Original Article

Characterization and concentration by selective flocculation/magnetic separation of iron ore slimes from a dam of Quadrilátero Ferrífero – Brazil

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A B S T R A C T
In this paper the results of size, chemical and mineralogical characterizations as well as the concentration tests performed with a slimes sample of iron ore from a dam at the Quadrilátero Ferrífero in the state of Minas Gerais, Brazil are presented. The sample was constituted by 75 % of slimes (particles ~10 μm). The main minerals identified by X-ray diffractometry and thermal analysis were: goethite, hematite, quartz and kaolinite. The sample grades were: Fe (~51.4 wt.%), SiO₂ (~15.1 wt.%), Al₂O₃ (~3.4 wt.%), P (0.097 wt.%) and LOI (~7 wt.%). During the optimized bench testing condition of selective flocculation/magnetic separation (pH = 10.5, sodium silicate and corn starch =50 g/ton, magnetic strength =3575 G) by experimental statistical planning design, the mass recovery was of 48.4 %. There was an increase of 8 % in Fe grade and a decrease of 50 % in both SiO₂ and Al₂O₃ grades when compared with the feed. Agglomeration studies with the obtained concentrate will be performed in future.

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1. Introduction

The world wide increasing demands of iron ore products in addition to the depletion of high-grade iron ore deposits enables the exploitation of low-grade iron ores with complex mineralogy. These ores have to be comminuted, classified by size, and concentrated in order to obtain products according to the quality demands for both the iron and steel industries, which represent about 90 % of the total iron ore world production [1]. The industrial process choice to concentrate them (gravimetry, magnetic separation and flotation) is based on their mineralogy and liberation size of valuable minerals from gangue [2,3].

The tailings discharged from the industrial process described before can represent up to 40 % of the feed input in these operations (around 275.5 million-ton in Brazil just in 2014). Depending on Fe grades of discharged tailings it could be worthwhile to reprocess these materials in order to recover the Fe, which can be used in the iron and steel industries or to apply them without concentration operations for others finalities such as brick manufacturing, concrete production, radiological protection and pigment in the production of paint for buildings [4–8].
Inverse cationic flotation, using starch as iron minerals depressant and amine as siliceous gangue collector, is used to concentrate fraction sizes −105 μm. During the 1970s in Brazil, the Samarco Mining Company was the pioneer in using this technique to process siliceous itabirite. The size degradation of goethites and in addition, the clay minerals (kaolinite and gibbsite) in low grade ores produce excessive slime quantities (particles -10 μm) in pulp, which undermines the flotation process performance due to their small mass and high specific surface area, which among other factors, cause the sliming coating phenomenon [9]. For this reason, the introduction of the desliming step before flotation process is mandatory.

In this work, characterization studies (physical, mineralogical, chemical and physicochemical) in an iron ore slimes sample were performed. Afterwards, selective flocculation/magnetic separation tests of this sample were conducted with the objective to recover it, so it could be blended with the flotation concentrates used in the pelletizing process.

2. Materials and methods

2.1. Materials

The iron ore slimes sample used in this work was collected at 21 points (the depth varied from 0.36 to 1.18 m) in the dam tailings of Fundão at the Samarco Mining site which collapsed on November 5th, 2015. In this dam, the slimes from the desliming operation of the flotation feed were deposited for many years. This material was dried, disaggregated, homogenized and quartered to obtain subsamples for the characterization studies (physical, mineralogical, chemical and physicochemical) and selective flocculation/magnetic separation. See Fig. 1.

The reagents used were: NaCl (trademark: Synth) to control the ionic strength of electrophoretic mobility determinations; NaOH (trademark: Synth) and HCl (trademark: Synth) to adjust the pH of solutions and pulps; sodium metasilicate ((Na₂SiO₃), XH₂O – trademark: Vetec) as dispersant and corn starch (trademark: Maizena) as flocculant.

2.2. Methods

2.2.1. Analysis of size, density and specific surface areas

Particle size analysis of the sample was performed by the particle size analyzer Cilas model 1064. The sodium metasilicate solution (250 mL) (0.1 % wt.) and the ultrasound (1 min) was used to disperse the particles for 2 min. Moreover, the specific weight of the sample was performed by gas pycnometer (Quantachrome Instruments model Ultrapyc 1200e) as methodology described by Silva et al. [1]. The specific surface area of the sample was measured using a Quantachrome model Nova 1200e surface and porosity analyzer using the methodology described by Pereira et al. [10].

2.2.2. Mineralogy and chemical composition

The main mineralogical phases of the iron ore slimes sample were determined by X-ray diffractometry (powder total method). A Panalytical X-ray diffractometer model X’pert Powder, equipped with a Cu-Kα radiation source (λ =1.5405 Å) was used. The samples were scanned from 5° ≤ 2θ ≤ 90°. The database PDF-2/ICDD was used to identify the minerals. In order to identify/confirm the clay minerals in the slimes sample, a thermal analysis was conducted on a TA Instruments model TGA Q50 thermogravimetric analyzer. The standard operational conditions of thermal analysis were: Temperature - 23−1000 °C, ramp - 10 °C/minute, N₂ flowrate =100 mL/minute (90 mL/minute for sample and 10 mL/minute for micro scale) and isotherm of 5 min at 1000 °C.

The grades of Fe₉₀, SiO₂, Al₂O₃, CaO, MgO, TiO₂, MnO, P of head sample and concentration products (concentrates and tailings) were performed by the X-ray fluorescence method. The loss of ignition (LOI) was determined by the gravity method. All chemical analysis was performed in the Chemical Analysis Laboratory at the Samarco Mining company.

2.2.3. Determination of electrophoretic mobility

Electrophoretic mobilities of iron ore slimes conditioned with and without sodium metasilicate at pH values 7 and 10.5 were performed by zetameter Zetasizer Nano Series – Malvern Instruments. For all measurements, 10⁻⁴ M NaCl solution was used to get constant ionic strength.

The experimental procedure of electrophoretic mobility determinations was performed as follows: 0.1 g of slimes sample was dispersed in 250 mL 10⁻⁴ M NaCl solution. The suspension after agitation by inversion rested for about 20 min (specific weight of 4.17 g/cm³) to settle the particles +10 μm, according to the law of Stokes. Then, the supernatant suspension was transferred to 50 mL beakers and the pH was adjusted to 7 or 10.5 with NaOH under constant agitation for 5 min. Subsequently, the suspension was introduced into the folded capillary cell to measure the electrophoretic mobility using the zeta meter. The electrophoretic mobility for each condition was determined as the average of the values obtained in three replicate measurements.
2.2.4. Selective flocculation/magnetic separation
For the selective flocculation/magnetic separation tests carried out with studied iron ore slimes, a complete factorial planning design with two replicates was used to study the effects of the following factors: pH (7 and 10.5), magnetic field (3575 and 5465 G), starch dosage (50 e 200 g/ton) on response variables: mass recovery, Fe recovery, grades of Fe, SiO₂, Al₂O₃, P and LOI. The sodium metasilicate dosage, previously determined by Ma [11], was of 50 g/ton for all tests.

For selective flocculation tests, 5 % pulp density was prepared with the addition of 75 g of iron ore slimes and ~1480 mL of tap water into 2000 mL flotation cell (CDC, model CFB-1000 EEPN). Thereafter, sodium metasilicate (50 g/ton) was introduced and the pH was adjusted to the desired value (7 or 10.5) and conditioned for 10 min at cell speed of 2000 RPM. Then, the cell speed was decreased to 1000 RPM. The starch was added (50 or 200 g/ton). Next, the pH was adjusted again to the desired value (7 or 10.5) and conditioned for 2 more minutes.

For the magnetic separation tests, a magnetic separator Carpc Inc. model WHIMS 3 x 4L – serial 210) was turned on and set to appropriate magnetic field (3575 and 5465 G) to separate the magnetic (concentrate) and non-magnetic (tailing) products. In order to remove the non-magnetic particles from the matrix (1.25 kg of steel spheres of 4.9 mm average diameter) and entrained with iron mineral flakes, was performed by washing the matrix with tap water for 5 min. Additionally, the bucket with non-magnetic product was removed. Another bucket was inserted and the magnetic field was turned off, and just then, the magnetic product was collected. Subsequently, the products from the magnetic separation were dried, weighted, quartered, ground and sent to chemical analysis to determine the grades of Feₜotal, SiO₂, Al₂O₃, P and LOI.

3. Results and discussion

3.1. Physical characterization
Fig. 2 depicts the size distribution of iron ore slimes sample. As it can be observed, about 75 % and 20 % of particles in the material were below 10 µm and 1 µm, respectively. Therefore, selective flocculation is necessary to concentrate this material, once the classical concentration methods (physical and flotation), used to concentrate low grade iron ores are extremely inefficient for this size fraction [12,13].

The specific weight of studied slimes was of 4.17 g/cm³. This value is higher than the specific weight (3.9 g/cm³) of an iron ore sample collected in 2015 from the feed of industrial flotation circuit of the Samarco Mining, which was studied by Cruz and Lima [14]. Therefore, it is possible to infer that the Fe grade of this material collected in the dam tailings was higher than the Fe grade of conventional flotation feed from this company. The specific surface area (BET) of these slimes was 14.8 m²/g (14,800 cm²/g). Usually, the specific surface areas of iron ore concentrate used in pelletizing processes are 1900 (±100) cm²/g [15].

3.2. Mineralogical and chemical characterization

Figs. 3 and 4 show, respectively, the X-ray diffractogram and thermogram of studied iron ore slimes. The identified minerals in X-ray diffractogram (Fig. 3) were: goethite (HFeO₂), hematite (Fe₂O₃), quartz (SiO₂) and kaolinite (Al₄(Si₂O₅)(OH)₈). Peaks around 300 °C and 500 °C in thermogram of Fig. 4, the goethite and kaolinite minerals were confirmed in these slimes. In accordance with literature, goethite and kaolinite dehydroxilation happen between 250 to 350 °C and 450 to 550 °C, respectively [16,17].

Table 1 presents the chemical composition and ignition loss (LOI) of the slimes sample. As it has been observed, the Feₜotal (51.4 wt. %) grade of this sample was much higher than the Feₜotal (31–45 wt. %) grades of iron ore exploited nowadays in
3.3. Physicochemical characterization

Fig. 5 shows the electrophoretic mobility of iron ore slimes studied at pHs 7 and 10.5 conditioned with and without sodium metasilicate. According to Fig. 5, the electrophoretic mobility of slimes has become more negative with pH increases in both conditions: conditioning with and without sodium metasilicate.

The isoelectric point (IEP) of hematite, reported in literature, varies from 4.8 to 7.4, and for goethite it varies from 6.7–8.2 [21–26]. These differences probably are due to different electrolytic solutions used in zeta potential determinations by several researchers as well as the impurities of analyzed samples. Quartz IEP varies from 1.4 to 2.2 [21,23–25]. In accordance with Hu et al. [27], the IEP of kaolinite is of 2.4. For this reason, electrophoretic mobility of studied slimes (Fig. 5) without dispersant (sodium metasilicate) at pH 10.5 was higher than at pH 7.

At pH 7 the dominant sodium metasilicate specimen is [Si (OH)₄]. At pH 10.5, and the decreasing order of species concentration is: [Si (OH)₄] > [Si (OH)₃] > [SiO₃(OH)₂] > [Si₄O₅(OH)₅] [28]. In accordance with Jordan et al. [29], the silicate species in the aqueous solution form complex =FeH₂SiO₄ and =FeH₂SiO₄⁻ on surfaces of iron oxides/hydroxides are in the following affinity order: goethite, hematite and magnetite. Ma [30] concluded that sodium silicate is an effective depressant for kaolinite in iron ore flotation only when there are positive sites on mineral surface and zeta potential values smaller than ~30 mV.

3.4. Selective flocculation/magnetic separation

Table 2 presents raw results of selective flocculation/magnetic concentration performed with slimes of iron ore sample, using 50 g/ton of dispersant (sodium metasilicate). It is observed that the mass recoveries varied from 47 % to 61 %. The obtained Fe grades varied from 55 wt.% to 59.3 wt. %, while SiO₂ varied from 7.3 wt.% to 12 wt.%.

Table 3 summarizes the estimated effects and coefficients of mass recovery, Fe recovery and grades of Fe, SiO₂, Al₂O₃, P and LOI of codified units for 95 % (alpha = 0.05) confidence level. Based on p-values presented at Table 3, was observed that no investigated factor was significant for Fe recovery and P grade in obtained concentrates.

The magnetic field (B) in magnitude range tested in this work, individually, did not have significance for any response variables analyzed. Only pH (A) had significance for Al₂O₃ grade. However, the enlargement of ultrafine iron mineral particles by polymer such as starch is mandatory to decrease loss of the ultrafine particles in magnetic separation [13]. Considering that all Al₂O₃ (3.39 wt. %) in Table 1 is coming from kaolinite (Al₄(Si₂O₇)(OH)₈), the goethite content can be at least 57.4 %. Therefore, it could be worthwhile to use high magnetic field (> 9000 G) to recover the goethite in this material and to increase the grade and recovery of Fe [2,18,31]. Fig. 6 presents the main factors, which had significance in the analyzed variable responses: mass recovery, grades of Fe, SiO₂, Al₂O₃, P and LOI. As it can be seen, the results from the selective flocculation/magnetic separation at pH 10.5 were better than at pH 7, which can be related with the higher efficiency of dispersion at pH 10.5, once the electrophoretic mobility in this pH value was more negative than at pH 7 (see Fig. 5). These results are coherent with literature data [32,33].

In the optimization tool of the Minitab 17 software (goal: maximize Fe grade and minimize SiO₂ grade), the optimized response was achieved at the following condition: pH = 10.5, magnetic field =3574 G and 50 g/ton of starch. In these conditions, the mass recovery was of 48.4 %,
Table 2 – Raw data of selective flocculation/magnetic concentration of iron ore slimes sample.

<table>
<thead>
<tr>
<th>Effects</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>Mass recovery (%)</th>
<th>Fe recovery (%)</th>
<th>Fe</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>P</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>50</td>
<td>50</td>
<td>48.3³, 46.8³</td>
<td>55.1³, 53.4³</td>
<td>57.4³, 58.4³</td>
<td>9.4³, 8.7³</td>
<td>2.2³, 1.8³</td>
<td>0.08³, 0.11³</td>
<td>5.7³, 5.3³</td>
<td></td>
</tr>
<tr>
<td>10.5</td>
<td>50</td>
<td>50</td>
<td>49.3³, 47.4³</td>
<td>57.2³, 55.3³</td>
<td>59.3³, 59.5³</td>
<td>7.3³, 7.1³</td>
<td>1.8³, 1.4³</td>
<td>0.11³, 0.11³</td>
<td>5.3³, 5.0³</td>
<td></td>
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<tr>
<td>7</td>
<td>50</td>
<td>50</td>
<td>49.5³, 49.0³</td>
<td>55.5³, 55.5³</td>
<td>57.1³, 56.1³</td>
<td>9.6³, 11.5³</td>
<td>2.1³, 2.2³</td>
<td>0.10³, 0.11³</td>
<td>5.9³, 6.6³</td>
<td></td>
</tr>
<tr>
<td>10.5</td>
<td>50</td>
<td>50</td>
<td>48.1³, 48.3³</td>
<td>55.8³, 55.9³</td>
<td>59.3³, 59.3³</td>
<td>7.9³, 7.1³</td>
<td>1.9³, 1.6³</td>
<td>0.11³, 0.12³</td>
<td>5.4³, 5.2³</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>200</td>
<td>200</td>
<td>47.4³, 47.3³</td>
<td>53.7³, 53.4³</td>
<td>58.3³, 58.2³</td>
<td>7.9³, 8.5³</td>
<td>2.1³, 2.1³</td>
<td>0.10³, 0.14³</td>
<td>5.7³, 5.6³</td>
<td></td>
</tr>
<tr>
<td>10.5</td>
<td>200</td>
<td>200</td>
<td>58.2³, 60.1³</td>
<td>63.0³, 66.4³</td>
<td>56.0³, 55.5³</td>
<td>11.3³, 12.0³</td>
<td>1.8³, 2.1³</td>
<td>0.08³, 0.09³</td>
<td>5.5³, 5.9³</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>200</td>
<td>200</td>
<td>53.6³, 54.1³</td>
<td>60.1³, 60.7³</td>
<td>57.3³, 56.8³</td>
<td>9.4³, 9.4³</td>
<td>2.3³, 2.2³</td>
<td>0.13³, 0.09³</td>
<td>5.7³, 6.0³</td>
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<td>58.9³, 59.0³</td>
<td>8.2³, 7.1³</td>
<td>1.7³, 1.6³</td>
<td>0.10³, 0.10³</td>
<td>5.3³, 5.2³</td>
<td></td>
</tr>
</tbody>
</table>

A – pH; B – magnetic field (Gauss); C – corn starch (g/tonne); LOI – loss of ignition, superscript – test order.

Table 3 – Summary of the estimated effects, coefficients for mass recovery, Fe recovery, grades of Fe, SiO₂, Al₂O₃, P and LOI of coded units for a confidence level of 95 % (alpha = 0.05).

<table>
<thead>
<tr>
<th>Term</th>
<th>Mass recovery (%)</th>
<th>Fe recovery (%)</th>
<th>Grade wt(%)</th>
<th>Fe</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>P</th>
<th>LOI</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Effect Coef p-value Sig.</td>
<td>Effect Coef p-value Sig.</td>
<td>Effect Coef p-value Sig.</td>
<td>Effect Coef p-value Sig.</td>
<td>Effect Coef p-value Sig.</td>
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<td></td>
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<tr>
<td>Constant</td>
<td>50.38 0.000 Yes</td>
<td>57.14 0.000 Yes</td>
<td>57.89 0.000 Yes</td>
<td>57.89 0.000 Yes</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>1.69 0.84 0.006 Yes</td>
<td>−0.89 −0.45 0.633 No</td>
<td>0.91 0.45 0.002 Yes</td>
<td>−0.63 −0.32 0.066 No</td>
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<tr>
<td>B</td>
<td>−0.55 −0.28 0.264 No</td>
<td>2.54 1.27 0.195 No</td>
<td>0.16 0.08 0.434 No</td>
<td>−0.22 −0.11 0.494 No</td>
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<td></td>
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<tr>
<td>C</td>
<td>4.12 2.06 0.000 Yes</td>
<td>−3.31 −1.66 0.103 No</td>
<td>−0.80 0.40 0.003 Yes</td>
<td>0.61 0.30 0.077 No</td>
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<td>−4.77 −2.39 0.000 Yes</td>
<td>0.77 0.38 0.680 No</td>
<td>1.38 0.69 0.000 Yes</td>
<td>−1.57 −0.79 0.001 Yes</td>
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<tr>
<td>AC</td>
<td>1.83 0.91 0.004 Yes</td>
<td>1.56 0.78 0.411 No</td>
<td>−1.24 −0.62 0.000 Yes</td>
<td>1.61 0.81 0.001 Yes</td>
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<td></td>
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<tr>
<td>BC</td>
<td>−1.30 −0.65 0.022 Yes</td>
<td>−1.21 −0.60 0.520 No</td>
<td>0.84 0.42 0.003 Yes</td>
<td>−1.11 −0.55 0.006 Yes</td>
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<tr>
<td>ABC</td>
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<td>−1.08 −0.54 0.563 No</td>
<td>0.79 0.39 0.004 Yes</td>
<td>−0.94 −0.47 0.014 Yes</td>
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<td>Standard deviation = 0.92 %</td>
<td>Standard deviation = 3.6 %</td>
<td></td>
<td>Standard deviation = 0.39 %</td>
<td>Standard deviation = 0.60 %</td>
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<table>
<thead>
<tr>
<th>Term</th>
<th>Al₂O₃</th>
<th>P</th>
<th>LOI</th>
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<td>Effect Coef p-value Sig.</td>
<td>Effect Coef p-value Sig.</td>
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<td>−0.37 −0.19 0.003 Yes</td>
<td>−0.005 −0.003 0.515 No</td>
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<tr>
<td>B</td>
<td>0.042 0.02 0.654 No</td>
<td>0.009 0.004 0.282 No</td>
<td>0.17 0.08 0.201 No</td>
</tr>
<tr>
<td>C</td>
<td>0.13 0.06 0.192 No</td>
<td>−0.002 0.001 0.851 No</td>
<td>0.04 0.02 0.0734 No</td>
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<tr>
<td>AB</td>
<td>−0.13 −0.06 0.187 No</td>
<td>0.005 0.003 0.505 No</td>
<td>−0.03 −0.17 0.022 No</td>
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<tr>
<td>AC</td>
<td>0.04 0.02 0.652 No</td>
<td>−0.016 0.008 0.071 No</td>
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<tr>
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<td>−0.004 0.002 0.579 No</td>
<td>−0.28 −0.14 0.050 Yes</td>
</tr>
<tr>
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<td>−0.12 −0.06 0.229 No</td>
<td>0.005 0.003 0.513 No</td>
<td>−0.04 −0.2 0.726 No</td>
</tr>
<tr>
<td>Standard deviation = 0.18 %</td>
<td>Standard deviation = 0.02 %</td>
<td>Standard deviation = 0.24 %</td>
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</table>

grades of Fe, SiO₂, Al₂O₃ and P were of 59.4 wt.%, 7.4 wt.%, 1.6 wt.%, 0.1 wt.%, respectively. Based on the promising previous results, the introduction of a selective flocculation/magnetic separation step after de-sliming of iron ore feed in industrial flotation plants, besides the recovery of values from dams in the Quadrilátero Ferrífero could be evaluated. Afterward, the obtained concentrates could be added with pellet feed in the appropriate proportion in the pelletizing industrial process. The consumption of lump ore from the Noamundi mines (Tata Steel, Jamshedpur ultra) was reduced by 10-15 % after briquetting the slimes, which increased the sustainability in Indian iron and steel industry [34].

4. Conclusions

Based on the characterization studies and selective flocculation/magnetic separation performed with an iron ore slimes sample, it was concluded that 75 % of the particles were below 10 μm and the specific weight was 4.17 g/cm³. The main identified minerals were: goethite, hematite, quartz and kaolinite. The material grades were: Fe (51.4 wt.%), Al₂O₃ (~3.4 wt.%), P (0.097 wt.%) and LOI (~7.0 wt.%). At the optimized condition of the selective flocculation/magnetic separation (pH 10.5, magnetic field = 3575 G and starch concentration = 50 g/ton), a mass recovery of 48.4 % and a concentrate with 59.4 wt.% Fe, 7.4 wt.%
SiO$_2$ and 1.6 wt.% Al$_2$O$_3$ was achieved. However, pelletizing studies using the obtained concentrates with the addition of appropriate proportions in pellet feed must be done.

**Conflict of interest**

The authors declare no conflict of interest.

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