floatability of hematite in comparison with the isolated use of dodecylamine [17]. This mixed collector also allows the lower floatability of quartz: the contact angle of quartz with the mixed collector is lower than that with a pure solution of dodecylamine [18,19]. Molecular dynamics simulation studies revealed that, compared with pure dodecylamine and pure sodium oleate, the compounds from the mixture of the two reagents are more compact and can form a compact monolayer on the water/air interface, indicating a greater activity on its surface. The hydrophobic carbon chains of mixed collectors extend towards the air at a smaller angle than in cases where pure reagents were used [20].

The use of mixed collectors made of fatty acids and dodecylamine was also investigated in other mineral systems, such as calcite/scheelite [21], muscovite [22], fluorite/calcite [23], and manganese/quartz [24]. In these examples, the results obtained with mixed collectors were better than those with individual collectors.

Therefore, in this study, we investigated the possibility of using a mixed collector made of fatty acids and dodecylamine in the direct flotation of an iron ore tailings.

## 2 Materials and methods

Iron ore tailings samples were collected from a mine in the Iron Quadrangle, MG, Brazil. These samples were extracted after decommissioning a tailings dam. A preliminary flotation test was performed on a sample without any reagents, in which there was no quartz flotation. This ensured that any residual amine adsorbed on the quartz surface would not impact other direct flotation tests using this sample. Figure 1 shows the grain-size distribution of the iron ore tailings sample. It is important to observe the low proportion of fines (<30  $\mu\text{m}$ ) – 5% – on the sample.

The mineral composition of the iron ore tailings was assessed through X-ray diffractometry (XRD) using a Philips-PANalytical PQ3710 and a CuK $\alpha$  radiation and graphite monochromator (range 3-90° 2 $\theta$ , step 0.02° 2 $\theta$ , time per count 3 s). Phases were identified using the International Center for Diffraction Data (ICDD) PDF-2, a collaborative product designed for the analysis of inorganic materials. The chemical elements in the sample were quantified through X-ray fluorescence (XRF), using a Thermo Scientific™ (EDFRX) ARL™ QUANT'X, equipped with a 50 W rhodium tube. The data was processed using WINTRACE software. The

Leitz/Leica optical microscope, model Orthoplan Pol, with Canon PowerShot S80 digital camera was used to obtain images of the iron ore tailings sample in the following granulometric ranges: [150-106]  $\mu\text{m}$ , [106-75]  $\mu\text{m}$ , [75-53]  $\mu\text{m}$ , and [53-38]  $\mu\text{m}$ . The degree liberation was obtained by Gaudin method.

The following reagents were used in this study: dodecylamine PA (DDA), in analytical grade, provided by Sigma Aldrich; a commercial fatty acid reagent (named AG); and sodium silicate, provided by Sulfal Química. The fatty acid composition of the AG reagent was assessed through gas chromatography using the chromatograph Agilent HP7820A (Table 1). The AG reagent was saponified with the addition of NaOH on a mass proportion NaOH:AG of 1:6 and subsequent stirring and dilution at 5% w/v. The DDA reagent was directly diluted in water in a 1% w/v solution. Before the flotation tests with the two reagents, the mixed collector was prepared by mixing in the following molar ratios of DDA/AG: 1:1.4; 1:2.8; 1:4.2; 1:5.6 and 1:7.0.

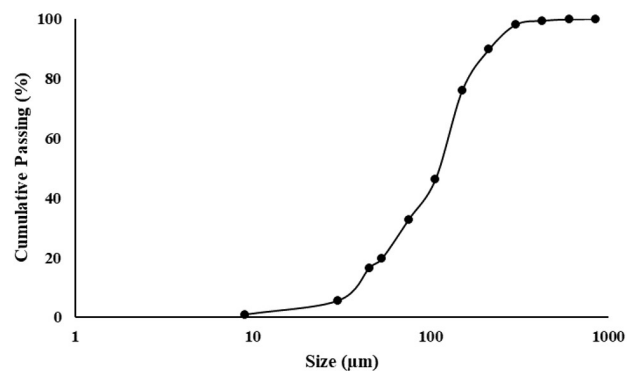


Figure 1. Grain-size distribution of the iron ore tailings sample.

**Table 1.** Distribution of fatty acids in the AG reagent

Fatty Acids						
Myristic	Palmitic	Stearic	Oleic	Linoleic	Linolenic	Others
C14:0	C16:0	C18:0	C18:1(9)	C18:2(9,12)	C18:3(9,12,15)	
0.4	17.5	5.1	28.6	40.1	0.2	6.1

The bench-scale flotation tests were initially performed in the rougher stage and later in the cleaner stage. Table 2 presents the conditions of the tests. Sodium silicate was used as a depressant of silica gangue at a dosage of 500 g/t and conditioned for 5 min. The collector was conditioned for 2 min, and the initial dosage was 100 g/t. No frother was used during the tests. There was no addition of reagents in the cleaner stage. NaOH and CH<sub>3</sub>COOH solutions (analytical grade purity) at 1% w/v were used to adjust the pH to 7.5 ± 0.2. The applied flotation cell was Denver's McDarma D12. Tests were carried out in duplicate.

### 3 Results and discussions

Figure 2 shows the diffractogram of the iron ore tailings sample, which consists of quartz (SiO<sub>2</sub>, 85%) and hematite (Fe<sub>2</sub>O<sub>3</sub>, 15%). The proportion of each mineral was obtained through Rietveld refinement. Table 3 shows the results of the granulochemical composition based on the XRF analysis. It should be noted that 54,2% of the Fe present in the initial sample is found in particle sizes of under 45 μm and 26.6% of the SiO<sub>2</sub> in particle sizes of over 150 μm. This data proves the greater presence of SiO<sub>2</sub> in the coarser fractions, and the greater presence of Fe in the finer fractions.

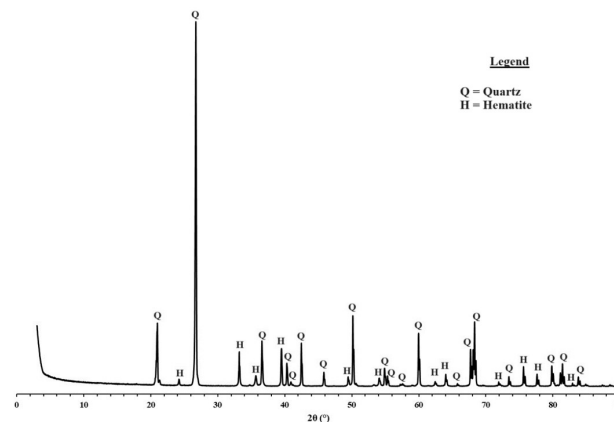
The chemical composition of the sample, when converted into percentages of the minerals quartz and hematite - identified in the XRD - shows the presence of 15.44% hematite and 83.90% quartz, values very close to those obtained by Rietveld refinement, corroborating the characterization techniques used.

Figure 3 presents the optical image analysis (OIA) obtained in an optical microscope for the size classes [150-106] μm, [106-75] μm, [75-53] μm, and [53-38] μm. The OIA shows that only quartz and hematite are present in the iron ore tailings sample. It also shows that very fine hematite grains are disseminated on the coarsest quartz grains.

This type of minerals association (quartz/hematite) certainly can negatively affect the recovery of hematite to the flotation concentrate. Based on this OIA, a liberation analysis was performed for both minerals quartz and hematite. These results are presented in Table 4. According to the liberation analysis performed it is observed that quartz reach a higher degree of liberation than hematite for all size classes analyzed, except for the size class [53-38] μm where the hematite presented higher liberation (94.09%) than quartz (92.18%). It is also observed that quartz liberation reached more than 81% for all size classes analyzed. Higher liberation degree for quartz minerals could not be observed

**Table 2.** Operational conditions of the bench scale flotation tests

Parameter	Information
Sample density	2.85 g/cm <sup>3</sup>
% solids on conditioning	50%
% solids on rougher stage	35%
% solids on cleaner stage	Variable (depends on the mass recovery on rougher stage)
Cell volume used in the rougher stage	1500 mL
Cell volume used in the cleaner stage	800 mL
Cell agitation level (on both stages)	1000 rpm
Air flow rate in flotation	4 L/min
Water used	Distilled water

**Figure 2.** Diffractogram of the iron ore tailings sample.

due to the association of fine hematite grains disseminated on the coarsest quartz particles.

Figure 4A shows the results of the tests with DDA and isolated AG as collectors. The DDA reagent was not efficient, as it produced a concentrate with only 21.46% Fe and 12.19% metallurgical recovery of Fe, indicating that this reagent is not a good collector for iron minerals, as presented in the literature [25-29].

The AG reagent significantly increased the Fe content of the iron ore tailings sample, obtaining 55.13% of Fe. The low recovery of SiO<sub>2</sub> in the concentrate with AG (0.83%) is also noteworthy, probably due to the dragging of quartz particles along with the foam. However, the mass recovery and, consequently, the metallurgical recovery of Fe in this test were also very low (3.56% and 18.23%, respectively).

This suggests that AG is very selective but has a low collecting efficiency for Fe-bearing minerals. The low collecting efficiency might be caused by a low collector dosage. Figure 4B shows the effects of increasing AG dosage in the flotation system. Increasing the AG dosage resulted in

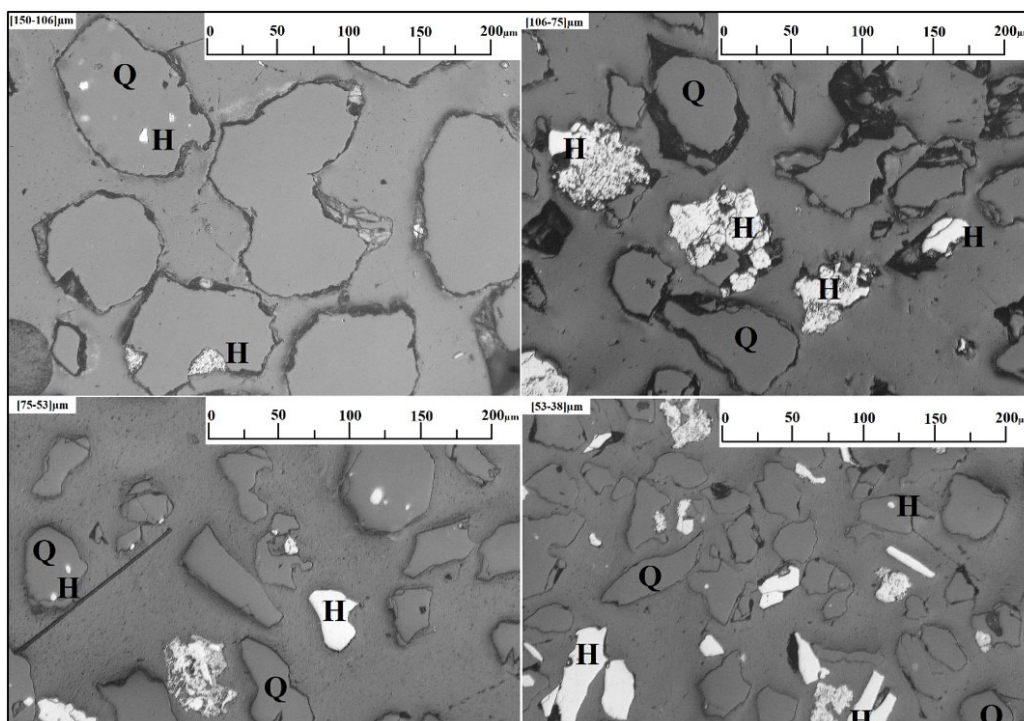


Figure 3. Optical microscope images of the iron ore tailings sample, showing quartz (Q) and hematite (H)

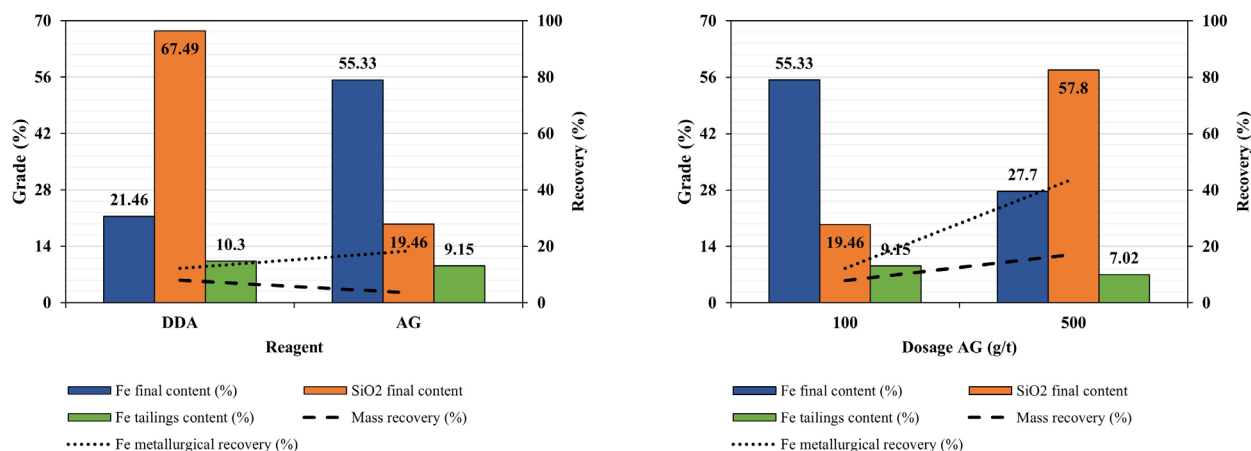


Figure 4. (a) Results of the tests with DDA and AG only. (b) Effects of changing the AG reagent dosage on Fe content and recovery.

Table 3. Chemical composition of the iron ore tailings sample

Size (µm)	Weight fraction (%)	Grade (%)				
		Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Mn	LOI
+ 212	10.21	3.35	95.30	0.11	0.01	0.35
212-150	13.70	4.63	92.80	0.07	0.01	0.18
150-106	29.84	5.07	93.00	0.07	0.01	0.40
106-75	13.48	6.18	91.50	0.07	0.01	0.30
75-53	13.09	9.17	87.00	0.04	0.01	0.29
53-45	3.13	13.60	81.00	0.12	0.01	0.23
45-30	11.03	36.90	46.90	0.27	0.01	0.44
30-9	4.70	29.20	51.90	0.83	0.02	1.17
-9	0.82	50.34	21.10	0.14	0.16	1.37
<b>Global</b>	<b>100,00</b>	<b>10,80</b>	<b>83,90</b>	<b>0,13</b>	<b>0,01</b>	<b>0,38</b>

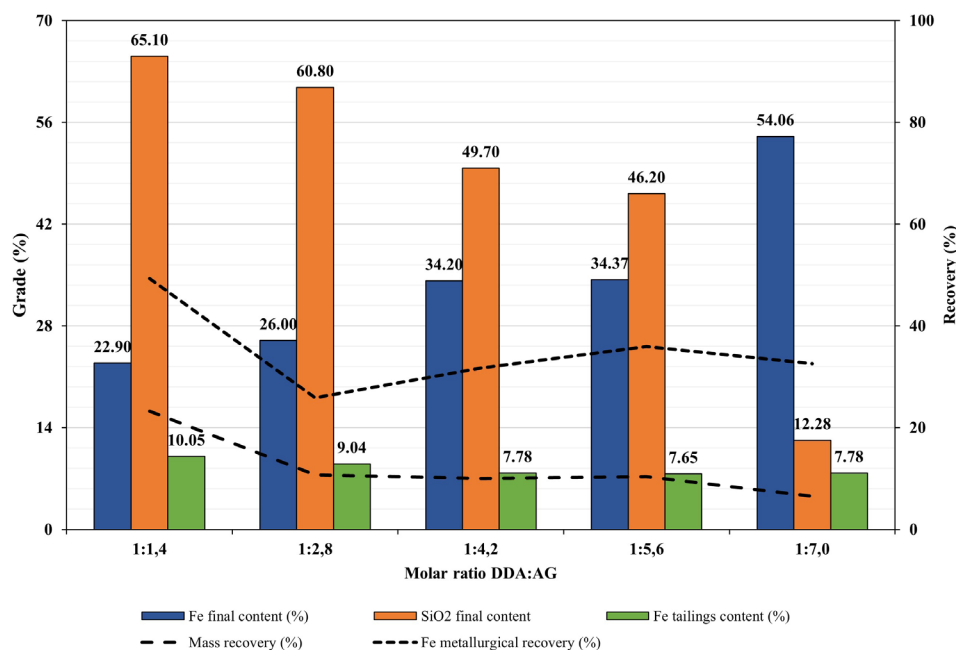
Table 4. Liberation analysis performed on the iron ore tailings sample

Mineral	Particle Size Classes			
	[150-106] µm	[106-75] µm	[75-53] µm	[53-38] µm
<b>Hematite</b>	36.46	71.20	60.95	94.09
<b>Quartz</b>	81.64	81.20	82.32	92.18

a drastic decrease in the Fe content of the final concentrate (55.13% to 27.70%), although there was a significant increase in mass (7.93% to 17.16%) and metallurgical recovery (12.19% to 44.02%). It should be noted that the tests using 500 g/t of AG reagent showed excess foaming. This may explain the lower selectivity shown with this dosage.

Figure 5 shows the results of the tests with the mixed collector at the analyzed molar ratios. The performance of





**Figure 5.** Results of the tests using mixed collectors at different molar ratios.

the mixed collector at a molar ratio of 1:1,4 was similar to the test performed with the DDA as a collector. However, the metallurgical recovery of Fe with the mixed collector (49.30%) was four times higher than that with the DDA reagent (12.19%). This suggests that the mixture of DDA and AG makes the mixed collector have a high collection efficiency, which might be explained by the lower repulsion between the ions of the monolayer, making it more compact and, therefore, more effective in collecting the minerals of interest [20]. Nevertheless, the high collection efficiency of the mixed collector at a molar ratio of 1:1.4 decreased its selectivity, with a recovery of SiO<sub>2</sub> of 18.04%, almost three times higher than that with the DDA (6.72%). This might be explained by the presence of DDA (a traditional silicate mineral collector) in the composition of the mixed collector.

When the molar ratio of AG was high in the composition of the mixed collector, the Fe content was also high: it reached 54.06% in the test where the molar ratio was 1:7.0. This value was slightly lower than the one obtained in the test with AG only. However, the metallurgical recovery of Fe obtained in the test with the mixed collector at a molar ratio of 1:7.0 was approximately 80% higher than in the test with AG only. It should be noted that intermediate DDA:AG ratios showed low selectivity among the minerals present, with high SiO<sub>2</sub> contents in the concentrate. This can be explained by the greater influence exerted by DDA, as it is in a higher proportion than the best result obtained (1:7.0). As for SiO<sub>2</sub> contamination in the concentrate, the results of the test with the mixed collector at a molar ratio of 1:7.0 were similar to the test with AG only (Figure 4).

Therefore, the mixed collector at a molar ratio of 1:7.0 can generate products with similar specifications to the AG reagent alone and in higher quantities, which makes

it an interesting alternative for iron ore tailings flotation. However, the results obtained with the mixed collector at a molar ratio of 1:7.0 were not ideal, since the amount of Fe that was discarded in the tailings was still relevant (after the flotation, 67.40% of the total Fe was discarded in the tailings).

Figure 6 shows the effects of increasing the dosage of the mixed collector at a molar ratio of 1:7.0. Increasing the dosage to 200 g/t did not yield better results than those obtained at a dosage of 100 g/t. There was an increase in mass recovery as predicted, causing an increase in Fe and SiO<sub>2</sub> metallurgical recovery. However, there was an abrupt decrease in Fe content in the final concentrate from 54.06% to 33.17%. Therefore, an increase in the dosage of the mixed collector at a molar ratio of 1:7.0 is not viable.

Considering that the coarse fraction (<150 μm) of the flotation feed contains 26.6% of the total SiO<sub>2</sub> in the sample and only 9.0% of the total Fe in the sample, a particle size cut was made at 150 μm with the aim of pre-concentrating this sample in order to obtain higher quality concentrates. Thus, the -150 μm sample has the following composition: 12.91% Fe; 81.22% SiO<sub>2</sub>; 0.14% Al<sub>2</sub>O<sub>3</sub>; 0.01% Mn and 0.42% LOI. Figure 7 shows the results of the tests with -150 μm sample. The results suggest that, by removing particles with a size greater than 150 μm from the flotation system, the quality of the final product increases significantly, with the final Fe content increasing from 54.06 to 64.04%. The SiO<sub>2</sub> content significantly decreased in the final concentrate (from 12.28% to 7.93%), and its metallurgical recovery decreased from 0.95% to 0.46%. This shows that the granulometric cut favored the selectivity between the minerals in the analyzed sample. However, there was a decrease in the mass and metallurgical recovery of Fe in the final concentrate.

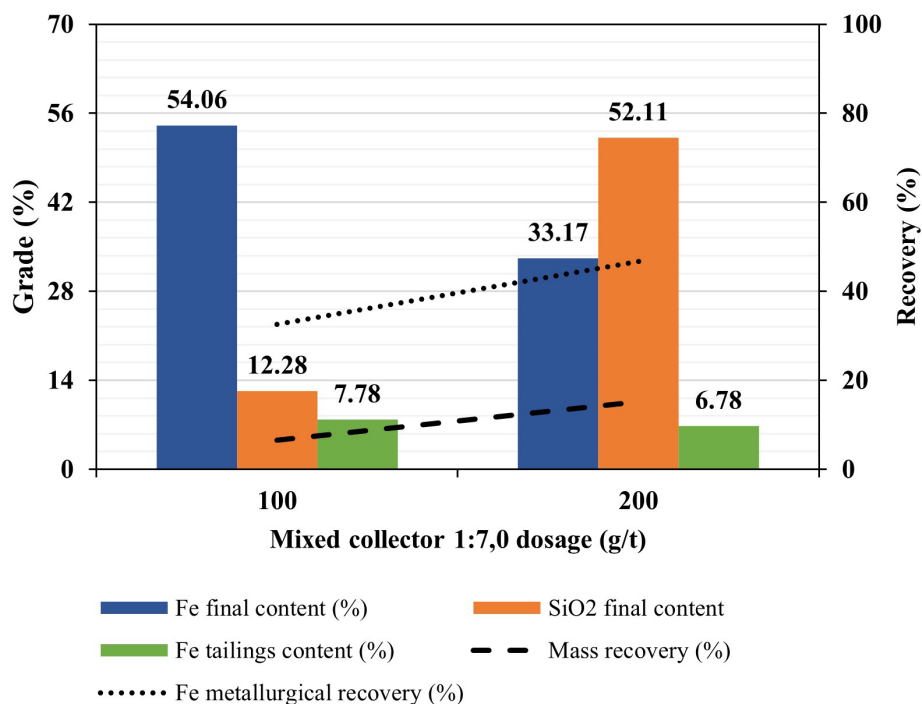


Figure 6. Effects of increasing the mixed collector dosage at a molar ratio of 1:7,0 on content and recovery.

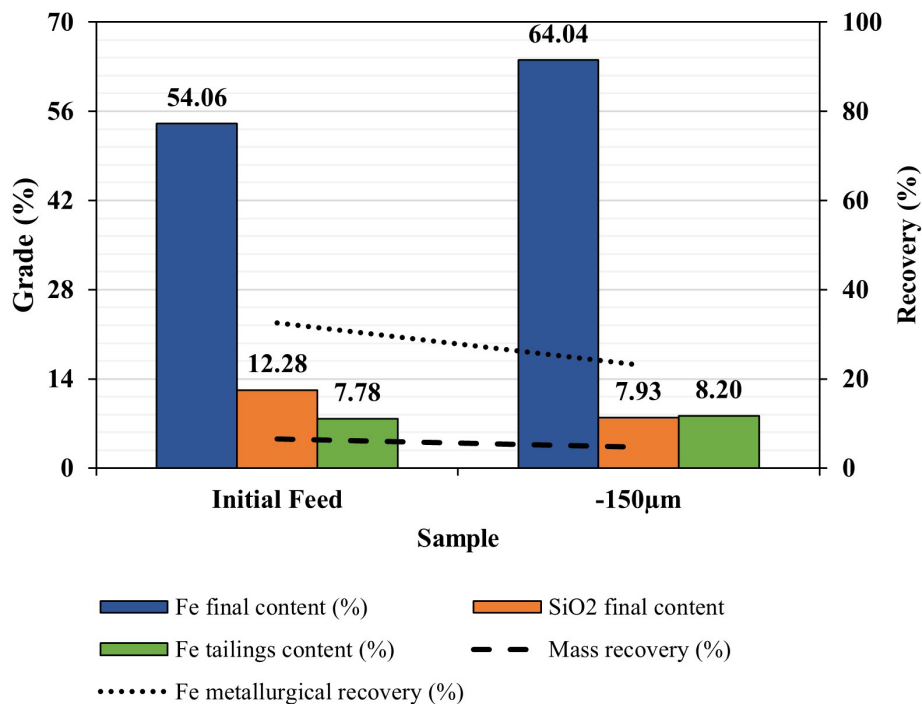


Figure 7. Effects of the 150 µm granulometric cut on the iron ore tailings sample.

#### 4 Conclusions

This study investigated the effects of using a mixed collector made of DDA and AG on the direct flotation of an iron ore tailings sample. The best results were obtained using a mixed collector at a molar ratio of 1:7.0, which obtained

54.06% of Fe and 32.60% of metallurgical recovery of Fe. Using only AG under the same operating conditions resulted in a slightly higher Fe content. However, the metallurgical recovery of Fe was 80% lower than that observed with the mixed collector at a molar ratio of 1:7.0. This suggests that the mixed collector has a greater collection efficiency and

selectivity. This aspect of the mixed collector is a consequence of the high degree of compaction of the monolayer, due to the lower electrostatic repulsion between ions of the same species, since the DDA positions itself between the AG ions. The increased dosage of the mixed collector at a molar ratio of 1:7.0 did not yield the expected results, since it reduced its selectivity. This points to another advantage of using mixed collectors: reducing the consumption of reagents in the flotation process.

The evaluation of the mass partition in the test with the mixed collector showed that coarser particles ( $> 150 \mu\text{m}$ )

were directed to the tailings. Therefore, a flotation test was performed only with particles  $< 150 \mu\text{m}$ , which reached 64.04% of Fe and 23.13% of metallurgical recovery of Fe. These results confirm the high efficiency of mixed collectors in the direct flotation of iron ore tailings.

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