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# THERMOMAGNETIC STUDY OF IRON ORE TAILINGS FROM QUADRILÁTERO FERRÍFERO - MINAS GERAIS/BRASIL

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**ABSTRACT** Within the international macro-economic scenario, the mineral sector is seeking innovative and sustainable solutions for their productive processes. However, to be able to direct the tailings toward a determined technology, it is fundamental to know its characteristics and specific properties. The objective of the study presented herein was to extract significant information regarding the various ranges of particle size existing within the tailings from iron ore processing, with special attention to its magnetic properties. For this were used different technical characterization. The results demonstrate that the granulometric distributions of the samples vary from 100µm to values of less than 4µm, with a significant presence of iron oxides and quartz. The thermomagnetic curves generated permitted identification of the Hopkinson Effect, Curie temperatures for magnetite and hematite. They also show the magnetic susceptibility of each grain size range, an important information when considering magnetic separation.

**KEYWORDS :** mineral processing tailings, magnetic susceptibility, technological characterization

## INTRODUCTION

During decades, iron ore processing has generated solid wastes with a significant iron content. Due to limitations of the current processing technologies and the inexistence of feasible economic solutions for the usage of these residues, they are deposited in piles or tailings dams. According to FEAM (2013), Minas Gerais, Brazil has 706 registered dams, of which 340 are iron ore tailings dams that result in a significant environmental impact, not to mention all of the risks associated with the dams.

To mitigate this impact, it is important to seek new methodologies with the application of technical solutions for a cleaner production, identifying each process phase that generates residues. In addition, technologies need to be determined for the recycling of these residuals, be it in the productive system itself or in a diverse productive system, establishing a concept for sustainable mining with zero residues for the greater benefit of environment and economy.

The physical and chemical characteristics of the tailings depend on the type of ore and its processing. Basically, the itabiritic iron ore waste deposited in tailings dams are a sum of two types of material from different phases of the treating process: one from the desliming phase (removal of very fine particles that are undesirable for the concentration process) and the other from the concentration phase (rich in quartz).

Desliming is a normal phase in iron ore treatment, due to the fact that ultrafine particles provoke a reduction in the efficiency and selectivity of the concentration operation. Sivamohan (1990) made a complete study about the recuperation of ultrafine particles in mineral processing and affirms that the problem is a consequence of small mass with high specific surface.

## MATERIALS AND METHODS

The methodology used for this study involved the collection and preparation of samples of tailings originating from different plants that process itabiritic iron ore. This was followed by technological characterization and thermomagnetic tests for different granulometric ranges. The samples used were collected from two types of residuals produced in mineral processing plants in the Quadrilátero Ferrífero - Minas Gerais with the first being from a concentration by flotation process, and the second being the sludge from a desliming process (ultrafines).

The thermomagnetic curves were generated by a Bartington MS3 magnetic susceptibility meter. Measurements were taken for temperatures varying from room temperature up to 720°C and then returned to room temperature, where the heating and cooling rate were 15°C/min.

## RESULTS AND DISCUSSIONS

The Figure 1 presents the granulometric distributions of the samples, obtained by wet sieving and cyclosizer. The values of d80 and the slime percent (fractions of less than 10µm) were, respectively, 18µm and 62% for ultrafine tailings, 70µm and 15% for flotation tailings.

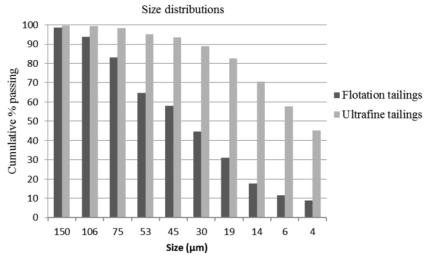


**Figure 1: Particle size distribution**

The Tables 1 and 2 display the global chemical compositions and by grain size range of the samples. Notice the increase in the amount of iron contained in the fractions below 45µm, while there is a predominance of quartz in the fractions above this value. Contaminants, such as alumina and phosphorus are concentrated in the fraction below 4µm. Loss on ignition showed the greatest values below 4µm, indicating the presence of iron hydroxides in this range.

Flotation tailings present 23.7% in weight of iron in its global chemical composition, 64.85% of quartz, 0.55% of alumina and 0.25% of phosphorous. However, in the interval between 15µm and 4µm, there is an average iron content of 53.6%, 19.9% of quartz, 1.19% of alumina and 0.04% of phosphorous (Table 1).

The global composition of the ultrafine tailings has 44.4% iron in weight, 21.05% quartz, 5.88% alumina, 0.142% phosphorous. In the interval between 14µm and 4µm, there is an average of 55.74% iron, 30.81% quartz, 1.33% alumina, and 0.061% phosphorous (Table 2). Tables 1 and 2 demonstrate that contaminants, such as phosphorous and alumina, have high concentrations in the ranges below 4µm.

The results of the stratified chemical analysis demonstrate that, for both tailings, the range between 15µm and 4µm contains material with an iron content of more than 53% and a high degree of liberation of the minerals. However, the behavior and the characteristics of the ultrafine particles become obstacles for feeding in the conventional concentration processes.

**TABLE – 1: Chemical Analysis By Size – Flotation Tailings**

| Size (µm) | Fe % | $SiO_2$ % | $Al_2O_3$ % | Mn % | P % | CaO % | MgO % | $TiO_2$ % | $K_2O$ % | $Cr_2O_3$ % | LOI % |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Global | 23.76 | 64.85 | 0.55 | 0.125 | 0.025 | 0.07 | 0.15 | 0.093 | <0.01 | <0.01 | - |
| + 150 | 3336 | 94.78 | 0.26 | 0.038 | 0.008 | 0.12 | <0.10 | 0.021 | 0.011 | <0.01 | - |
| - 150 + 106 | 1548 | 97.6 | 0.14 | 0.014 | 0.007 | 0.049 | <0.10 | 0.011 | <0.01 | <0.01 | - |
| - 106 +75 | 2685 | 96 | 0.17 | 0.021 | 0.007 | 0.039 | <0.10 | 0.03 | <0.01 | <0.01 | - |
| - 75 +53 | 6073 | 91.07 | 0.25 | 0.027 | 0.011 | 0.043 | <0.10 | 0.04 | <0.010 | <0.01 | 0.06 |
| -53 +45 | 10.71 | 83.71 | 0.32 | 0.045 | 0.012 | 0.082 | 0.11 | 0.067 | <0.01 | <0.01 | 0.14 |
| -45 +30 | 34.43 | 49.91 | 0.28 | 0.073 | 0.017 | 0.069 | <0.010 | 0.11 | <0.01 | <0.01 | 0.15 |
| -33 +21 | 28.28 | 58.31 | 0.39 | 0.078 | 0.023 | 0.075 | <0.10 | 0.103 | <0.01 | <0.01 | 0.26 |
| -21 +15 | 39.69 | 41.35 | 0.57 | 0.107 | 0.022 | 0.094 | 0.16 | 0.134 | <0.01 | 0.011 | 0.48 |
| -15 +7 | 51805 | 23.47 | 0.96 | 0.187 | 0.03 | 0.142 | 0.36 | 0.171 | 0.018 | 0.011 | 0.78 |
| -7 +4 | 55.5 | 16.34 | 1.42 | 0.28 | 0.051 | 0.28 | 0.65 | 0.237 | 0.032 | 0.013 | 1.3 |
| -4 | 39.95 | 24.17 | 7.99 | 1642 | 0.16 | 0.2 | 1.14 | 0.28 | 0.116 | 0.062 | 6.09 |

**TABLE – 2: Chemical Analysis By Size – Ultrafine Tailings**

Chemical Analysis by size: ultrafine tailing.

| Size (µm) | Fe % | $SiO_2$ % | $Al_2O_3$ % | Mn % | P % | CaO % | MgO % | $TiO_2$ % | $K_2O$ % | $Cr_2O_3$ % | LOI % |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Global | 44.4 | 21.05 | 5.88 | 1657 | 0.142 | 0.46 | 1.19 | 0.213 | 0.059 | 0.01 | - |
| + 150 | 43.64 | 32.08 | 2.72 | 0.944 | 0.084 | 0.327 | 0.61 | 0.222 | 0.046 | 0.024 | - |
| - 150 + 106 | 32.21 | 51.8 | 0.76 | 0.292 | 0.045 | 0.232 | 0.29 | 0.135 | <0.01 | <0.01 | - |
| - 106 +75 | 28.03 | 57.48 | 0.59 | 0.22 | 0.032 | 0.202 | 0.23 | 0.104 | <0.01 | <0.01 | - |
| - 75 +53 | 23.73 | 63.29 | 0.69 | 0.2 | 0.033 | 0.262 | 0.39 | 0.095 | <0.01 | <0.01 | 0.98 |
| -53 +45 | 20.05 | 68.85 | 0.65 | 0.189 | 0.03 | 0.3 | 0.34 | 0.084 | <0.01 | 0.02 | 0.75 |
| -45 +30 | 48.61 | 28.76 | 0.41 | 0.189 | 0.032 | 0.124 | 0.1 | 0.13 | <0.01 | <0.01 | 0.64 |
| -30 +19 | 43.24 | 35.58 | 0.57 | 0.213 | 0.033 | 0.248 | 0.23 | 0.119 | <0.01 | <0.01 | 0.86 |
| -19 +14 | 49.17 | 27.02 | 0.74 | 0.227 | 0.038 | 0.278 | 0.33 | 0.14 | <0.01 | <0.01 | 1.01 |
| -14 +6 | 54.68 | 17.86 | 1.03 | 0.28 | 0.048 | 0.351 | 0.53 | 0.191 | <0.01 | <0.01 | 1.4 |
| -6 +4 | 56.8 | 12.95 | 1.64 | 0.39 | 0.075 | 0.427 | 0.97 | 0.238 | 0.024 | <0.01 | 1.96 |
| -4 | 38.15 | 15.51 | 12.38 | 3442 | 0.242 | 0.221 | 2.03 | 0.27 | 0.133 | 0.014 | 9.34 |

The Tables 3 and 4 show the principal mineralogical phases identified in the tailings for the iron ore processing. Quartz is the predominant phase in the coarse particle fractions and hematite in the finer particle fractions. The most abundant mineralogical phases of the iron ore are the monocrystalline tabular hematite and the monocrystalline granular hematite. Goethite presents itself in greater quantity in the range below 7µm in both of the samples.

**TABLE – 3: Mineralogical Analysis By Size – Flotation Tailings**

Mineralogical analysis: Flotation tailings

| Size (µm) / wt.% | HTM | HGM | HTP | HGP | HL | MA | MG | GO | A | QZ | O |
|---|---|---|---|---|---|---|---|---|---|---|---|
| + 150 | 0.0 | 0.0 | 0.0 | 0.0 | 0.38 | 0.0 | 0.38 | 0.38 | 0.19 | 98.46 | 0.19 |
| - 150 + 106 | 0.0 | 0.0 | 0.0 | 0.0 | 0.57 | 0.0 | 0.0 | 0.19 | 0.0 | 99.05 | 0.19 |
| - 106 +75 | 0.0 | 0.4 | 0.0 | 0.0 | 1.59 | 0.0 | 0.2 | 0.0 | 0.20 | 97.42 | 0.2 |
| - 75 +53 | 1.67 | 1.12 | 0.0 | 0.0 | 3.53 | 0.19 | 0.0 | 0.19 | 0.19 | 92.94 | 0.19 |
| -53 +45 | 3.23 | 2.85 | 0.57 | 0.0 | 4.17 | 0.0 | 0.0 | 0.57 | 0.57 | 86.91 | 1.14 |
| -45 +30 | 31.45 | 12.24 | 0.38 | 0.0 | 10.73 | 1.32 | 0.19 | 0.19 | 0.0 | 43.31 | 0.19 |
| -33 +21 | 30.99 | 10.92 | 0.0 | 0.0 | 4.87 | 0.39 | 0.19 | 0.0 | 0.0 | 52.44 | 0.19 |
| -21 +15 | 41.69 | 9.47 | 0.17 | 0.0 | 2.66 | 0.33 | 0.0 | 0.17 | 0.17 | 45.18 | 0.17 |
| -15 +7 | 56.27 | 25.49 | 0.0 | 0.0 | 1.18 | 0.59 | 0.0 | 1.96 | 0.0 | 14.12 | 0.39 |
| -7 +4 | 65.80 | 17.56 | 0.0 | 0.0 | 0.55 | 1.66 | 0.0 | 4.81 | 0.0 | 8.32 | 1.29 |

MTH = monocrystalline tabula hematite; MGH = monocrystalline granular hematite; PTH = polycristalline tabular hematite
PGH = polycristalline granular hematite; LH = lobular hematite; ME = martite; MG = magnetite; GO = goethite; A = aggregate; QZ = quartz; O = others.

**TABLE – 4: Mineralogical Analysis By Size – Ultrafine Tailings**

Mineralogical analysis: Ultrafine tailings

| Size (µm) / wt.% | HTM | HGM | HTP | HGP | HL | MA | MG | GO | A | QZ | O |
|---|---|---|---|---|---|---|---|---|---|---|---|
| + 150 | 2.41 | 0.72 | 0.24 | 0.00 | 31.33 | 14.26 | 3.61 | 6.02 | 0.48 | 36.14 | 4.58 |
| - 150 + 106 | 1.69 | 1.32 | 0.38 | 0.19 | 21.09 | 10.55 | 0.38 | 3.20 | 1.69 | 56.12 | 3.39 |
| - 106 +75 | 1.31 | 1.31 | 0.37 | 0.00 | 26.59 | 7.68 | 0.37 | 3.00 | 0.56 | 57.49 | 1.31 |
| - 75 +53 | 2.26 | 4.33 | 0.38 | 0.19 | 12.43 | 3.01 | 0.38 | 2.82 | 0.75 | 71.19 | 2.26 |
| -53 +45 | 5.24 | 3.66 | 0.17 | 0.17 | 6.81 | 1.22 | 0.17 | 3.66 | 0.00 | 76.96 | 1.92 |
| -45 +30 | 34.56 | 19.47 | 0.18 | 0.00 | 7.02 | 4.74 | 0.53 | 2.81 | 0.00 | 30.00 | 0.7 |
| -30 +19 | 36.36 | 11.27 | 0.00 | 0.00 | 7.27 | 0.36 | 0.00 | 2.00 | 0.00 | 40.73 | 2 |
| -19 +14 | 49.38 | 16.40 | 0.18 | 0.00 | 1.25 | 2.32 | 0.36 | 3.92 | 0.00 | 23.89 | 2.32 |
| -14 +6 | 50.20 | 23.83 | 0.00 | 0.00 | 0.78 | 0.98 | 0.00 | 8.40 | 0.00 | 14.26 | 1.56 |
| -6 +4 | 66.55 | 17.63 | 0.00 | 0.00 | 0.54 | 0.54 | 0.00 | 10.43 | 0.00 | 3.24 | 1.08 |

MTH = monocrystalline tabular hematite; MGH = monocrystalline granular hematite; PTH = polycristalline tabular hematite
PGH = polycrystalline granular hematite; LH = lobular hematite; ME = martite; MG = magnetite; GO = goethite; A = aggregate; QZ = quartz; O = others.

The X-ray diffractograms by grain size range for both samples (Figures 2 and 3) identified are duction of the intensity of the quartz peaks and an increase in the hematite peaks as the granulometry decreased. The kaolinite peaks increased in the granulometric fraction below 4µm.

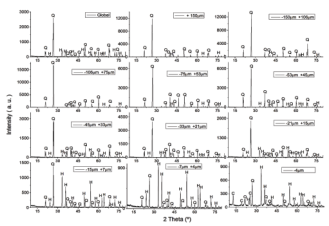

**Figure 2: X-Ray Diffraction of flotation tailings**

Notice also in Figure 2 that the predominance of quartz is verified in the global samples from the flotation tailings. In a different manner, Figure 3 displays the predominance of hematite in the global sample of the ultrafine tailings. These results are quantitatively proven by the global chemical analyses of the samples (Tables 1 and 2).

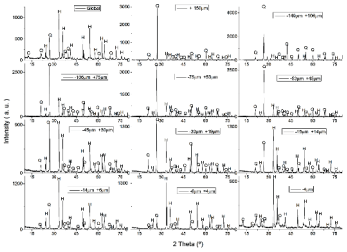

**Figure 3: X-Ray Diffraction of ultrafine tailings**

A thermomagnetic study of the samples involves thermal processing in air and without the addition of other chemical components, in which the Curie temperatures for magnetite (580°C) and hematite (675°C) were determined, evidenced by an increase of magnetization susceptibility near the critical temperatures, denominated the Hopkinson effect[3].

Figure 4 shows the thermomagnetic test in air results for the global sample and of the elven grain size ranges of the flotation tailings.

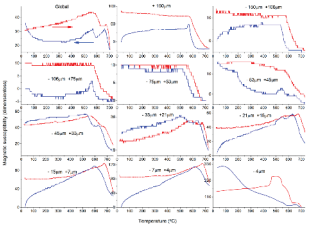

**Figure 4: Magnetic susceptibility of flotation tailings**

The curve of the global sample demonstrates the presence of hematite and magnetite in accordance with the mineralogical analysis. The curves of the fractions demonstrate a different behavior from that of the global sample, due to their different mineralogical compositions.

Notice that in the fraction greater than 150µm, there is a much greater magnetic potential than in the fraction between 45µm and 150µm, related to the volume of magnetite present (0.38%), in spite of the large amount of quartz present (98.46%). The magnetic potential is mainly influenced by the volume of magnetite. Observe that the Hopkinson peak is at 580ºC, which is characteristic of magnetite. The fractions between 150µm and 45µm have cooling curves that present negative magnetic susceptibility indexes, evidencing the diamagnetic behavior of the material, due to the predominance of quartz in these fractions as seen by the mineralogy in Table 3.

The minerology of the flotation tailings presented a magnetite weight of 0.19% in the fractions between 45µm and 33µm and for 33µm and 21µm, hematite was present with values of 56.12% and 47.17%, respectively. These minerals were identified in the thermomagnetic study by the Hopkinson peak characteristics, where magnetite peaked at 580ºC and hematite at 675ºC. In these specific cases, it was verified that the curves are considered to be reversible; in other words, there was no mineralogical transformation during the heating process in air.

For the fractions between 21µm and 15µm, 15µm and 7µm, and 7µm and 4µm, the curves observed in the thermomagnetic study are considered irreversible, confirming the mineralogical transformation. In addition, it can be verified that there is great geometric similarity in the heating and cooling curves, evidencing the mineralogical similarity of the mineralogical fractions.

The Figure 5 presents the results of the thermomagnetic test of the global sample and the eleven granulometric fractions of the ultrafine tailings. The curve for the global sample demonstrated the presence of hematite and magnetite as per the mineralogical analysis presented. The curves for the fractions from 150µm to 6µm presented behavior different from that of the global sample, being that only the fraction lesser than 6µm behaved similar to the global sample.

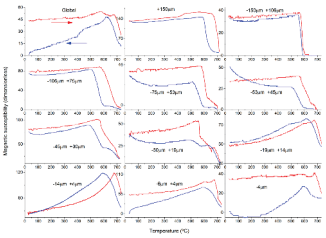

**Figure 5: Magnetic susceptibility of flotation tailings**

In Figure 5, it can also be observed that the fraction larger than 150µm has a greater magnetic potential than the fractions smaller than 150µm, due to the amount of magnetite present, a content of 3.61%, which is a much greater value than the other fractions, as displayed in the mineralogy analysis of Table 5. Another important observation is the reversible characteristic of the curves, demonstrating that there was no mineralogical transformation in the fractions from 150µm to 6µm, whereby only fractions smaller than 6µm and the global sample present irreversible curves.

## CONCLUSIONS

The thermomagnetic study demonstrated that the tailings present curves of magnetic susceptibility typical of materials containing iron oxides. The flotation tailings presented negative magnetic susceptibility in the fractions between 150µm and 45µm, indicating diamagnetic behavior due to the predominance of quartz. In the fractions between 21 and 15µm, 15µm and 7µm, and 7µm and 4µm, the material presented a positive magnetic susceptibility and irreversible themomagnetic curves, indicating mineralogical transformations. The ultrafine tailings presented a magnetic potential for concentration greater than that of the flotation tailings due to the amounts of magnetite and hematite present in the sample. Another important factor is the reversibility of the curves, demonstrating that there was no mineralogical transformation in the fractions between 150µm and 6µm, being that this only occurs in the fraction smaller than 6µm and in the global sample. Investigation of the magnetic susceptibility of the tailings proved to be an important technique for the technological characterization of the studied material.

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