

# Mineralogical Characterization of Iron Ore Tailings from the Quadrilatero Ferrifero, Brazil, by Eletronic Quantitative Mineralogy

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Received: February 22, 2019; Revised: September 20, 2019; Accepted: October 02, 2019

The mineralogical characterization studies search for the best processing route, with the lowest environmental impact, aiming to improve the use of mineral resources. The electronic quantitative mineralogy (EQM) provides quickly and accurately great information about the characteristics of these materials. This work aims to characterize iron ore tailings by EQM as the main tool. It has selected seven samples of itabirite ores flotation tailings from the main mining regions of the Quadrilatero Ferrifero, Brazil. All samples were mostly composed by quartz and iron minerals, with a low presence of mixture particles of these minerals - less than 20% of the sample mass. Due to the difference between the size of particles of quartz and iron minerals, it has observed an opportunity to reprocess the fractions  $-37\mu\text{m} + 5\mu\text{m}$  of the studied tailings, with a potential recovery of 12% of the total sample mass generated for AM4, AM6 and AM7 and more than 5% for samples AM1, AM2, AM3 and AM5.

**Keywords:** *Electronic Quantitative Mineralogy, Mineralogical characterization, Iron ore tailings.*

## 1. Introduction

The studies about reprocessing mining tailings has been developed due to the great companies and society concerns about the environmental impacts generated by deposition of these materials in tailings dams, either using new technologies in the treatment of low-grade ores or in other applications. Those studies must invest in the characterization of these materials because they have not been applied to an environmental liability yet. Thus, such materials were arranged without further knowledge of their physical, chemical and mineralogical characteristics or even of their response to a possible reprocessing.

As applied in the current study, the quantitative electronic mineralogy technique (EQM) for the mineralogical characterization by automatic quantification of mineral phases, by image analysis, aims to determine and quantify the minerals present in a sample, differentiating the minerals with economic value (minerals-ore) and the non-economic minerals (gangue minerals). For the characterization, a sample could vary according to the mineralogy and the intrinsic properties of the ore, the specific searches, the period available for analysis, the processing routes and the available financial resources<sup>1</sup>.

The EQM uses software coupled to a modern scanning electron microscope (SEM), equipped with at least one energy dispersion spectroscopy (EDS) analyzer, for chemical microanalysis<sup>2</sup>. The system also uses backscattered electron (BSE) images, in which the gray level of each pixel is proportional to the average atomic number of that point<sup>1,3</sup>, in order to facilitate the composition information provided by the microanalysis and the the different mineral particles boundaries identification. Whether the minerals have elements with near or equal atomic numbers (e.g., spheralite and chalcopyrite) and gray levels indistinguishable from each other, the identification is based exclusively on energy dispersion spectra<sup>4</sup>.

The automated image analysis systems SEM- integrated, such as, TIMA (TESCAN Integrated Mineral Analyzer), MLA (Mineral Liberation Analyzer) and QEMSCAN (Qualitative Evaluation Minerals by Scanning Microscope), are actually tools for the application of the EQM technique.

Although EQM be a high complex apparatus and has an high initial investment cost, it is a fast and accurate quantitative analysis tool that provides reliable results of degree of release and characteristics of mineral associations, partition of the chemical elements of interest, mineralogical composition, particle size distribution, and potential mineral ore recovery<sup>5</sup>.

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In addition, EQM makes it possible to obtain information not available with other techniques, such as the frequency of particles per fraction for all mineral phases and the release spectra for all mineral phases.

In Brazil, several researches have been developed to take advantage of mining tailings in the manufacture of coproducts and to know deeply, the mineralogical characteristics of these materials is fundamental for these works accomplishment. It is a first step in assessing the technical and economic tailing feasibility. The characterization of itabirite ore flotation tailings from different Quadrilátero Ferrífero (QF) regions can provide valuable information for potential applications.

The QF is characterized by important iron ore reserves, which over time it have become gradually poorer due to the lower iron content itabirites<sup>6</sup>. With the gradual reduction of iron content, the minerals characterization studies become even more important in order to ensure the production of concentrates in the quality specifications required metallurgical process. Thus, new technological solutions are being developed to take advantage of these environmental liabilities in a sustainable manner for the purpose of minimize its accumulation in tailings dams<sup>7,8,9</sup>.

Iron ore tailings come from concentration processes such as concentrating spirals, magnetic separation and flotation. The flotation process is a heterogeneous mixture of particles suspended in an aqueous phase (pulp). It has used the differentiating property of surface reactivity, based on the fundamentals of physicochemical interfaces to separate minerals by dispersion in aqueous solution containing reagents that control their surface properties for effective separation. In practice, this happens by adding reagents to a ground ore pulp. Then, by dispersing air in the flotation machine, the particles of certain minerals attach to air bubbles and are carried by them to the foam layer, where they are collected as a foam rich in certain minerals<sup>10</sup>.

The floated material (tailings) is composed by gangue minerals, in its largest quartz mass, and the sunken (concentrated) by iron minerals. The inefficiency of the flotation process or the high variability of ROM (Run of Mine) may cause low metallurgical recovery<sup>11</sup>. According to Tolentino (2010), flotation tailing is made up on average of 13% iron oxide and the remainder is basically quartz. Considering 70% by mass of tailings has a particle size less than 149 $\mu\text{m}$ , its characteristic is similar to sand used in construction.

Figueiredo (2017) conducted a characterization study of silica sands and iron ore tailings with the intention of using it in foundry molding processes. From the analyzed materials, the tailings reached 93.35% quartz ( $\text{SiO}_2$ ) and 6.20% hematite ( $\text{Fe}_2\text{O}_3$ ), after processing. It was the material which presented the best potential use as base sand for the foundry industry.

Fontes, Mendes, Silva and Peixoto (2016) used iron ore dam tailings as a raw material for the production of mortars and coatings. The material used was characterized with 46% hematite and 24% quartz. Material thinner than natural aggregate, more than 50% of the passing mass at 75 $\mu\text{m}$ .

The results proved that the use of tailings is viable, if it does not exceed 20% of the mortar composition.

There are several studies on the utilization of the iron ore tailings. Therefore, the characterization of these tailings from different QF regions is fundamental to identify their potential applications, depending on their physical, chemical and mineralogical characteristics. Therefore, the use of EQM is recommended to the iron ore tailings characterization. So far, none scientific research has been carried out for the characterization of the main QF's iron ore tailings.

## 2. Materials and Methods

This study has characterized seven samples of iron ore tailings from the flotation process of the main processing plants located in different regions of the Quadrilátero Ferrífero, as shown in Figure 1.

Samples underwent dewatering, drying, homogenization and quartering processes; aliquots were prepared by the chemical analysis and EQM assays.

The quantitative chemical analysis of the samples was performed by X-ray fluorescence (XRF) in "Panalytical" Zetium model with melted samples. It has performed volumetric analysis in order to determine the FeO content. The LOI value was determined by the gravimetric method. XRF is an instrumental elemental chemical analysis technique widely used in geological materials (ore and tailings, for example). And this chemical analysis technique were used by these authors Tolentino (2010), Shimizu (2012), Fontes, Mendes, Silva and Peixoto (2016), Figueiredo (2017).

The system used by EQM was the TIMA software version 1.5.24, associated to the SEM / FEG model MIRA3 LMH, with excitation energy for the electron beam of 25kV, beam diameter of 70nm, magnification of 180x, image resolution of 3 $\mu\text{m}$  and average of 200.000 particles analyzed per tailings sample. Regarding the tests, the samples were embedded in epoxy resin, in the form of pellets with a diameter of 30mm, polished and covered with carbon film. The time analysis for each pellet was on average of 90 minutes.

According to Sampaio (2016)<sup>13</sup>, TIMA presents difficulties to distinguish ferrous mineral content in the very near oxygen, such as magnetite and hematite, and he suggests classifying them together as iron oxides. In this paper, it was decided to characterize a single phase encompassing the hematite and magnetite, "hematite/magnetite".

According to Rodrigues (2016)<sup>14</sup>, it is very rare to find monominerals particles of quartz in the QF iron mineralizations and there will be at least one inclusion of hematite, magnetite or goethite grain, however small ones. Thus, the particle released consists of more than 90% of the interest mineral in its composition.

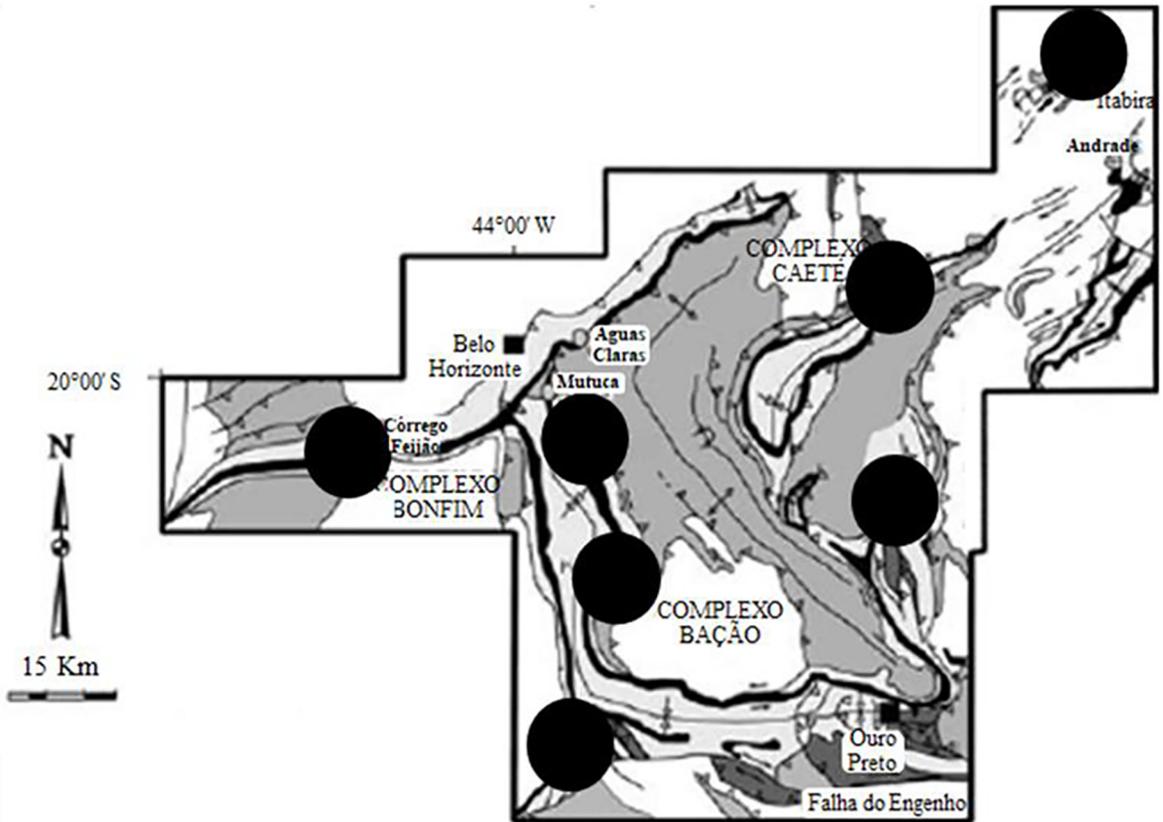


Figure 1. Samples location on the Quadrilatero Ferrifero (AM1 - AM7).

(Adaptated to Dorr, 1969 *apud* Rosière, 1993b)<sup>12</sup>

### 3. Results and Discussion

The samples overall chemical composition obtained by X-ray fluorescence (XRF) and volumetry is shown in Table 1, along with its loss on ignition.

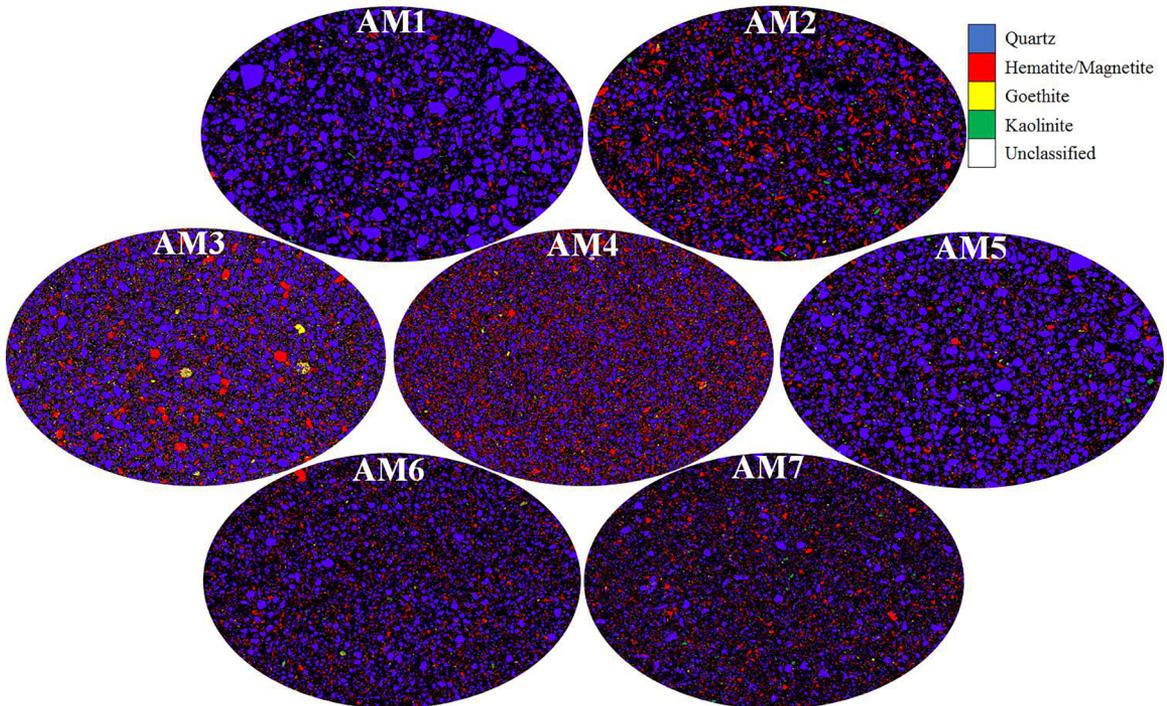
Samples AM1 and AM5 present higher SiO<sub>2</sub> content (respectively, 81.57% and 79.36%). The highest Fe content is present in samples AM4 (32.77%) and the lowest in AM1 and AM5, with 11.40% and 12.70%, respectively. Once they are samples of iron ore tailings, the other elements and compounds present relatively low values. The Al<sub>2</sub>O<sub>3</sub> content in AM2, 2.01%, the value of LOI in AM3, 2.63% and the Mn content in AM6, 0.128% are mainly outstanding.

The flotation tailings samples from different regions of Quadrilatero Ferrifero, present different characteristics of size, grain shape and distribution of mineral phases, although they are composed mostly by hematite/magnetite and quartz. The phase map obtained by EQM, allows visualization of individual characteristics of the particles present in the samples, as shown in Figure 2.

Figure 2 shows how the samples are different: the samples AM1 and AM5 have the largest sizes of quartz particles, AM5 having the rounded grains. AM2 has a larger amount of thicker hematite/magnetite, also this particles are more elongated. AM3 presents particles varying in size and shape, with thicker goethite material, thick and fine quartz and also hematite / magnetite.

Table 1. Chemical composition of the samples by XRF and volumetry.

Content (%)	AM1	AM2	AM3	AM4	AM5	AM6	AM7
Fe	11.40	27.38	24.98	32.77	12.70	23.86	26.75
SiO <sub>2</sub>	81.57	57.36	60.16	51.31	79.36	64.23	57.84
Al <sub>2</sub> O <sub>3</sub>	0.41	2.01	0.76	0.90	0.82	0.50	1.26
Mn	0.018	0.074	0.047	0.018	0.016	0.128	0.017
P	0.008	0.036	0.056	0.027	0.022	0.020	0.008
MgO	0.04	0.05	0.07	0.10	0.00	0.18	0.06
LOI	0.10	1.64	2.63	0.64	1.47	0.57	0.83



**Figure 2.** Map of mineral phases per samples by EQM (1 : 500 $\mu$ m).

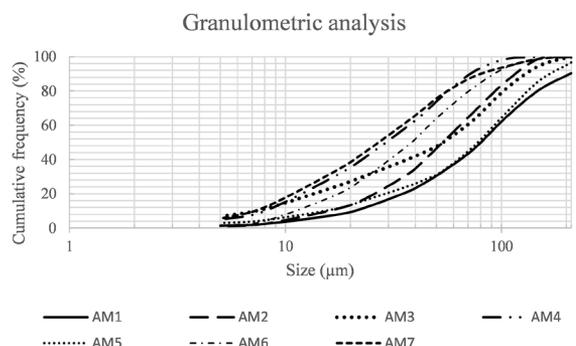
AM4 exhibits a greater homogeneity in the particle size distribution and large amount of finer iron-bearing particles. AM6 and AM7 have finer hematite/magnetite particles and the coarsest particles are predominantly quartz.

Quartz is the predominant mineral in all samples, except for sample AM4, consisting of approximately 47% of hematite/magnetite and 43% of quartz. The other samples present more than 50% in mass of quartz. The samples AM1 and AM5 are the ones with the highest participation of quartz, 83% and 75%, respectively (Table 2).

The AM3 and AM4 samples stand out for the larger compositions of goethite. Minerals unclassified have higher stakes in AM3 and AM7, around 3%. It's important to highlight that the analyzes with less than 5% of unclassified phases are considered reliable ones.

Knowing the particle size analysis, it is possible to verify the high amount of particles having less than 37 $\mu$ m size and AM4 in this AM7 samples, around 60%, and 10% below 8 $\mu$ m.

AM3 also presents 10% of its particles smaller than 8 $\mu$ m, while AM1 and AM2 have only 2% and AM5 and AM6, 4%. The AM1 and AM5 samples present the larger mass of coarse particles, comparing to the other samples with 20% and 14% on the mass 149 $\mu$ m, respectively (Figure 3).



**Figure 3.** Samples granulometric analysis by EQM.

**Table 2.** Samples mineralogical composition by EQM.

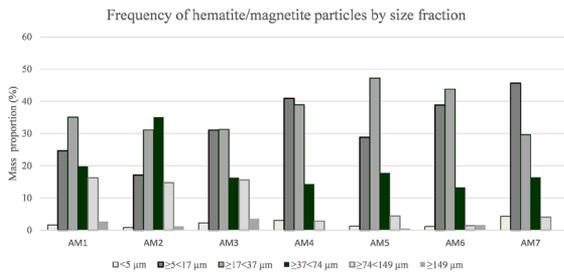
Mineralogical composition (%)	AM1	AM2	AM3	AM4	AM5	AM6	AM6
Quartz	82.66	53.70	54.44	42.62	75.08	60.73	50.05
Hematite/magnetite	14.50	42.04	28.91	46.69	18.21	33.95	39.47
Goethite	0.90	1.96	12.62	7.09	4.02	2.83	4.55
Kaolinite	0.36	0.67	0.12	0.54	0.66	0.31	1.81
Unclassified	0.83	1.20	2.80	2.32	1.71	1.50	3.02
Other minerals	0.75	0.43	1.11	0.74	0.32	0.68	1.10

The cumulative size distribution of samples by EQM shows the sample  $D_{50}$  range between 25 $\mu\text{m}$  and 80 $\mu\text{m}$ . AM4 and AM7 with finer particles and AM1 and AM5 with thicker particles. The distribution curves of AM1 and AM5 are very similar, even though they are samples from different regions of the Quadrilatero Ferrifero, as well as AM4 and AM7 (Figure 3).

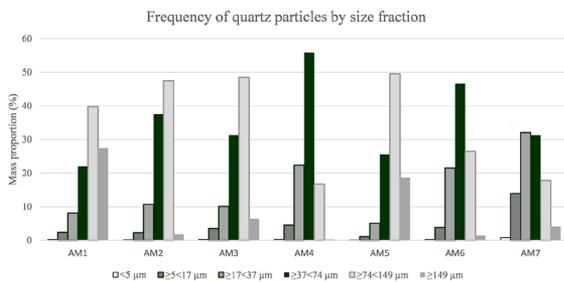
The frequency of the hematite/magnetite particles by size fraction, by EQM, is shown in the graph of Figure 4.

Most of the hematite/magnetite particles have sizes between 5 $\mu\text{m}$  and 37 $\mu\text{m}$ , with AM4 and AM6 having about 80% of their particles in this size range. AM5 has more than 45% of the mass of these particles with sizes between 17 $\mu\text{m}$  and 37 $\mu\text{m}$ . AM1, AM2 and AM3 have about 15% of the mass of this phase with a size between 74 $\mu\text{m}$  and 149 $\mu\text{m}$ . Since AM1 and AM3 are the thickest samples with ~20% of the mass greater than 74 $\mu\text{m}$ . AM7 is the sample with the finest hematite / magnetite particles, approximately 50% of mass below 17 $\mu\text{m}$ . While AM2 presents the largest mass of coarser particles in this phase, about 50% above 37 $\mu\text{m}$ .

For being samples of the iron ore tailings, it is must also characterize the quartz present in the sample (Figure 5).



**Figure 4.** Frequency of hematite/magnetite particles by size fraction of the samples by EQM.

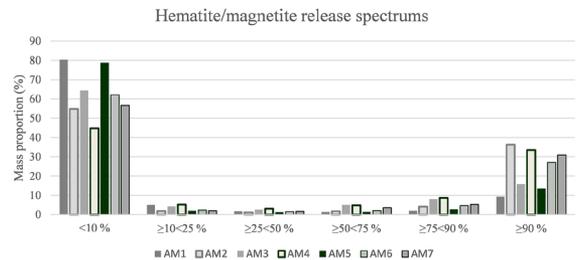


**Figure 5.** Frequency of quartz particles by size fraction of the samples by EQM.

The quartz inside in the samples with larger particle sizes, presents the greater mass in the range of 17 to 149 $\mu\text{m}$ . AM1 and AM5 present around 27% and 19% of the mass of their particles with a size superior to 149 $\mu\text{m}$ , respectively. They are the samples that present thicker quartz, about 70% above 74 $\mu\text{m}$ . AM7 stands out for the presence of 15% of these particles with size inferior to 17 $\mu\text{m}$ , whereas in the other samples they do not reach 5% of their masses.

AM2, AM3 and AM5 have about 50% of particles in the size range between 74 and 149 $\mu\text{m}$ . While AM4 and AM6 present 56% and 47% of the quartz mass in the range of 17 to 37 $\mu\text{m}$ , respectively. AM4 is the sample that has the largest mass of fine quartz particles, more than 60% below 37 $\mu\text{m}$ .

The mineral release analysis performed by EQM is shown in Figure 6 by means of the distribution of the hematite/magnetite particles in release classes, or rather the hematite/magnetite release spectrum.



**Figure 6.** Hematite/magnetite release spectrums of the samples by EQM.

The hematite / magnetite release spectrums show the low presence of mixture particles in all samples, less than 20%. Thus, the mixture particles are in the release classes greater than 10% and less than 90%. While the released particles present more than 90% of the mineral of interest, the particles with less than 10% of the mineral of interest are particles of other minerals. In this study, the samples are constituted basically by hematite/magnetite and quartz, so the particles with less than 10% hematite / magnetite in their composition are quartz particles. The samples AM2 and AM4 are the ones with the highest percentage of mass of hematite / magnetite particles released, around 35%. The samples AM1 and AM5 present approximately 80% of their mass corresponding to quartz particles and AM4 is the only one that has less than 50% of this mineral. AM1, in addition has the least amount of hematite / magnetite released (less than 10%) is the one with the lowest mass of this mineral phase in its composition, 14.5%.

As samples are composed by small mixture particulates and fine particles of hematite/magnetite, most of which are released, it is possible to predict the recovery potential of iron in the tailings samples by the EQM characterization study. Table 3 presents the mass recovery and metallurgical recovery for each sample, by means of a particle size separation process, in the granulometric range between 5 $\mu\text{m}$  and 37 $\mu\text{m}$ .

The iron content in the fraction  $-37\mu\text{m} + 5\mu\text{m}$  of samples AM1, AM2, AM3, AM4, AM5, AM6 and AM7 were 41.35%, 29.96%, 35.40%, 33.88%, and 44.45 %, 33.99% and 26.56%, respectively. The mass of goethite increases considerably in the finer fractions, smaller than 37 $\mu\text{m}$ . In the fraction smaller than 5 $\mu\text{m}$ , goethite represents more than 80% of the mass in all the samples.

**Table 3.** Potential mass and metallurgical recovery samples by EQM.

Sample	(%)							
	Size distribution	Mass recovery	Quartz	Hematite/magnetite	Goethite	Others minerals	Fe content	Metallurgical recovery
AM1	+37 $\mu$ m	78.67	91.73	6.99	0.77	0.52	4.45	36.30
	-37+5 $\mu$ m	20.04	11.97	67.63	11.80	8.60	41.35	85.98
	-5 $\mu$ m	1.29	2.04	11.92	82.36	3.69	46.31	6.20
	Global	100.00	82.66	14.50	0.79	2.05	9.64	-
AM2	+37 $\mu$ m	68.41	66.09	30.38	1.68	1.85	20.18	48.84
	-37+5 $\mu$ m	30.66	14.11	52.31	28.11	5.46	29.96	32.50
	-5 $\mu$ m	0.93	2.34	12.14	83.29	2.23	46.81	1.54
	Global	100.00	53.70	42.04	1.73	2.53	28.27	-
AM3	+37 $\mu$ m	59.76	72.42	15.83	8.92	2.83	9.10	32.63
	-37+5 $\mu$ m	33.43	13.27	60.98	18.58	7.17	35.40	71.00
	-5 $\mu$ m	6.81	1.15	8.69	84.24	5.92	48.60	19.86
	Global	100.00	54.44	28.91	9.66	6.99	16.67	-
AM4	+37 $\mu$ m	40.35	74.66	19.17	5.49	0.67	11.19	15.30
	-37+5 $\mu$ m	54.77	19.67	58.54	18.58	3.20	33.88	62.91
	-5 $\mu$ m	4.88	0.76	8.02	90.19	1.02	52.55	8.70
	Global	100.00	42.62	46.69	5.66	5.03	29.49	-
AM5	+37 $\mu$ m	75.57	88.36	5.18	2.89	3.56	2.98	20.12
	-37+5 $\mu$ m	21.50	5.98	73.76	14.97	5.29	44.45	85.50
	-5 $\mu$ m	2.93	0.90	11.20	81.18	6.72	45.82	12.01
	Global	100.00	75.08	18.21	3.05	3.66	11.18	-
AM6	+37 $\mu$ m	51.66	85.14	10.36	2.07	2.43	6.25	14.42
	-37+5 $\mu$ m	46.73	18.03	58.99	19.88	3.09	33.99	70.89
	-5 $\mu$ m	1.61	1.09	5.14	93.09	0.68	55.68	4.00
	Global	100.00	60.73	33.95	2.13	3.19	22.41	-
AM7	+37 $\mu$ m	37.39	70.38	21.39	3.45	4.77	13.17	19.30
	-37+5 $\mu$ m	57.17	28.48	46.59	19.96	4.97	26.56	59.49
	-5 $\mu$ m	5.44	2.37	11.80	83.73	2.10	47.18	10.05
	Global	100.00	50.05	39.47	3.49	6.99	25.53	-

The potential gains with the mass recoveries in each sample are quite different, depending on the mass in the fraction -37 $\mu$ m+5 $\mu$ m. AM4 and AM7 show the largest gains in mass, with approximately 55%. Its metallurgical recoveries are around 60%, with 33.88% iron content in AM4 and 26.57% iron content in AM7. AM1 and AM5, although presenting the lowest mass recoveries, approximately 20%, have the highest metallurgical recoveries around 85% and iron content around 41% and 44%, respectively.

After particles separation in the fraction -37 $\mu$ m + 5 $\mu$ m, a concentration process was simulated considering the feed with the mass of this fraction. Table 4 presents the simulation results of a possible concentration process.

The simulation presented considered iron content of 66% and metallurgical recovery of 70% for all samples. These parameters were considered to obtain a sub-product in the pellet feed fraction. The results show an opportunity for reprocessing of the -37 $\mu$ m + 5 $\mu$ m fraction of the studied

**Table 4.** Simulation of a possible concentration process.

Simulation of concentration process (-37+5 $\mu$ m)	AM1	AM2	AM3	AM4	AM5	AM6	AM7
	(%)						
Fe content (feed)	41.35	29.96	35.40	33.88	44.45	33.99	26.56
Fe content (concentrate)	66.00	66.00	66.00	66.00	66.00	66.00	66.00
Metallurgical rec.	70.0	70.0	70.0	70.0	70.0	70.0	70.0
Mass rec. (released particles)	28.4	24.7	17.4	24.6	33.1	30.0	22.4
Total tailings mass recovery	5.7	7.6	6.0	13.5	7.1	14.0	12.8

tailings, with reuse around 12% of the total tailing mass generated for samples AM4, AM6 and AM7 and more than 5% for samples AM1, AM2, AM3 and AM5.

#### 4. Conclusion

All samples were mostly composed by quartz and hematite/magnetite, with a low presence of mixture particles these minerals, less than 20% of the sample mass. The  $D_{50}$  of the samples range from 25 $\mu\text{m}$  and 80 $\mu\text{m}$ . The coarsest fraction (+37 $\mu\text{m}$ ) is basically constituted by liberated particles of quartz. The samples superfine fraction (-37 $\mu\text{m}$ ) presented bigger mineralogical variability, with great presence of iron-bearing minerals mass. The fraction concentration process simulation (-37 $\mu\text{m}$  + 5 $\mu\text{m}$ ) showed a reprocessing opportunity of these tailings, which can lead to use around 12% mass of tailing generated in AM4, AM6 and AM7 and 5% in AM1, AM2, AM3 and AM5.

#### 5. Acknowledgments

The authors thank FAPEMIG (Minas Gerais Research Support Foundation), Gorceix Foundation and the REDEMAT (Graduate Program on Thematic Network in Materials Engineering) of UFOP/UEMG (Federal University of Ouro Preto/State University of Minas Gerais) for the encouragement and support to realize this study.

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