Original Article

Characterisation and magnetic concentration of an iron ore tailings

Pedro Domingos Dauce\textsuperscript{a}, Guilherme Bernardes de Castro\textsuperscript{b}, Margarida Márcia Fernandes Lima\textsuperscript{c}, Rosa Malena Fernandes Lima\textsuperscript{b,}\textsuperscript{*}

\textsuperscript{a} Instituto Superior Politécnico de Tete, Tete, Mozambique
\textsuperscript{b} Universidade Federal de Ouro Preto, Departamento de Engenharia de Minas, Ouro Preto, MG, Brazil
\textsuperscript{c} Universidade Federal de Ouro Preto, Departamento de Engenharia Mecânica, Ouro Preto, MG, Brazil

\textbf{ARTICLE INFO}

\textbf{A R T I C L E  I N F O}

Article history:
Received 22 January 2018
Accepted 9 July 2018
Available online 31 August 2018

Keywords:
Iron ore
Tailings
Particle size
Mineralogy
Magnetic concentration

\textbf{A B S T R A C T}

The Quadrilátero Ferrífero mineral province is one of the most important sources of iron ore in Brazil. Nowadays this region produces 65% of the Brazilian iron ore. However, since the 1970s, there has been a continuous depletion of high Fe grade ores, obliging the mineral companies to concentrate low Fe grade ores in order to reach the quality demand of the steel mills. It is estimated that 400 kg of tailings is produced for each tonne of beneficiated iron ore. This means there is a very big volume of tailings dumped from industrial plants over the years. Owing to the inefficiency of the industrial operations, the tailings deposited in tailings dam have Fe grades similar to or higher than the Fe grades of some iron ores exploited nowadays (30–45 wt%). Therefore, the reprocessing of this material can recover millions of tonnes of discharged Fe and decrease the volume of the existent tailings, which can be interesting for the environmental and economic sustainability of this region. Herein, we present the characterisation of a tailings sample from a gravity concentration circuit of a mine located in northwest of Quadrilátero Ferrífero with the objective of proposing a concentration route for this material. The main characteristics of this tailings sample were: $d_{50} = 4$ mm; grades of 30.3 wt% Fe and 55.4 wt% SiO$_2$, while the identified minerals by XRD, optical microscopy and thermal analyses were quartz, hematite (martitic, lamellar, granular, sinuous, specularitic), goethite (alveolar, amphibolitic, botryoidal), magnetite, kaolinite (5.3 wt%) and apatite (0.7 wt%). Liberation of quartz for size fraction – 0.150 mm was of 80%. Bench magnetic concentration of this material increased the Fe grade up to 23 wt% and decreased the SiO$_2$ grade up to 39.4 wt% in the obtained concentrates, which is satisfactory for the rougher concentration step.

© 2018 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).

\textsuperscript{*} Corresponding author.
E-mails: rosa@ufop.edu.br, rosa.engminas@gmail.com (R.M. Lima).
https://doi.org/10.1016/j.jmrt.2018.07.015
2238-7854© 2018 Brazilian Metallurgical, Materials and Mining Association. Published by Elsevier Editora Ltda. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/4.0/).
1. Introduction

The total world iron ore production in 2014 was 3.22 billion tonnes. Brazilian production was 412 million tonnes (12.8 wt%) of which 10% was granular and 90% fines (sinter feed: 64.8%; pellet feed: 25.2%) with 63.5 wt% Fe on average. The Minas Gerais State (Quadrilátero Ferrífero) is the biggest iron ore producer (64.8%) in Brazil [1].

Owing to the low grade of the iron ore (31–45 wt% Fe), beneficiation is mandatory in Quadrilátero Ferrífero. Beneficiation for the steel making industry consists of comminution and size classification followed by concentration [2]. Based on the ore characteristics (e.g. particle size, mineralogy and grade), gravity concentration (jig and Humphreys spiral), magnetic separation (low and high-field) and inverse cationic flotation (especially applied to pellet feeds) are used [3,4]. It is estimated that for each tonne of beneficiated iron ore, 400 kg of tailings are produced. Based on the 2014 Brazilian production, about 275.5 million tonnes of waste was disposed of in landfills and tailing dams in 2014.

The failure of the Sarmarco tailings dam on November 5, 2015 was the biggest environmental disaster in the history of Brazilian mining. However, this episode constituted a turning point for the Brazilian mineral industry because practices were put in place to guarantee environmental sustainability and the minimisation of tailings discharge [5–8]. Several studies have demonstrated the use of iron ore tailings in brick manufacturing, concrete production and radiological protection as well as other applications [9–11].

When using drum-driven magnetic separation (1685 G), Alayali et al. [12], achieved separation of magnetic grains of oxides and iron-bearing minerals present in slags (average chemical composition (wt%): 32.62 CaO, 19.28 SiO2, 2.71 Al2O3, 22.61 Fe2O3, 7.52 MnO, 8.05 MgO, 0.13 K2O, 0.28 Na2O, 0.52 TiO2, 0.28 S) from the Kardemir integrated iron and steel mills, Karabuk, Turkey by selecting appropriate slag grain size and the gap between the drum and blades depending upon the required recovery of a product and the field of the usage area of a product.

It is well known that the mud (10 μm) from the de-mudding of the feed circuits of the flotation has Fe grade of 49–54 wt%, whereas wastes of gravity concentrations have Fe grade higher than 30 wt%. In both cases the discharged waste has Fe grade higher than the Fe grade of the ore feed [2]. Therefore, efforts are made to concentrate the tailings in order to increase (i) the total Fe recovery of beneficiation plants and (ii) the net worth of the by-products (bricks, concrete, etc.). In this study, we discuss the particle-size distribution, chemical composition and the mineralogy of a tailings sample of iron ore come from gravity (jigging) concentration with the objective to evaluate the possibility to concentrate this material for steel making industry.

2. Methods

A tailings sample from the jigging in the beneficiation plant of iron ore from the Complexo Serra Azul mine at the northwest of Quadrilátero Ferrífero, Brazil was used. The initial 580 kg sample was homogenised and split to aliquots for particle-size analysis, chemical and mineralogical analyses. The preparation of the sample used in the preliminary magnetic concentration tests was comprised fragmentation/classification to 1.4 mm.

2.1. Particle size and chemical composition

The particle-size distribution of initial sample was determined by combined (wet and dry) sieving (6.3–0.037 mm). The magnetic concentration sample was also sieved (1.4–0.037 mm) and then subjected to laser diffraction (Cilas 1064) (−0.037 mm).

Fe, SiO2, Mn, P and Al2O3 were determined by titrimetry. The loss of ignition (LOI) was measured by thermogravimetry (Q50 TA). The total weight loss and thermogravimetry were carried out at 25–1000 °C, 10 °C/min, with isothermal heating at 1000 °C for 5 min and 100 mL/min gas flux (N2 5.0) (10 mL/min for thermoscaling and 90 mL/min for purging).

2.2. Mineralogy

After sieving, the particles retained in each sieve were grouped into the following sizes: >4.76 mm, 4.76–2.38 mm, 2.38–1.4 mm, 1.4–0.840 mm, 0.840–0.297 mm and <0.297 mm. A Pananalytical model X’pert³powder diffractometer, with a Cu tube and Ni filter, was used at 40 mA and 45 kV. The 2θ scan range was 5–90°C and data were collected by using the X-ray Data Collector software. Minerals were identified based on the PDF-2 database in the HighScore Plus software.

The microtexture and quartz liberation (Gaudin method) were studied by optical microscopy (LEICA model DMLP) using aliquots of the retained particles in each sieve (initial sample), which were mounted in thin sections and polished.

2.3. Magnetic concentration

In the preliminary magnetic concentration, a magnetic separator Carpo Inc. model WHIMS 3X4L – serial 210–97 was used. The Minitab 17 software was used to plan and analyze the experimental results (2² with replicate). The following parameters were investigated: sample type (global, +74 μm) and magnetic field magnitude (3575 and 5465 Gauss). The analysed response variables were the mass recovery, the Fe and SiO2 grade in the concentrates, Fe recovery, and the Gaudin selectivity index (S.I.) between quartz and iron minerals.

For each magnetic concentration test, at first the matrix (basket full of steel spheres of 1.5 cm diameter) was inserted between the electromagnets of equipment. Afterwards, the magnetic field intensity was fixed to the desired value (3575 or 5465 Gauss). Then, with the water tap open (flow rate of 2.791 min⁻¹), a 500 g sample of dry tailings (global or +74 μm) was fed into the equipment. When it was observed that no particles were dropping into the non-magnetic bucket product, the bucket was replaced by a new one. Then, the equipment was turned off and the magnetic product was collected in the new bucket. Finally, both the magnetic (concentrate) and non-magnetic (tailings) products were filtered, dried, weighed (to calculate mass
recovery) and prepared for chemical analyses in order to determine the grades of Fe and SiO₂ in the products obtained (concentrates and tailings) and to calculate the Fe recovery.

3. Results and discussion

3.1. Particle size and chemical composition

Fig. 1 shows the particle-size distribution of the iron ore jiggings tailings sample and the concentration feed sample (grinding/classification at 1.4 mm). As observed, the d₈₀ of the tailings sample is approximately 4000 μm (4 mm) with only 4% of the particles below 37 μm (0.037 mm). The d₈₀ of the concentration feed sample is 840 μm (0.840 mm), with only 4% of particles below 10 μm (0.010 mm) (mud).

The Fe grade of the tailings sample (Table 1) is 30.3 wt% and can be used as feed in industrial beneficiation plants that use iron ore with Fe grade between 25 wt% and 45 wt% [13]. In addition, the tailings sample between 0.297 mm and 1.4 mm has SiO₂ greater than 65 wt% and approximately 20 wt% Fe.

3.2. Mineralogy

The XRD data for the particle-size classes in Table 1 are given in Table 2. Based on the identified minerals (Table 2) and chemical composition presented at Table 1, it is possible to infer that the main mineral phases decrease in the following order: quartz (SiO₂), hematite (Fe₂O₃) and goethite (FeO·OH). Magnetite (Fe₃O₄) was identified only in the >2.38 mm fraction. The lack of magnetite below 2.38 mm could be related to its low content (<5%), since the mineral identification in XRD patterns is possible only for contents higher than 3–5 wt%. As well known, the magnetite content of ores from Quadrilátero Ferrífero, is usually smaller than 5 wt% [2].

Fig. 2 shows typical microphotographs of the iron ore tailings sample, with hematite microtextures. In Fig. 2(a), the quartz (Qz) and porous martitic hematite (HmM) suggest that quartz liberation can readily occur. Fig. 2(b) shows the intergrowths of magnetite (Mg) and martitic hematite (HmM) and lamellar hematite (HmL) (centre). Fig. 2(c) shows martitic hematite (HmM), sinuous hematite (HmSi) and quartz (Qz). Fig. 2(d) shows granular hematite (HmG), specularitic hematite (HmS), quartz (Qz) and goethite (Goe).

Fig. 3 shows reflected light images of goethite morphologies. In Fig. 3(a) and (b), the botryoidal goethite (GoeB) in the centre is surrounded by quartz (Qz) and martitic hematite (HmM). Fig. 3(c) shows amphibolitic goethite (GoeAm), quartz (Qz) and martitic hematite (HmM) and Fig. 3(d) shows alveolar goethite (GoeAl) with pores (Po) of various sizes. Other minerals identified by optical microscopy are kaolinite (Kao) and apatite (Ap) (Figs. 3(d) and 4). The latter is seen inside the pores of alveolar goethite (GoeAl).

### Table 1 – Chemical analysis by fraction.

<table>
<thead>
<tr>
<th>Size (μm)</th>
<th>Weight (%)</th>
<th>Grade (wt%)</th>
<th>Distribution (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe</td>
<td>SiO₂</td>
<td>Al₂O₃</td>
</tr>
<tr>
<td>+4760</td>
<td>7.6</td>
<td>38.3</td>
<td>37.0</td>
</tr>
<tr>
<td>−4760 + 2380</td>
<td>21.9</td>
<td>36.9</td>
<td>39.6</td>
</tr>
<tr>
<td>−2380 + 1400</td>
<td>19.7</td>
<td>28.7</td>
<td>52.5</td>
</tr>
<tr>
<td>−1400 + 840</td>
<td>16.1</td>
<td>19.7</td>
<td>65.8</td>
</tr>
<tr>
<td>−840 + 297</td>
<td>16.2</td>
<td>20.4</td>
<td>65.7</td>
</tr>
<tr>
<td>−297</td>
<td>18.5</td>
<td>39.1</td>
<td>39.5</td>
</tr>
<tr>
<td>Total</td>
<td>100</td>
<td>30.3</td>
<td>50.4</td>
</tr>
</tbody>
</table>

### Table 2 – Minerals identified by X-ray diffraction in each size fraction.

<table>
<thead>
<tr>
<th>Fraction size (μm)</th>
<th>Minerals identified</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quartz</td>
</tr>
<tr>
<td>+4760</td>
<td>Yes</td>
</tr>
<tr>
<td>−4760 + 2380</td>
<td>Yes</td>
</tr>
<tr>
<td>−2380 + 1400</td>
<td>Yes</td>
</tr>
<tr>
<td>−1400 + 840</td>
<td>Yes</td>
</tr>
<tr>
<td>−840 + 297</td>
<td>Yes</td>
</tr>
<tr>
<td>−297</td>
<td>Yes</td>
</tr>
</tbody>
</table>
A typical thermogram of the iron ore tailings sample is shown in Fig. 5 (4.76–2.38 mm). There is a thin peak at 50 °C ascribed to the water loss in kaolinite and the peaks at 100–130 °C are ascribed to the loss of water physically adsorbed on kaolinite and goethite. The two peaks between 175 and 310 °C are due to the dehydroxylation of crystalline goethite to hematite (2FeO·OH → Fe₂O₃·H₂O). The kaolinite dehydroxylation (Al₂[Si₂O₅](OH)₄ → Al₂O₃ + 2SiO₂ + 2H₂O) to mullite between 450 and 700 °C. Around 750 °C, there is a small endothermic peak. In the pure goethite thermogram, a fourth mass loss occurs between 310 and 1000 °C. This loss belongs to the decomposition of remnant structural hydroxyl and partly to nonstoichiometric hydroxyl units [14–17].

The liberation spectrum of quartz is shown in Fig. 6. As particle size decreases more quartz is liberated. The quartz liberation reaches 80% below 0.150 mm.

In the sample, the goethite is of 22.7% by weight. This was determined by assuming that Al₂O₃ (2.1 wt%) and P (0.13 wt%) were derived from kaolinite (5.3 wt%) and hydroxyapatite

![Photomicrographs](image-url)
Fig. 3 – Images (reflected light) of goethite present in different size fractions of iron ore tailings: (a) 150–105 µm: botryoidal goethite (GoeB – centre) surrounded by quartz (Qz) and martitic hematite (HmM), (b) 592–420 µm: crystal of botryoidal goethite (GoeB) evidencing its growth zoning, (c) 150–105 µm: Mixt particle (centre) containing amphibolitic goethite (GoeAm) and quartz (Qz), (d) 6300–4760 µm: alveolar goethite (GoeAl) with pores (Po) of various sizes and grains of kaolinite (Kao).

Fig. 4 – Images (reflected light) of kaolinite (Kao) and apatite (Ap) inside pores in alveolar goethite (GoeAl) (>6.3 mm): (a) under polarised light, and (b) under crossed nicols.
(0.7 wt%). The LOI (3.0 wt%) is attributed to thermal decomposition of goethite, kaolinite and hydroxyapatite.

It is well known that goethite is very friable and in comminution, it causes a high proportion of fine particles that are finer than 10 μm (0.010 mm), which makes selectivity in gravity and flotation concentration difficult. Thus, the comminuted ore with high goethite content must be de-mudded. De-mudding causes the loss of Fe-rich mud. To reprocess the characterised tailings in this study, it could be pre-concentrated by magnetic separation to eliminate the liberated gangue minerals (quartz – 50 wt%). Next, depending on the quality of the coarse concentrate, it must be comminuted up <105 μm and inverse flotation can be applied.

3.3. Magnetic concentration

Table 3 lists the results of the magnetic concentration tests. The mass recovery and Fe recovery are the highest for the global sample at the highest magnetic field (5465 Gauss). But the Gaudin selectivity index (S.I.) between Fe and SiO₂ are the highest for the +74 μm fraction, especially for a magnetic field of 5465 Gauss, which is consistent with the optimal performance of magnetic separation equipments [18]. However, the results for the global sample are not much smaller than the results for the +74 μm fraction; this was due to the global sample having only 9.1% of particles passing the 74 μm (0.074 mm) sieve (Fig. 1).

In general (see Table 3), Fe increased more than 20 wt% and SiO₂ decreased more than 35 wt% in the concentrates compared with the feed sample (Table 1). This is satisfactory for a rougher concentration step, since the objective of this study was to get a poor pre-concentrate, owing to the quartz liberation characteristic of the initial sample (see Fig. 6). It means that there are no liberated particles (mixed particles) of quartz from iron minerals in the concentrates. Subsequently, the concentrates can be purified by magnetic concentration using a weaker magnetic field and the mixed particles can be ground and concentrated by inverse flotation [3]. Another alternative is to blend the concentrates with rich by-products (with content of Fe, SiO₂, Al₂O₃, P, etc.) the qualities required for steel making.

**Table 3 – Raw data of the tests of concentration magnetic.**

<table>
<thead>
<tr>
<th>Magnetic field (Gauss)</th>
<th>Sample (μm)</th>
<th>Mass recovery (%)</th>
<th>Grade (wt%)</th>
<th>Fe recovery (%)</th>
<th>I.S.</th>
</tr>
</thead>
<tbody>
<tr>
<td>3575 (−)</td>
<td>Global (−)</td>
<td>34.69</td>
<td>52.19</td>
<td>18.74</td>
<td>56.12</td>
</tr>
<tr>
<td>3575 (−)</td>
<td>Global (−)</td>
<td>32.65</td>
<td>53.30</td>
<td>16.96</td>
<td>55.88</td>
</tr>
<tr>
<td>5465 (+)</td>
<td>Global (−)</td>
<td>40.40</td>
<td>49.69</td>
<td>22.37</td>
<td>63.25</td>
</tr>
<tr>
<td>3575 (−)</td>
<td>+74 (+)</td>
<td>35.35</td>
<td>52.88</td>
<td>17.48</td>
<td>61.58</td>
</tr>
<tr>
<td>5465 (+)</td>
<td>+74 (+)</td>
<td>38.38</td>
<td>52.38</td>
<td>20.90</td>
<td>64.92</td>
</tr>
<tr>
<td>5465 (+)</td>
<td>Global (−)</td>
<td>41.41</td>
<td>51.49</td>
<td>19.58</td>
<td>68.77</td>
</tr>
<tr>
<td>3575 (−)</td>
<td>+74 (+)</td>
<td>34.34</td>
<td>52.87</td>
<td>18.15</td>
<td>59.09</td>
</tr>
<tr>
<td>5465 (+)</td>
<td>+74 (+)</td>
<td>38.38</td>
<td>52.74</td>
<td>17.80</td>
<td>66.35</td>
</tr>
</tbody>
</table>

Fig. 5 – Thermogram of 4760–2380 μm of jiggling iron ore tailings.
Table 4 – Summary of estimated effects and coefficients for mass recovery, Fe and SiO$_2$, Fe recovery and Gaudin selectivity index (S.I.).

<table>
<thead>
<tr>
<th>Term</th>
<th>Mass recovery (wt%)</th>
<th>Fe (wt%)</th>
<th>SiO$_2$ (wt%)</th>
<th>Fe recovery (wt%)</th>
<th>S.I.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Effect</td>
<td>coef</td>
<td>p</td>
<td>sig</td>
<td>Effect</td>
</tr>
<tr>
<td>Constant</td>
<td>36.950</td>
<td>0.000</td>
<td>Yes</td>
<td></td>
<td>52.193</td>
</tr>
<tr>
<td>A</td>
<td>5.385</td>
<td>2.693</td>
<td>0.001</td>
<td>Yes</td>
<td>−1.235</td>
</tr>
<tr>
<td>B</td>
<td>−0.675</td>
<td>−0.337</td>
<td>0.339</td>
<td>No</td>
<td>1.050</td>
</tr>
<tr>
<td>A×B</td>
<td>−1.85</td>
<td>−0.925</td>
<td>0.041</td>
<td>Yes</td>
<td>0.920</td>
</tr>
<tr>
<td>Standard deviation – 0.880469</td>
<td>Standard deviation – 0.758436</td>
<td>Standart deviation – 1.62062</td>
<td>Standard deviation – 2.20151</td>
<td>Standard deviation – 0.312250</td>
<td></td>
</tr>
</tbody>
</table>

A, magnetic field; B, sample; coef, coefficient; sig, significance.
Table 4 summarises the estimated effects and coefficients of the mass recovery, the grades of Fe and SiO₂ in the obtained concentrates, the Fe recovery, and the Gaudin selectivity index (S.I.) of codified factors for the 95% confidence level (α = 0.05). The magnetic field and sample did not significantly affect the Fe and SiO₂ grades and the Gaudin selectivity index (S.I.).

4. Conclusions

We conclude that $d_{80}$ of the initial tailings is 4mm and the sample is comprised of the following minerals: quartz (47.9 wt%), hematite (martitic, specularitic, lamellar and sinuous), goethite (alveolar and botryoidal) (22.7 wt%), kaolinite (5.3 wt%) and apatite (0.7 wt%). Kaolinite and apatite occur preferentially inside the pores of alveolar goethite. Quartz liberation is greater than 90% for the –105 μm fraction. The global sample contains 30.3 wt% Fe, 50.4 wt% SiO₂, 2.1 wt% Al₂O₃, 0.13 wt% P and 3 wt% LOI. After magnetic concentration, the mass and Fe recovery are greater than 40 wt% and 64 wt%, respectively, for a magnetic field of 5465 Gauss. An increase of 20 wt% in Fe and decrease of 35 wt% in SiO₂ is seen in the obtained concentrates when compared with the magnetic concentration feed, which is deemed satisfactory for rougher operation.

Conflicts of interest

The authors declare no conflicts of interest.

Acknowledgments

We would like to acknowledge Fapemig (PPM – 00082-14) and UFOP (Research Fund) for financial support, the mining company for providing the tailings sample and chemical analysis, the Instituto Superior Politécnico de Tete (Moçambique) in coordinating with the Cinop project (Holland) for scholarship to Dauce, P.D. and CNPq for scholarships to Castro, G.B. and Lima, R.M.F.

References


