

Evaluation of the effects of different hydroxamates and a sulfosuccinamate in the direct flotation of iron ore tailings

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Abstract

Recent events involving iron ore tailings dam collapses have emphasized the need for developing alternative ways of depositing tailings. This study aims to evaluate the effects of using three hydroxamates and a sulfosuccinamate as collectors in the direct flotation of iron ore tailings, using a bench flotation. The best result obtained on bench flotation was tested in a pneumatic cell. The sample used in this study (10.8% of Fe) consists of quartz and hematite. The Aero[®] 6494 Promoter hydroxamate showed the highest selectivity among the analyzed reagents, reaching 64.31% of Fe in the final product of the test with the < 150 µm sample. The possibility of using these reagents as constituents of a mixed collector with dodecylamine was also investigated. In this case, the Aero[®] 845 Promoter sulfosuccinamate yielded the best results, with 62.84% of Fe and 59.01% of metallurgical recovery of Fe. The synergistic effect between the two reagents might be explained by the stability of the monolayer formed by the intercalation of the ions from the dodecylamine and the Aero[®] 845 Promoter, which reduces the electrostatic repulsion between ions of the same species. This mixed collector was tested in a pneumatic cell. However, it had a lower performance in terms of Fe metallurgical recovery than in a bench-scale cell test, mainly for the finest particles, due to the greater control of the process's variables in the bench cell.

Keywords: iron ore tailings, direct flotation, hydroxamate, sulfosuccinamate, mixed collector, dodecylamine, pneumatic cell.

1. Introduction

The depletion of major mineral deposits, along with the increasing demand for industrial products, has led to the exponential growth of mining waste in recent years. Thus, the management of mining waste has become a major issue for the global society and has significantly increased financial, environmental, and social costs (Zhang *et al.*, 2011; Capasso *et al.*, 2019; Figueiredo *et al.*, 2021). In Brazil, this issue has gained notoriety after the collapse of the iron ore tailings dam

owned by mining companies Samarco (2015) and Vale (2019). About sixty million cubic meters of iron ore tailings were released into the surrounding area, causing irreparable environmental damage and the loss of about 300 human lives (Armstrong *et al.*, 2019; Pastran & Mallett, 2020; Silva Rotta *et al.*, 2020).

Several alternatives have been proposed to reduce the risks of iron ore tailings deposition, such as manufacturing geopolymers (Figueiredo *et al.*, 2021;

Ferreira *et al.*, 2022; Li *et al.*, 2022a) and reprocessing Fe-bearing minerals in iron ore tailings by anionic direct flotation. Fatty acids are commonly used as iron oxide collectors, and sodium hexametaphosphate is a reagent that is typically used for the depression of silica gangue. However, fatty acids can be replaced by hydroxamates, sulfosuccinates, or sulfosuccinamates.

Hydroxamates are considered chelating collectors, and their adsorption in

the mineral system is chemical. It may occur through chemisorption or chemical reaction through the formation of a precipitate on the solid-liquid interface. This precipitate can lead to excessive consumption of reagents and hinder the performance of the collector in the flotation system (Parker *et al.*, 2012). In general, the formation of chelates does not occur with Si^{4+} ions and alkali metals. The most stable chelates are formed with Fe^{3+} , Cu^{2+} , Nb^{5+} , and Ta^{5+} ions (Nagaraj, 1987; Marabini *et al.*, 2007). The adsorption of hydroxamates occurs at a wide range of pH values. The maximum hydrophobicity has been observed at a pH of 9, where the molecular and ionic species coexist, which leads to an increase in the mineral's adsorption density on the surface and floatability (Marabini *et al.*, 2007; Zhou *et al.*, 2015). Hydroxamates are used in the flotation of various minerals, such as manganese (Zhou *et al.*, 2015), titanium (Yoon *et al.*, 1992), niobium (Gibson *et al.*, 2015), tin (Wu & Zhu, 2006), copper

(Hope *et al.*, 2015), and iron (Raghavan & Fuerstenau, 1975), among others.

The sulfosuccinates and sulfosuccinamates are derived from succinic acid, featuring the sulfonate and carboxylate groups. Like hydroxamates, they can form chelates. Thus, their adsorption occurs through chemisorption. The maximum floatability occurs in a system with high acidity ($\text{pH} < 4$), since, under these conditions, the precipitates are soluble, which allows the arrival of a greater number of molecules from the collector to the mineral surface (Chen *et al.*, 2005). In the mining industry, sulfosuccinates and sulfosuccinamates are most used in the flotation of cassiterite (Angadi *et al.*, 2015; Leistner *et al.*, 2016).

Another way of improving the selectivity and reducing the total consumption of reagents in the flotation process is to produce mixtures of collector reagents before their use in flotation. The adsorption power of the mixed collector is greater than that of a single reagent collector since

the mixed collector covers both strong and weak sites (Bradshaw *et al.*, 1998). Mixed collectors can also form compact monolayers on the water/air interface, resulting in greater surface activity (Wang *et al.*, 2016a). Several authors have conducted research applying a mixture of dodecylamine and sodium oleate in different mineral systems, such as calcite/scheelite, muscovite, fluorite/calcite, manganese/quartz, among others (Bradshaw *et al.*, 1998; Wang *et al.*, 2016a; Wang *et al.*, 2016b; Xu *et al.*, 2016; Li *et al.*, 2022b).

Therefore, this study aimed to conduct tests on a laboratory scale to evaluate the possibility of using hydroxamates and sulfosuccinamate as collectors in the direct flotation of iron ore tailings, as well as to analyze the feasibility of using mixed collectors made of dodecylamine and the tested hydroxamates and sulfosuccinate. Finally, the best operational conditions obtained on a laboratory scale were replicated in a pneumatic cell to assess the replicability of the results.

2. Materials, Reagents, and Methods

The iron ore tailings samples were extracted from a decommissioned tailings dam in the state of Minas Gerais, Brazil. A preliminary flotation test was

conducted on this sample without the addition of any reagents to ensure that any residual amine adsorbed on the quartz surface would not impact the direct flo-

tation tests. In the preliminary test, there was no flotation of any particles. Fig. 1 shows the particle size distribution of the iron ore tailings sample.

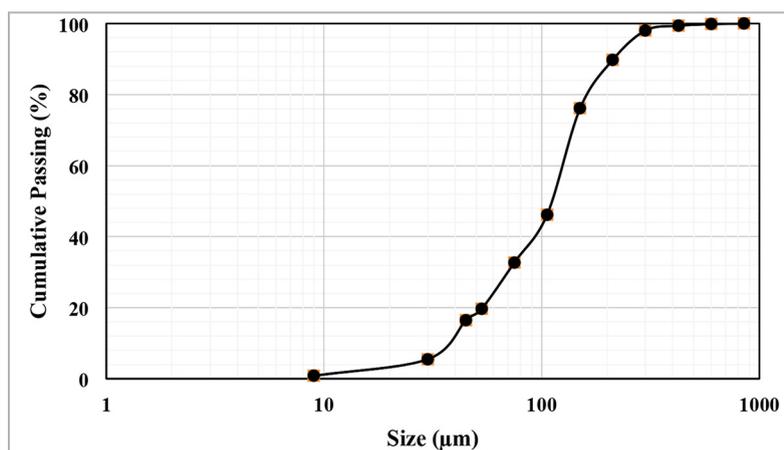


Figure 1 - Particle size distribution of the iron ore tailings sample.

The mineral distribution of the sample was assessed through X-ray diffractometry (XRD) using a Philips-PANalytical PQ3710 and a $\text{CuK}\alpha$ radiation and graphite monochromator (range $3\text{-}90^\circ 2\theta$, step $0.03^\circ 2\theta$, time per count 3 s). The phases were identified using the International Center for Diffraction Data (ICDD) PDF-2 and quantified using the Rietveld method. The chemical elements found in the iron ore tailings sample were quantified through X-ray fluorescence (XRF).

Aero® 6493 Promoter, Aero® 6494 Promoter, and Aero® OX 102 Promoter hydroxamates and an Aero® 845 Promoter sulfosuccinamate were used in this study – all provided by Solvay. Dodecylamine (DDA), in analytical grade, provided by Sigma Aldrich, was used for the composition of the mixed collector. Sodium hexametaphosphate, in analytical grade, provided by Synth, was used as a depressant for silicate minerals. The reagents were prepared by diluting them in water

to obtain a 1% w/v solution. The mixed collector was prepared by pre-mixing the DDA solutions and the tested hydroxamates and sulfosuccinamate. The molar ratio of DDA:collector used in the composition of the mixed collector was 1:7. This ratio was chosen due to preliminary results obtained by the author in other studies that are still in the process of being published.

The bench-scale flotation tests were conducted in the rougher and cleaner

stages. The flotation cell used was Denver's McDarma D12. A 1500 mL tank was used in the rougher stage and an 800 mL one was used in the cleaner stage. The use of tanks of different volumes is due to the need to maintain a similar percentage of solids in the cleaner stage. Reagent conditioning was performed at 50% of solids by mass, while flotation occurred at 35% of solids by mass in the rougher stage and no adjust of proportion of solids by mass in the cleaner stage. The chosen depressant was used at a dosage of 500 g/t and conditioned for 5 min. The chosen collector

was conditioned for 2 min, and the dosage was 100 g/t. The adopted dosages were defined according to preliminary tests carried out by the authors, where it was shown that the collector dosage cannot be higher than 100 g/t, as well as the depressor dosage does not need to be higher than 500 g/t, under penalty of reducing the Fe metallurgical recovery. No froth was used during the tests. There was no addition of reagents in the cleaner stage. NaOH and CH₃COOH solutions (analytical grade purity) at 1% w/v were used to adjust the pH level to 7.5±0.2.

The Pneufлот® pneumatic cell was used to conduct tests on a pilot scale to evaluate the replicability of the best test results obtained in bench-scale flotation. For this purpose, 15 kg of the sample were used for the experiment. The objective was to replicate the operating conditions of the bench-scale flotation test. The pneumatic cell test was conducted in a closed circuit, with the sunk material returning to the feed tank. In this way, the tests were performed only in a rougher stage, considering that it was carried out until complete exhaustion.

3. Results and Discussion

3.1 Bench-Scale Flotation Tests

Fig. 2 shows the diffractogram of the IOT sample, which consists of quartz (SiO₂, 85%) and hematite (Fe₂O₃, 15%). No other

minerals were detected, due to the intensity of the quartz peaks, the main component of the sample. Table 1 shows the results

of the chemical composition based on the XRF analysis. These results confirm the low Fe content of this iron ore tailings sample.

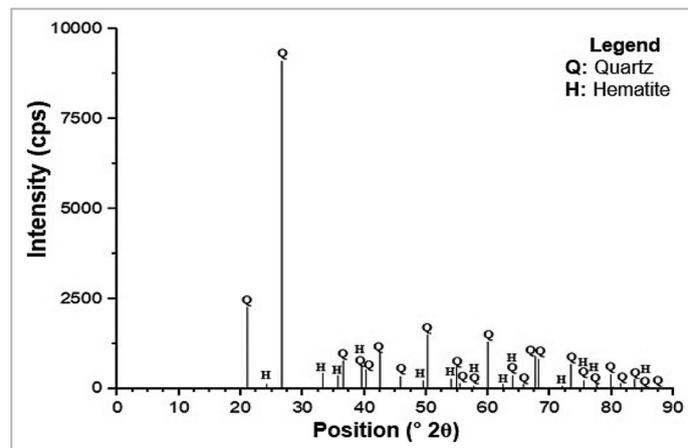


Figure 2 - Diffractogram of iron ore tailings sample.

Table 1 - Chemical composition of the iron ore tailings sample.

Size (µm)	Weight fraction (%)	Grade (%)				
		Fe	SiO ₂	Al ₂ O ₃	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
Global	100.00	10.80	83.90	0.13	0.01	0.38

Fig. 3 shows the results of the tests on the iron ore tailings sample with each hydroxamate and sulfosuccinamate being used in isolation in the flotation system. The graph indicates that all four reagents

had a satisfactory performance in the direct flotation of iron ore. However, Aero® 6494 Promoter had the best performance among the tested reagents, with 61.94% of Fe and 35.89% of metallurgical recovery

of Fe in the final concentrate. In contrast, Aero® OX 102 Promoter was the least selective reagent among them, with 53.09% of Fe and 50.57% metallurgical recovery of Fe in the final product.

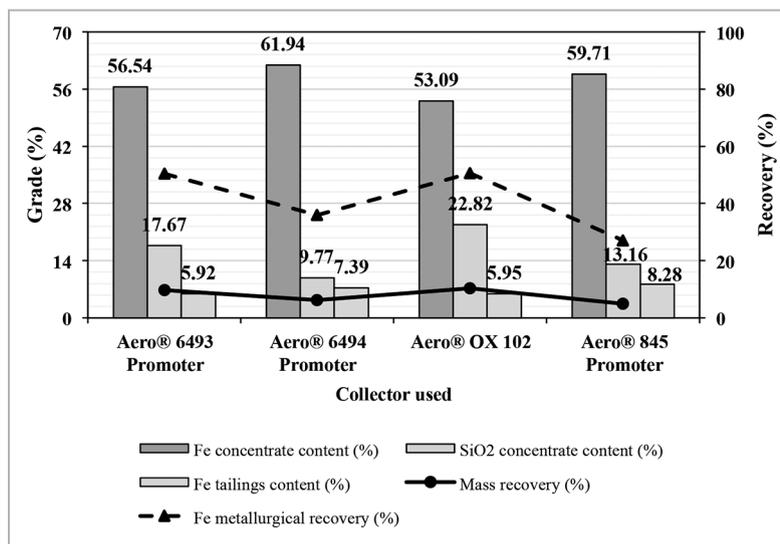


Figure 3 - Results of the tests using the hydroxamates and sulfosuccinate in isolation.

Fig. 4 shows the results of the tests carried out under the same operating conditions as the tests featured in Fig. 3, but, in this case, the feed was previously submitted to a 150 µm granulometric cut.

In terms of Fe content, all the reagents had a better performance compared with the tests with the initial sample (Fig. 3). This behavior was expected, since flotation is not as efficient with particles larger than

150 µm. It is worth noting that the test with the Aero® 6494 Promoter reagent resulted in 64.31% of Fe, which confirms its high selectivity in the direct flotation of iron ore.

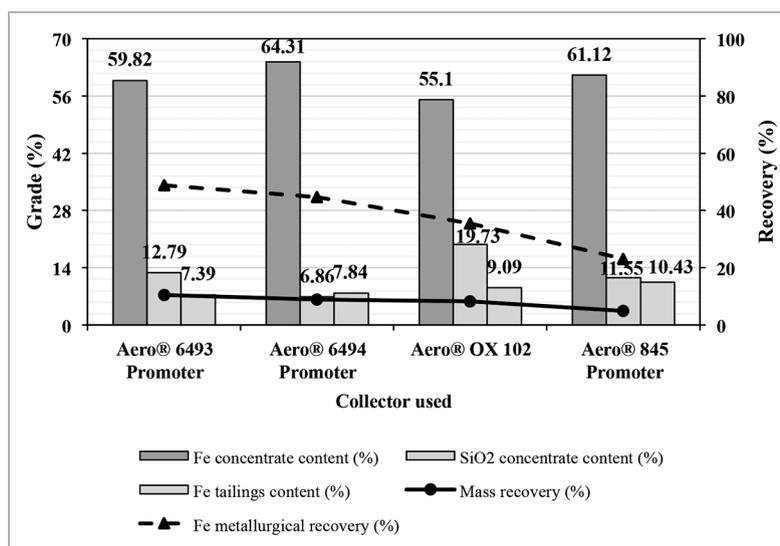


Figure 4 - Results of the test using the hydroxamates and sulfosuccinate in isolation, with the < 150 µm sample as the feed.

Fig. 5 shows the results of the tests using the mixed collector made of hydroxamates and sulfosuccinamate at a molar ratio of DDA:reagent of 1:7. The feed used in these tests was the < 150 µm sample. The results show that the mixed collectors made of hydroxamates did not have a satisfactory performance, with a drastic decrease in the selectivity of the iron oxides in the final product. The worst result was obtained with the mixed collector made with the Aero® 6494 Promoter, which resulted in only 26.89% of Fe and 25.50% of metallurgical recovery

of SiO₂ in the final product. This contrasts with the data described in Fig. 3 and Fig. 4, where the Aero® 6494 Promoter was the most selective reagent in terms of Fe-bearing minerals. Thus, the mixed collector made of DDA and hydroxamates did not have the expected synergistic effect to improve the quality of the final product.

In contrast, the Aero® 845 Promoter sulfosuccinate had the opposite effect at a molar ratio of 1:7. It resulted in 62.84% of Fe and 59.01% of Fe metallurgical recovery in the final product. Thus, it is significantly better

than sulfosuccinamate as a collector (which resulted in 61.12% of Fe and 23.13% of Fe metallurgical recovery). The mixed collector drastically increased the collection efficiency of the hematite and the quality of the resulting concentrate. This might be explained by the synergistic effect between the DDA and the Aero® 845 Promoter in the mixture, due to the stability of the monolayer formed by the intercalation of the ions of the two reagents, which reduces the electrostatic repulsion between ions of the same species (Wang *et al.*, 2016b).

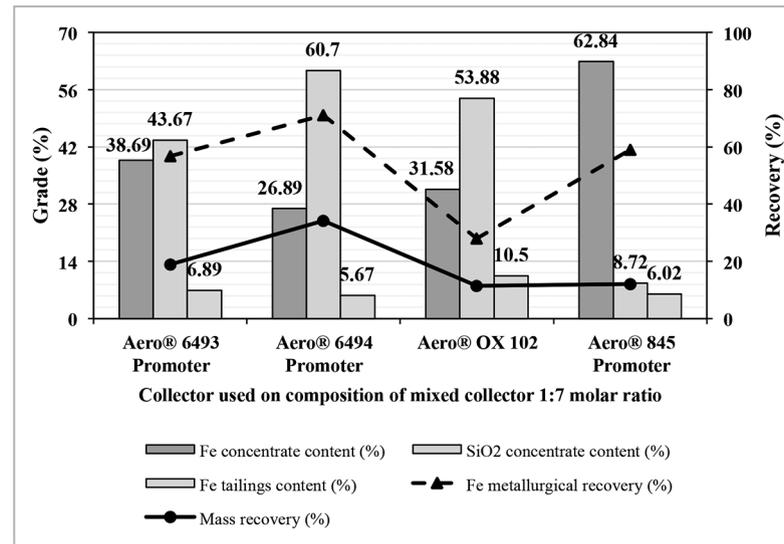


Figure 5 - Results of the tests using hydroxamates and sulfosuccinate in the mixed collector at a molar ratio of 1:7, with the < 150 μm sample as the feed.

Fig. 6 shows the best result obtained on the bench-scale flotation tests presented on Fig. 3,4 and 5, respectively. The best result when analyzing the Fe final content and the Fe metallurgical recovery is the test performed with the mixed collector

using DDA and Aero® 845 Promoter, using the <150 μm sample. This test showed the highest value of Fe metallurgical recovery (59.01%) compared to the others, despite not having obtained the highest Fe final content among the analyzed tests.

The justification for this is due to the lower loss of Fe-bearing minerals with the use of the mixed collector between DDA and Aero® 845 Promoter, with the lowest Fe content in the tailings among the tests analyzed (6.02%).

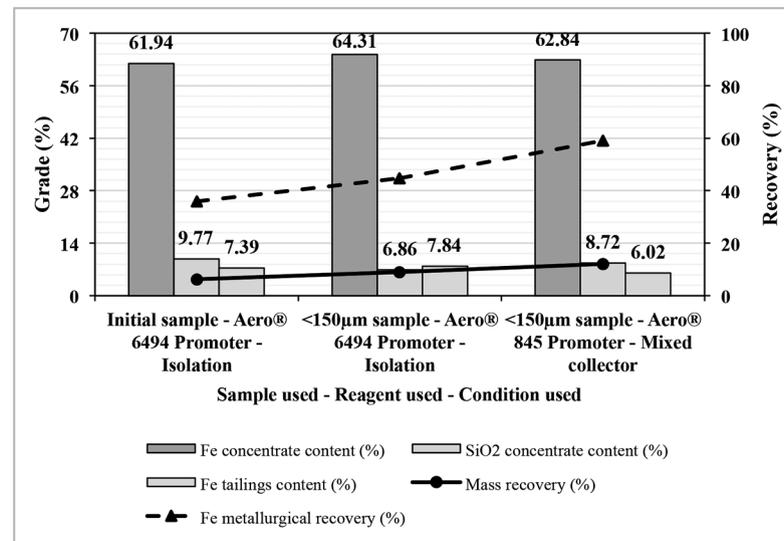


Figure 6 - Comparative with the best results on the bench-scale flotation tests.

3.2 Tests with the pneumatic cell

According to the results of the bench-scale flotation tests, the test performed with a mixed collector made of DDA and Aero® 845 Promoter was replicated in a pneumatic cell. Preliminary tests were carried out using 100 g/t of the mixed collector, in which the foam was not mineralized. The dosage was changed to 200 g/t under the same conditions. Then, a dosage of 400 g/t was used in the pneumatic cell. It is expected that the dosage of the reagents in pneumatic cell flotation is higher than in bench-scale

cell flotation.

Fig. 7 shows the cumulative results of the test with the pneumatic cell over flotation time. In the first 10 minutes, most of the mass had floated (68.66% of the total mass), which resulted in the highest final Fe content observed in this study (62.52% of Fe). As flotation continued, the concentrate's quality decreased, indicating that the selectivity presented by the mixed collector in the first minutes had decreased.

The results of the pneumatic cell test were lower than those of the bench-scale test in terms of final Fe content and metallurgical recovery, despite the mixed collector dosage being four times higher than that of the bench-scale cell. One explanation is that, since the test was carried out on a bench scale, the flotation system was more controlled and resulted in less loss. In pilot-scale equipment, such as the pneumatic cell, the efficiency of the test was lower, due to the nature of the process.

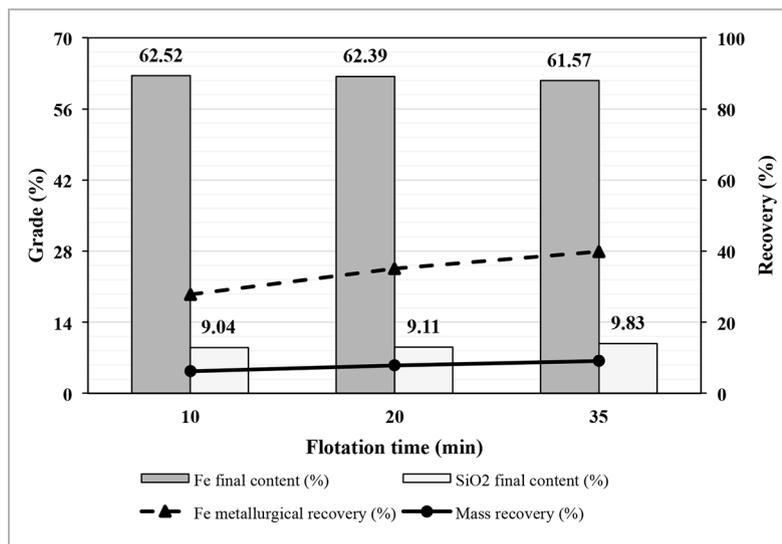


Figure 7 - Results of the test on a pneumatic cell using a mixed collector at a molar ratio of DDA and Aero® 845 Promoter of 1:7 and a dosage of 400 g/t.

Fig. 8 presents the Fe metallurgical recovery over particle size range in the tests performed on a pneumatic cell and a bench cell. The results show that the bench cell was more efficient in the flotation of fine particles compared with the pneumatic cell, which is illustrated by the steady decline in

the metallurgical recovery of Fe in the bench cell flotation test. The greater control of the process's variables in the bench cell test explains this behavior. With larger particles (> 106 μm), the results were similar, but the bench cell test had a lower metallurgical recovery of Fe (5.6%) than the pneumatic cell test

(7.0%). Despite the small difference, this result was expected, since, in the pneumatic cell, there is less turbulence inside the flotation cell compared with a bench flotation cell. This lower turbulence results in a greater probability of the particle/bubble aggregate reaching the surface and being collected.

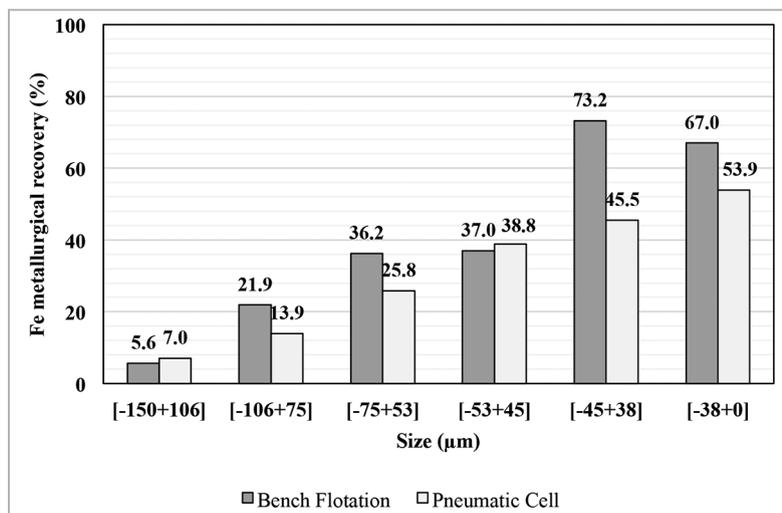


Figure 8 - Fe Metallurgical recovery of over particle size for each flotation equipment.

4. Conclusion

The effects of using different hydroxamates and sulfosuccinamate as collectors in the direct flotation of iron ore tailings were evaluated in this study. The results showed that all analyzed reagents had a satisfactory performance. The Aero® 6494 Promoter was the reagent that had the highest selectivity, for both the initial sample (61.94% of Fe) and the < 150 μm sample (64.31% of Fe).

However, when evaluating the

possibility of using these reagents as constituents of a mixed collector at a molar ratio of DDA:Reagent of 1:7, none of the tested hydroxamates presented the expected results, since the resulting Fe content was very low. Nevertheless, the Aero® 845 Promoter sulfosuccinamate, when used in the composition of the mixed collector, had a spectacular performance, with 62.84% of Fe and 59.01% of metallurgical recovery of Fe. This might be explained by the stability

of the monolayer formed by the intercalation of the ions from the DDA and the Aero® 845 Promoter, which reduces the electrostatic repulsion ions of the same species.

The test with the mixed collector made of DDA and Aero® 845 Promoter in a pneumatic cell had a lower performance than the test in a bench cell, especially in terms of the metallurgical recovery of Fe. In addition, it was necessary to quadruplicate the dosage of the

mixed collector in the pneumatic cell test to mineralize the foam. The lower efficiency of scale-up equipment, such as the pneumatic cell, compared with bench-scale cells, especially for the finest particles, is due to the greater control of the operational variables that exist in the bench cell, when compared to the pneumatic cell.

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