PROCESSING OF IRON ORE FINES FROM ALSWAWEEN KINGDOM OF SAUDI ARABIA

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Abstract: Iron ores located in the Alswaween area (Saudi Arabia) are of finely disseminated nature. They require ultrafine grinding for considerable degree of liberation. In this paper, different upgrading techniques were tried for their processing. The applied upgrading techniques included selective flocculation and column flotation as recent efficient technologies in fines upgrading. Each technique was investigated and optimized separately. Results showed that neither selective flocculation nor column flotation can be successfully used alone to produce high quality iron concentrate especially when using iron ore of size fraction 100% -0.075 mm. The best quality concentrate was found to have 55% Fe and 57% Fe when applying selective flocculation and column flotation, respectively at their optimum operating conditions. Meanwhile, the previously obtained results can be significantly improved when grinding the ore below 45 µm and applying selective flocculation as cleaning step for the concentrate obtained from the column flotation. Thus, it is possible to obtain concentrate having iron content of 63.55% Fe with 52.3% yield, which means an iron recovery in concentrate of ~80%.

Keywords: iron ore fines, flocculation, column flotation, selectivity, dissemination, starch

Introduction

Iron and its products as steel and cast iron are key players in many industries. The main supply of iron and its products are ores with minor recycled amounts. Consequently, research concerned with the different possible processing techniques of run-of-mine iron ores and iron tailings is in progress (Pradip, 1994; Stephen and Misty 2007; Royand and Das, 2008). Furthermore, the increasing market pressure for improved quality has forced iron concentrate producers to re-examine their process flow-sheets and evaluate alternate or supplemental processing routes. For example, the requirement for higher quality pellets imposes that the silica content should be lowered...
to levels ranging from 2.0% SiO₂ to below 1.0% SiO₂ (Elmidany and Ahmed, 2008). Thus, it can be said that although there are many typical techniques for upgrading of iron ores (Colombo, 1986; Luo et al., 2005; Elmidany and Ahmed, 2008), the previously mentioned restricted industrial specifications coupled with the depletion of the high-grade ores make it difficult to select suitable upgrading flowsheets. This can be considered as a challenge, knowing that the techniques applied for successful processing of ultra-fine low grade iron ores are limited and require sharp operating conditions (Weissenborn et al., 1994a, 1994b; Ahmed et al., 2007). This challenge is typical in Saudi Arabia. There exist huge reserves of iron ore in the Alswaween area. The valuable minerals are highly disseminated in the associated gangues. To liberate both components from each other it requires ultrafine grinding. The finely ground ores are usually difficult-to-process with classical upgrading technologies (Stephen and Misty 2007; Royand and Das, 2008). As a result this work aims at applying recent upgrading technologies like selective flocculation and/or column flotation to recover the iron bearing minerals from the gangue minerals of the Alswaween area ore. Selective flocculation and column flotation are considered for that ore because they are among the most efficient techniques applied for treatment and separation of valuable minerals from their associated gangues at high degree of fineness (Iwasaki and Lipp, 1982; Rao et al., 1985; Arol and Iwasaki, 2003; Ahmed et al., 2007). During testing of the ore using such techniques to upgrade the ore, different operating parameters will be investigated. This includes investigating the pH, dose of dispersant (sodium silicate) in addition to the dose and type of flocculant (starch) as parameters affecting selective flocculation technique. On the other hand, parameters considered in studying the column flotation for the same purpose will include collector dose and flotation pH. As a final step in the study, a set of experiments will be implemented to answer the question what will be the effect of the two techniques, that is column flotation and selective flocculation, if they do exist at the same iron processing flowsheet?

Experimental

Iron ore sample preparation

A Saudi iron ore sample from the Alswaween area was selected for this study. The sample was subjected to primary and secondary crushing using jaw and roll crushers, respectively. The secondary crushed product was further ground in a ball mill working in a closed circuit with a 5 cm diameter cyclone rig. The cyclone operating conditions were adjusted to make cut off at 0.075 mm or 0.045 mm size fraction. The purpose of the ball mill close circuit was to yield a 100% -0.075 or 100% -0.045 mm product that will be considered as a feed for a series of selective flocculation and column flotation laboratory tests. The uppermost liberated sample (100% -45 µm) was upgraded using a sequence of column flotation followed by selective flocculation, applying the predetermined optimum conditions for each of them i.e. when using the 100% -75µm feed, to improve the process recovery, and thus its economics.
Characterization of the iron ore and its concentrates

The considered iron ore sample was physically and chemically characterized. Physical characterization of the sample was conducted by size analyses and petrographic microscope studies in addition to XRD testing to identify the mineralogical distribution and nature of dissemination among the valuable minerals and their associated gangues. However, mineralogical composition of the sample was conducted by X-ray diffraction patterns of the specimen, using X-ray Philips-PW 1010 diffractometer operated with a cobalt radiation target having Fe-filter at 30 kV and 20 mA. During the investigation, characterization was implemented by a routine chemical analysis. In this analysis, Fe was determined using standard method of sample fusion and dissolution (Ahmed et al., 2007).

Selective flocculation tests

In this series of tests, the procedure adopted by Ahmed and his coworkers (2007) was strictly followed. In this procedure, sample dispersibility in aqueous solution was considered as a prerequisite for flocculation. As a result the optimum pH and sodium silicate dosage for highest dispersibility was first investigated. In this series, samples (100% -0.075 mm) were agitated in distilled water for 5 min at 1500 rpm in a 2-dm³ container (at 0.5% solid) with the required dosage of sodium silicate at the desired pH (HCl and NaOH as pH regulators). The sample was allowed to settle freely in a 0.5-dm³ cylinder for 3 minutes. The mass settled after 3 min was then determined. The percent dispersion was calculated by expressing the mass still dispersed after three minutes as mass percent compared to the sample total mass (Ahmed et al., 2007). After determining the optimum dispersion conditions different series of selective flocculation tests were carried out. The series was designed to study effect of starch type and dosage. Different types of starches (potato, corn, wheat, and dextrin) were used as natural polymers. They were freshly prepared as 1% solutions in an alkaline media. A 1% starch solution was prepared by dissolving 1 g of starch and 0.5 g of sodium hydroxide to a volume of 100 cm³ of distilled water. The resultant solution was heated to 84 °C and then rapidly cooled to room temperature using ice. The heating and cooling of the solution was carried out within five minutes. In each series the dispersed sample was transferred to a 38 cm in length and 4.7 cm in diameter glass cylinder fitted with side outlets at 4 cm and 11 cm from the bottom. The required dose of flocculants was added from a slow running pipette for further one min. The set-up was inverted 10 times at 180° and then left to settle for 3 min. The non-flocculated fraction (tailing) was withdrawn through the side outlets. Both the flocculated (concentrates) and non-flocculated fractions (tailings) were separately collected, dried, weighed and subjected to necessary chemical analyses as previously mentioned.
**Column flotation of iron samples**

The column flotation setup used in this study is shown in Fig. 1. For each test, 3 kg of the ground iron ore (100% -0.075 mm feed) was conditioned (for 2 min) in 45-dm³ tank at 60% solids at alkaline pH for depressing gangue bearing minerals using 0.3 g/Mg sodium silicate. Later on, the pulp was further conditioned for 4 minutes with the targeted dosage of collector. The conditioned pulp was then diluted to 10% solids by weight and the required dose of frother was added. The slurry was next fed into the column by pumping until the pulp level reached a defined fixed height where the rest of the column height was assigned for the mineralized froth. In the whole series of tests, the froth height was kept at approximately 0.25 m. As a result slurry pumping rates as a feed to the column were calibrated to keep constant level allowing froth to form above this level. Air was introduced to the column for 10 minutes. Concentrate was collected and the slurry left in the column and tanks were collected as flotation tailing. Flotation and conditioning parameters (Table 1) were held constant throughout the test unless otherwise stated. Chemicals used in flotation tests were oleic acid as iron collector, sodium silicate as silica depressor, and MIBC as frother. All these chemicals were of reagent grade.

<table>
<thead>
<tr>
<th>Table 1. Parameters of Alswaween iron ore flotation using column flotation</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Conditioning</strong>*</td>
</tr>
<tr>
<td>Parameter</td>
</tr>
<tr>
<td>Solids %</td>
</tr>
<tr>
<td>Time (minutes)</td>
</tr>
<tr>
<td>pH</td>
</tr>
<tr>
<td>Impeller speed (RPM)</td>
</tr>
<tr>
<td>Solids charge (g)</td>
</tr>
<tr>
<td>Water type</td>
</tr>
</tbody>
</table>

*Operating conditions were selected according to previous studies (Flint et al., 1992; Bhaskar et al., 1993)
Results and discussion

Sample characterization

Complete chemical analyses of the original iron sample indicate that the sample contains 41.58% Fe. The sample was high in SiO$_2$ and CaO contents (29% and 4.2%, respectively, Table 2). The contents of MgO, MnO, K$_2$O, Na$_2$O, Cl, ZnO, TiO$_2$, P and Al$_2$O$_3$ were minor. On the other hand, the XRD pattern shows that the iron bearing minerals are mainly hematite and magnetite while the main gangue minerals in the sample are quartz and calcite (Fig. 2).

Table 2. Complete chemical analyses of the original investigated iron sample

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Fe%</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>CaO</th>
<th>MgO</th>
<th>Mn</th>
<th>TiO$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>41.58</td>
<td>29.1</td>
<td>1.5</td>
<td>4.2</td>
<td>1.5</td>
<td>0.07</td>
<td>0.15</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Constituent</th>
<th>S</th>
<th>P</th>
<th>K$_2$O</th>
<th>Na$_2$O</th>
<th>V$_2$O$_5$</th>
<th>ZnO</th>
</tr>
</thead>
<tbody>
<tr>
<td>%</td>
<td>0.11</td>
<td>0.31</td>
<td>0.016</td>
<td>0.021</td>
<td>0.002</td>
<td>0.005</td>
</tr>
</tbody>
</table>
Microscopic liberation analysis (Fig. 3) of the ore shows that in the coarser size fractions percentage of interlocking is very high where the percentage of free iron minerals in the first coarse fractions is less than 35%. However, complex interlocking...
and disseminated nature of the ore can be seen as below 87% of the iron bearing minerals can be liberated below the 45 μm size. Achieving high purity concentrate in beneficication of this ore is probably quite difficult due to the complexity of interlocking. Proper comminution is required to break the interlocking and attain good liberation in this case.

**Iron upgrading applying selective flocculation**

**Dispersibility tests**

Figures 4a and 4b show the effect of pH and sodium silicate dosage on iron dispersibility. Figure 4a indicates that an improvement of the ore dispersibility can be obtained by increasing the pH. The phenomenon can be fitted with a polynomial curve with a correlation coefficient of 0.99. This indicates that the iron dispersibility is directly proportional to the pulp pH with a plateau reached at pH of 10.5–11. On the other hand, at this ideal pH (10.5), an increasing the dose of sodium silicate improves the dispersibility for the whole-investigated pH range (Fig. 4b). Thus, from both Figures 4a and 4b, it can be concluded that the optimum dispersibility conditions are at pH 10.5 and sodium silicate dose of 1 kg/Mg.

**Fig. 4. Effect of pH and sodium silicate on iron ore dispersibility a) effect of pH at sodium silicate dosage of 1 kg/Mg, b) effect of sodium silicate dosage, at pH = 10.5 used feed in both series is 100% – 0.075 mm**
Selective flocculation optimization

Effect of flocculant type

Basing on the results presented in Figure 4, several preliminary flocculation tests, using different types of starches as flocculants, were conducted. The operating conditions applied in this series were as follows: pH 10.5, 1 kg/Mg sodium silicate, solid percent 5%, flocculant dose of 1.0 kg/Mg and iron feed size is 100% -0.075 mm. The results of this series show that the different used flocculants return different responses and potato starch provides the best results among the group in reference to the concentrate yield and iron assay and hence the iron recovered in the concentrate fraction (Fig. 5). The iron concentrate obtained when using potato starch was containing ~55% Fe with iron recovery approaching 80% while all other starches returned worse responses. It can be seen that the obtained results are supported with literature interpretations (Subramanian and Natarajan, 1991; Weissenborn et al., 1994 b; Panda et al., 2011), where adsorption of potato starch on the surface of the iron bearing minerals is higher compared to that of other starches at the conditions under investigation.

Effect of flocculant dose

The hitherto results concluded the preference of potato starch in selective flocculation of the iron sample under investigation. As a result optimization of the potato starch dosage was implemented. Results of such tests are shown in Figure 5. It can be seen that the most pure iron concentrate can be attained at the dosage of potato starch of 1 kg/Mg. This concentrate has iron content approaching 55% Fe with iron recovery of approximately 80%. However, potato starch doses higher than 1 kg/Mg hampers the process selectivity (Table 6). This may be attributed to liberation considerations.
Column flotation of iron ores

Optimization of selective flocculation, as a technique for processing of the investigated iron ore, lead to the conclusion that it is not possible to obtain iron concentrates of commercial applications. Therefore, the column flotation was adopted as an alternative for selective flocculation. The technique will be investigated regarding the pH values suitable for iron flotation from their gangues besides the most suitable amount of oleic acid collector that should be used.
Effect of flotation pH

The quality and quantity of the iron concentrate obtained when it is floated using oleic acid as collector applying column flotation technique varies with changing flotation pH. In the case of the investigated iron ore, the results show that pH 6 represents the optimum pH (Fig. 7). This is because at this pH the cleanest concentrate was attained. At pH values higher or lower than 6 the iron concentrate obtained have neither good assays nor high recoveries. As a result in the subsequent study pH value of 6 will be considered. In fact this may be attributed to the adsorption of oleic acid collector on the surface of the iron bearing minerals (Flint et al., 1992; Bhaskar et al., 1993; Xinghua et al., 2012).

Effect of collector dose

Considering the results shown in Figure 7, further tests were implemented for the purpose of knowing the most suitable dosage of oleic acid as a collector. Results obtained from this series are presented in Figure 8. It clearly shows that the most clean iron concentrate can be achieved at collector dosage of 1.2 kg/Mg. At this collector dosage a concentrate having iron assay of ~57 % Fe with a recovery of approximately 86% which is much better compared to the purest concentrate achieved in the case of selective flocculation. In spite of the preference of the results obtained when applying the column flotation technique, yet still the obtained concentrate is beyond the commercial application for pellets production. To solve this problem further improvement will be tried by further grinding of the ore for achieving higher degree of liberation.

![Fig. 8. Effect of oleic acid collector dosage on the column flotation response of iron ore](image-url)
**Process further improvement**

From the hitherto discussed results, it is obvious that the maximum grade that can be achieved using either selective flocculation or column flotation alone is low i.e. 55% Fe and 57% respectively at the optimum conditions. Even when the floated concentrate was cleaned by selective flocculation, the concentrate quality is still low (Fe% = 58.9%). This is mainly attributed to the low degree of liberation of the considered iron ore. Thus seeking for a higher degree of liberation, the ore was further ground to 100% -45 µm as previously mentioned in the experimental section. Table 3 shows different tests applied for the iron ore sample considered previously obtained results and optimum conditions.

**Table 3. Different tests implemented for improvement of iron concentrate specifications to meet commercial applications**

<table>
<thead>
<tr>
<th>Test #</th>
<th>Technique</th>
<th>Operating conditions</th>
<th>Obtained iron concentrate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Yield, %</td>
</tr>
<tr>
<td>1</td>
<td>Column flotation followed by selective flocculation</td>
<td>–0.075 feed at previously determined optimum conditions</td>
<td>56.3</td>
</tr>
<tr>
<td>2</td>
<td>Selective flocculation alone</td>
<td>–0.045 feed at previously determined optimum conditions</td>
<td>61.3</td>
</tr>
<tr>
<td>3</td>
<td>Column flotation alone</td>
<td>–0.045 feed at previously determined optimum conditions</td>
<td>62.7</td>
</tr>
<tr>
<td>4</td>
<td>Column flotation followed by selective flocculation</td>
<td>–0.045 feed at previously determined optimum conditions</td>
<td>52.3</td>
</tr>
</tbody>
</table>

**Conclusions**

The results of this investigation clearly established that effective separation of iron ore concentrate from finely disseminated iron ore sample of Alswaween area in Saudia Arabia by column flotation and selective flocculation is possible. The effectiveness of separation is greatly influenced by the feed particle size and pH (in both techniques), type of flocculant (in flocculation), collector dosage (in flotation). The separation efficiency is better in case of fine particle size because of liberation considerations of the iron particles from the associated gangues. The results of the investigations showed that it is possible to obtain concentrate having iron content of 63.55% Fe with 52.3% yield, which correspond to an iron recovery in concentrate of ~80% when grinding the ore to below 45 µm and applying selective flocculation for the column flotation concentrate. This product can be utilized in the steel plant as a sinter feed material.
Acknowledgment

This paper was funded by the deanship of scientific research (DSR), King Abdulaziz University, Jeddah, under grant no. (15_135_D1432). The authors, therefore, acknowledge with thanks DSR technical and financial support.

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