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# Application of magnetic separation and reverse anionic flotation to concentrate fine particles of iron ore with high sulfur content

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**Abstract:** The sulfur content in iron ore causes technical problems in the process of sintering iron ore in steel and alloys, and environmental problems in discharging the tailing. The major challenge in the iron ore processing plant is handling the finer particles. The key objectives of this research included the concentration of Band Narges Mine iron ore (< 150 µm) as well as the reduction of the sulfur content to achieve a marketable product. First, the mineralogical characterization of iron ore was established, which showed that $Fe_3O_4$, $SiO_2$, and CaO were the predominant minerals in the ore body. Moreover, magnetite particles with a size of < 150 µm were mainly locked into the associated gangue mineral. Second, metallurgical experiments were conducted, including magnetic separation and froth flotation. To increase the iron grade and recovery and decrease the sulfur content, two separate process flowsheets were tested, three steps of magnetic separation with a magnetic field strength of 2000 G were used in the first process flowsheets, followed by regrinding to < 74 µm and application of a three-stage reverse flotation. The overall iron grade and recovery were 76.38% and 67.9%, respectively, from this flowsheet. A five-stage successive reverse flotation followed by three stages of magnetic separation at 1000 G was carried out in the second flowsheet. The final recovery and grade of iron for this flowsheet were 77.15% and 64.3%, respectively. The ultimate content of sulfur was estimated at 0.74%.

*Keywords:* iron ore, fine particles, magnetic separation, reverse anionic flotation, sulfur

## 1. Introduction

One of the most common elements in the earth's crust is iron. Magnetite ($Fe_3O_4$), Siderite ($FeCO_3$), and hematite ($Fe_2O_3$) minerals are the primary sources of iron for industrial purposes. The steel industry demands high-purity iron with low impurity content to manufacture high-quality steel and minimize operation and maintenance costs (Arol & Aydogan, 2004; Filippov et al., 2014). The final size of comminution of mineral ore depends on the nature of mineralization such as the grade, grain size, mineral distribution in the ore deposits, and associated gangue minerals. The liberation of iron minerals from the gangue mineral usually requires crushing and grinding. The purification and handling of fine iron minerals are challengeable, as it makes the process flowsheet more complicated by adding multiple upgrading steps to the flowsheet or even various processing methods (Mathur et al., 2000).

Magnetic separation is the most common method used for the beneficiation of iron ores including magnetite and hematite. This simple and inexpensive method efficiently employs the magnetic susceptibility difference between ferro- or paramagnetic minerals (e.g. magnetite or hematite) with diamagnetic ones (e.g. silicate and carbonate minerals) to separate them (Barry A. Wills & Finch, 2015). Generally, dry or wet low-intensity magnetic separators are used for ferromagnetic minerals, whereas high-intensity magnetic separators are widely used for paramagnetic mineral separation. (Dwari et al., 2013). Magnetic, gravity, and hydrodynamic drag forces are the major forces that control the overall reaction of particles in the magnetic field. While gravity and hydrodynamic forces try to throw away particles from the magnetic field region, the magnetic force is responsible for attracting magnetic

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particles. The final efficacity of the magnetic separation will therefore be determined by the predominance of one of the mentioned forces. Particle sizes are one of the successful criteria for evaluating the governing power. For coarse particles, the gravity force is dominant, and for finer particles, the hydrodynamic force is dominant (Arol & Aydogan, 2004; Filippov et al., 2014).

The importance of fine iron ore beneficiation will increase in the future because the lower grade ore deposits will require liberation of iron ore minerals at finer sizes. Generally, this fine iron ore will be recovered to produce a pelletizing concentrate with very strict chemical and physical specifications. In addition, because of the increasing demand for iron ore, there are now more opportunities to produce by-product iron ore from mining operations producing other commodities. In the past, the associated iron ore minerals would report to final tailings, but now there is potential value to be realized from the by-product revenue. Pelletizing concentrates are currently produced mainly by various combinations of flotation and magnetic separation. The selection of the beneficiation route will depend on ore mineralogy and considerations around plant capacity and final concentrate quality. The main economic iron minerals are magnetite and hematite, where hematite is paramagnetic and magnetite is ferromagnetic. Therefore, it means that magnetic separation can be applied in principle to all fine iron-ore beneficiation plants. Whereas flotation has a considerable advantage over magnetic separation, the real advantage of flotation over magnetic separation in fine iron-ore beneficiation is that treatment of -10 µm iron ore is possible in flotation. The feed is deslimed at 10 µm and the -10 µm stream is considered to be final tailings, even though there is often a significant amount of contained iron ore (Dworzanowski, 2012). Flotation of iron ore is a complex physicochemical separation process widely used as the main technique to beneficiate non-magnetite iron ores. Also, it is usually used to further upgrade magnetite iron ore as well (Bahram Rezai et al., 2010; Eskanlou et al., 2018; Nakhaei & Irannajad, 2018). This method can be categorized as direct anionic flotation of iron oxides and reverse anionic or cationic flotation of associated gangue minerals, mainly quartz, and sulfur. Reverse cationic flotation is the most common method used for the flotation of gangue minerals, mainly quartz while depressing iron oxides by starch in alkaline media. The main disadvantage of using this method include 1) Losing iron in the de-sliming stage 2) The high cost of amine collectors such as primary fatty amines and quaternary ammonium compounds (Ma, 2012; Yuhua & Jianwei, 2005). Thus, reverse anionic flotation was developed to address the issues regarding reverse cationic flotation. Instead of fatty amines, fatty acids were used as collectors. Besides, the method is less sensitive to the presence of slimes, therefore it is not needed to do the desliming process before flotation. Some studies reported that higher recovery and selectivity can be obtained via reverse anionic flotation (Araujo et al., 2005; Eskanlou et al., 2019; Filippov et al., 2010).

The presence of large quantities of fine particles in the feed does not only causes the loss of a substantial amount of iron ore but also poses serious long-term environmental problems. Enrichment of iron ores with locked impurities always requires comminution for mineral release and during this process, significant amounts of fines are generated, whereas processing fine particles of iron ore is difficult. Thus, it is necessary to search for innovative methods for purifying iron ore to avoid the loss of enormous amounts of iron ore. In this study, the beneficiation of a magnetite iron ore (Band Narges) was studied to concentrate fine iron ore and eliminate impurities. One of the main objectives of this study was how to process fine iron ore particles with a high content of sulfur. In this experiment, two separate flow sheets were designed to purify the fine particles of iron oxide. Magnetic separators accompanied by the flotation process were used in the first flowsheet to isolate impurities. In the second flowsheet, flotation was initially used followed by magnetic separation to obtain the desired products. Also, the effects of particle size, pH, sodium silicate, and collector synergism were investigated in the flotation phase to improve the floatability of gangue minerals.

## 2. Materials and methods

### 2.1. Materials

Around 100 kg iron ore was received by Lorestan University from Band Narges Mine (Kashan, Iran) for the mineralogical studies, and the feasibility analysis of the beneficiation process. The ore was crushed using a jaw crusher, cone crusher, and ball mill to liberate the valuable minerals from associated gangue minerals as well as to provide an acceptable range of particle size for physical and physicochemical separations. To analyze the chemical composition and phase characterization ring mills were employed for further size reduction (< 75 µm) of the ore.

The chemical composition study of the ore sample was carried out using X-ray fluorescence spectroscopy (XRF) by the Lorestan University (Lorestan, Iran) Philips PW 2404 apparatus. XRF analysis results showed that iron oxides in the form of $Fe_2O_3$ constituted about 58% of the sample by weight, FeO (20.4%), $SiO_2$ (15.2%), and CaO (10.1%), respectively, were the second and third main oxides. In addition, the sample had small quantities of oxides of Mn, Al, and Mg. There was 1.9% S in the ore sample, which is a significant detrimental factor for iron ores and causes sale penalties. The phase characterization of the ore was performed at Lorestan University using a Philips PW3710 and X'Pert MPD X-ray powder diffractometer (XRD) operated with Cu-Ka radiation at 40 kV and 30 mA. Magnetite and hematite were identified as the abundant economical iron-bearing minerals, while quartz, calcite, and pyrite were observed as gangue minerals by the XRD analysis.

Table 1. Chemical composition of the sample by the XRF technique (Wt%)

| **Compound** | $Fe_2O_3$ | FeO | SiO2 | CaO | S |
|---|---|---|---|---|---|
| **Content (%)** | 58 | 20.4 | 15.2 | 10.1 | 1.9 |

### 2.2. Methods

#### 2.2.1. Crushing, grinding and optical microscope studies

The most expensive parts of mineral processing are crushing and grinding. The choice of suitable size particles to liberate valuable minerals from associated gangue helps in avoiding either over-grinding or the absence of mineral unlocking. Over grinding do not only increases the cost of processing but also creates fine particles which show a negative impact on the product grade and recovery (Asadi et al., 2018). On the other hand, inappropriate liberated minerals cause the rejection of valuable minerals into the tailings parts. The degree of mineral freedom should therefore be calculated. Optical mineralogy studies were conducted to determine the degree of freedom of iron ore particles and to choose the appropriate size of grinding. Representative samples from different size fractions (-63, 63-74, 74-88, 88-105, 105-125, and 125-150 µm) were used for the mineralogical studies. Figure 1 displays a microscopic optical image at a fraction of 125-150 µm. It can be seen from Fig. 1 that magnetite mineral was fairly liberated from the associated gangue mineral at fraction of 125-150 µm.

The Davis tube test was used for 15 g of representative samples of the original sample at four size fractions at field strength 2000 Gauss (Fig. 2) to confirm the results of the optical mineralogy analysis. The concentration of magnetite for each Davis tube test was analyzed using the XRF to determine the grade. The recovery was calculated using Eq. 1 (Dehghani et al., 2013; Khosravi et al., 2020; Rahimi et al., 2012):

$$R\% = \frac{C \times c}{F \times f} \times 100 \tag{1}$$

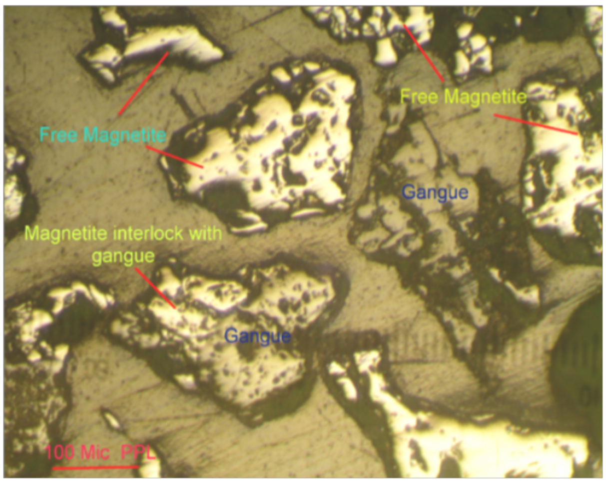


Fig. 1. Optical microscopic image of ore sample in 125-150 µm size fraction

where $C$ is the weight of the concentration, $c$ is the grade of the concentration, $F$ is the weight of the feed, and $f$ is the grade of the feed.

According to the results in Fig. 2, the maximum separation efficiency was achieved at fraction of 100-150 µm. In addition, the results indicated by decreasing particle size, the recovery of iron decreases. Because despite larger particles, the smaller particles appeared to obey the water flow instead of the magnetic force.

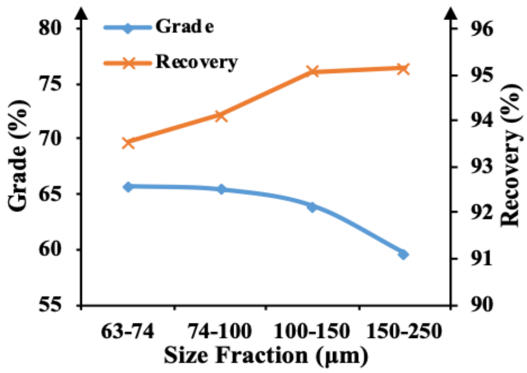


Fig. 2. Results of Davis tube test at various size fractions

### 2.2.2. Dry magnetic separation

To investigate the magnetic separation efficiency of the fine iron particles, it was decided to perform dry magnetic separation using Model MIH for the sample at three magnetic field intensities of 1000, 1500, 2000 G in four size fractions of 1000-2000, 150-1000, -150, and -74 µm. For all the tests, the rate of feeding was set at 10 kg/min.

### 2.2.3. Reverse flotation experiments

Flotation is typically applicable for particles of a finer size where other physical approaches fail to concentration it. Reverse anionic flotation experiments were performed using a Denver flotation machine in this study. At an agitation rate of 1100 rpm and pulp density of 20 Wt.%, the experiments were carried out in a 5-dm$^3$ cell. Preparation time was set at 5, 2, and 3 min, respectively, after adding a depressant, a collector, and froth reagents. Alke 742 (anionic collector based on fatty acids) produced at the Isfahan Copolymer Chemical Co. Isfahan, Iran, was used as the collector, Dirol as a collector/frother from a local producer reagent, and iron oxide as a sodium silicate depressant. The pH modification was performed using NaOH or $H_2SO_4$.

## 3. Results and discussion

### 3.1. Magnetic separation-flotation flowsheet

Three-stages magnetite separation followed by three-stages reverse flotation to purify the mineral was performed after the crushing and grinding. The flowsheet for this phase is shown in Fig. 3.

### 3.1.1. Magnetic separation experiments

#### 3.1.1.1 Rougher stage of magnetic separation

Figure 4 (a-b) demonstrates the effects of particle size and strength of the magnetic field on the grade and recovery of iron particles. As can be seen in Fig. 4(a), the highest grades for all magnetic field intensities were obtained at fractions of < 150 µm. A reasonable amount of iron minerals was released from gangue minerals in this size fraction. Almost all particles below 74 µm were liberated and by decreasing particles sizes, more gangue particles were reported to the magnetic component because smaller particles had a higher surface area, consequently, they had a higher tendency to agglomerate through various mechanisms such as increasing the electrical force between particles. In addition,

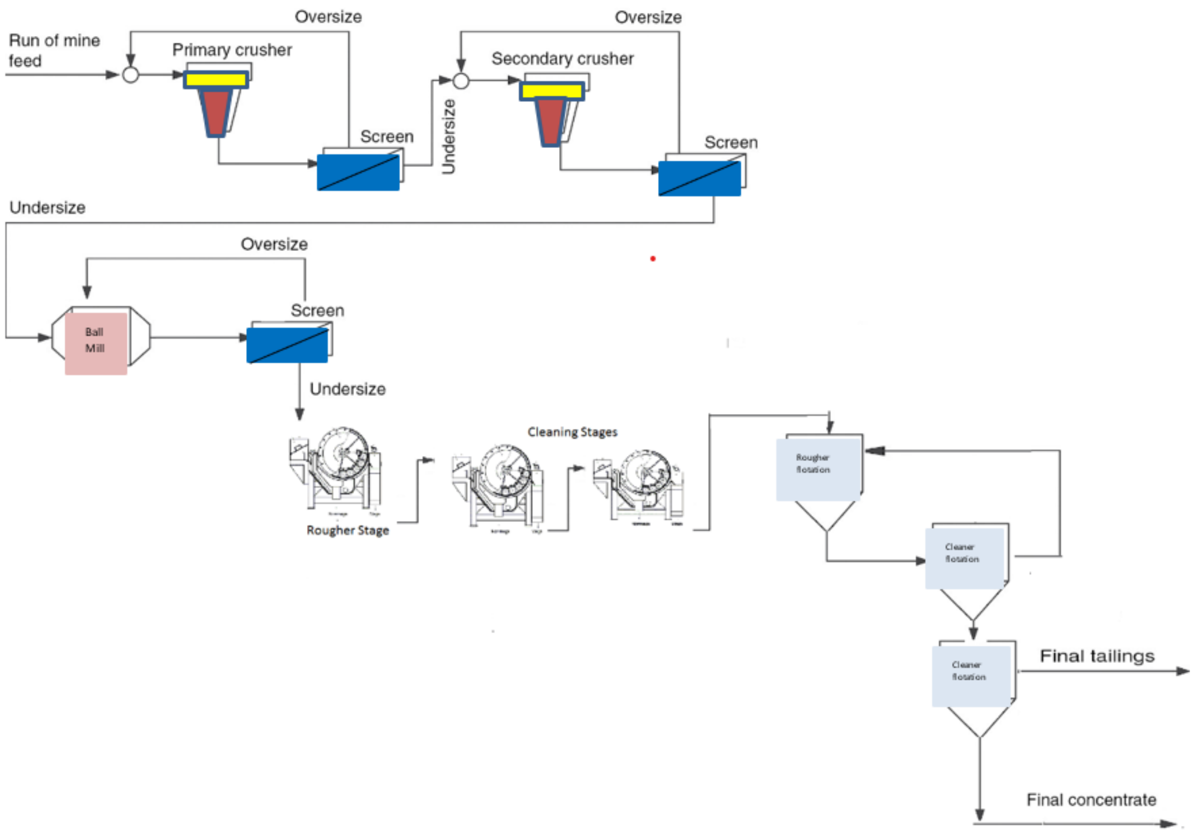


Fig. 3. Flowsheet of three-stage magnetite separation followed by three-stage reverse flotation

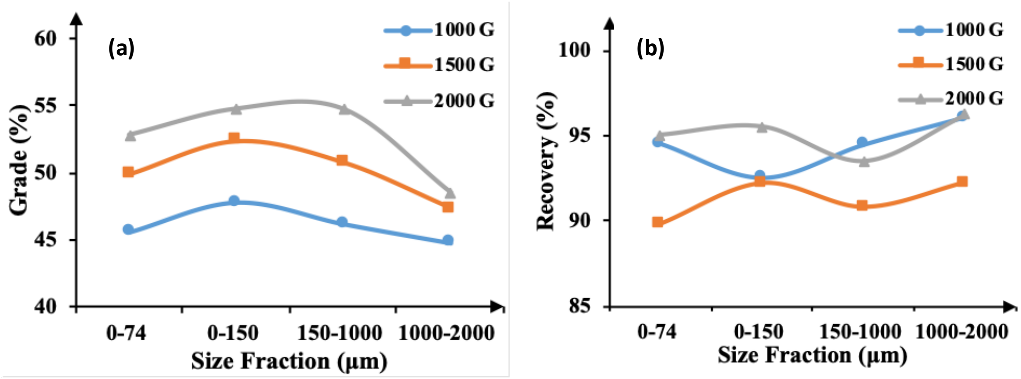


Fig. 4. Grade (a) and recovery (b) of iron ore at different size fractions and different magnetic field intensities

particle agglomeration may result in blocking iron minerals in the feeding field, thereby it prevents them to enter the orbit and report to the magnetic part. Maximum recovery of iron was achieved at 0-150 μm and 1000-2000 μm fractions. The fraction of 1000-2000 μm had a lower grade as compared to the size fraction of 0-150 μm because the degree of particle freedom was lower for coarse particles. In other words, the amount of locked iron minerals with associated gangues will increase by increasing the particle sizes, hence the grade dramatically drops. In conclusion, the best results were obtained at a magnetic field strength of 2000 G for the particle size < 150 μm.

### 3.1.1.2. Cleaner stage of magnetic separation

The steel and other associated mineral markets expect iron concentrate to have a grade of < 60% iron. To improve the iron content, the cleaning procedures were carried out on the products of the rougher stage. The grade and recovery results are illustrated in Fig. 5. As can be shown from Fig. 5, the iron grade increased from 54.74% in the rougher process to 58.65 and 60.85% in the first and second cleaner phases, respectively. Also, promising recoveries of 93.85% and 95.03% were obtained in each cleaning stage. The total recovery of magnetic separation was 85.22% which indicated a proper recovery compared to conventional metallurgical processes. However, the magnetite separation stages products had an acceptable grade and market recovery, but chemical analysis revealed that the sulfur content of the products in the concentrate exceeded the range (1.08%). Thus, it should be minimized because of its adverse impact on the steel industry. A series of flotation experiments were carried out to remove or reduce the sulfur content to solve these problems.

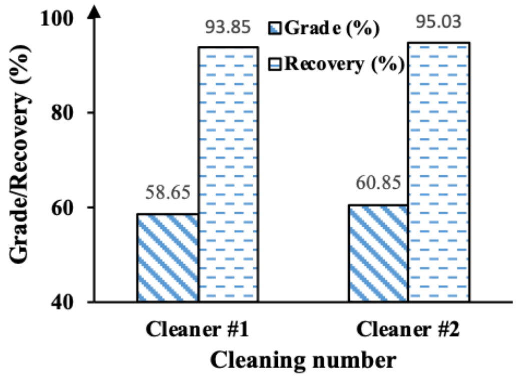


Fig. 5. Effect of cleaning stages of magnetic separation on grade and recovery (particle size of < 150 μm and magnetic field intensity of 2000 G)

### 3.1.2. Flotation experiments

Flotation experiments were conducted after the magnetic separation experiments. Flotation is the other promising method for separating iron minerals with small particle sizes from gangue minerals. This approach is particularly applied in cases where the amount of sulfur is over-limited in the ore. The flotation method is strongly dependent on particles sizes. Therefore, representative samples with size ranges of 0-150 and 0-74 μm fractions were selected for the flotation experiments (Asadi et al., 2018; Dehghani et al., 2013; Rahimi et al., 2012). Various effective parameters were considered for the flotation experiments and the optimum conditions were obtained.

#### 3.1.2.1. Effect of pulp pH

Pulp pH is another essential factor to improve the grade and recovery of minerals in the flotation. To obtain the optimum pH, the flotation experiments were done in the alkaline area (9 < pH < 11.5) using 600 g/Mg Dirol and 200 g/Mg Alke 724 as the collector and 500 g/Mg sodium silicate as the depressant. Figure 6 (a-b) demonstrates the effect of pH at two different particle sizes on the iron flotation. It can be seen from Fig. 6 that the iron grade decreases with increasing pH. Also, the grade and recovery were improved by decreasing particle sizes in the experiments. Based on the flotation performance, the highest grade and recovery were obtained at pH 9 for the smaller particles fraction.

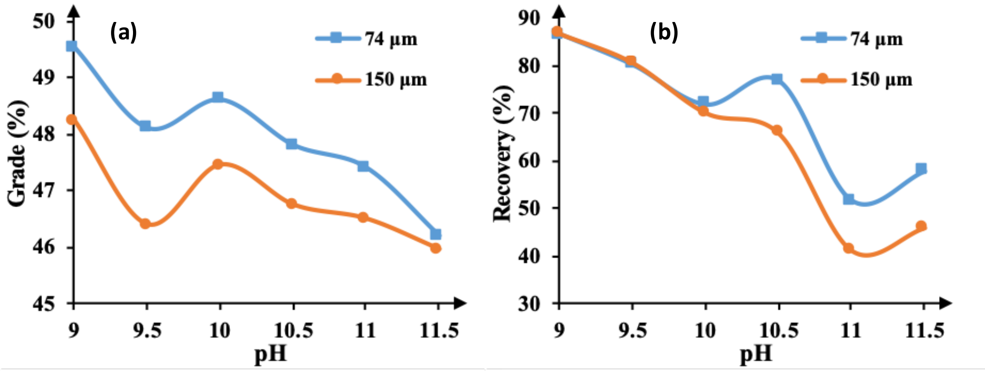


Fig. 6. Effect of pH and particle size on (a) grade (b) recovery

#### 3.1.2.2. Effect of sodium silicate dosage

Sodium silicate is a proper depressant for the reverse flotation of iron oxides (Tohry et al., 2019). Its dosage plays an important role in the depression or floatability of gangue minerals (Feng & Aldrich, 2005). The effect of this depressant was studied on flotation performance (Fig. 7). The experiments were designed and carried out using various dosages of sodium silicate (500, 600, 700, 800, 900, and 1000 g/Mg), and 200 g/Mg Dirol, 600 g/Mg Alke 724 at optimized pH of 9 for both particle sizes. The highest grade and recovery of iron ore were obtained at 600 g/Mg depressant. The lowest grade and recovery were obtained at 800 g/Mg and 900 g/Mg of the depressant dosage, respectively. The increasing depressant dosage (> 600 g/Mg) resulted in a decrease in the grade and recovery. Based on the results, the dosage of 600 g/Mg depressant was chosen as an optimum condition.

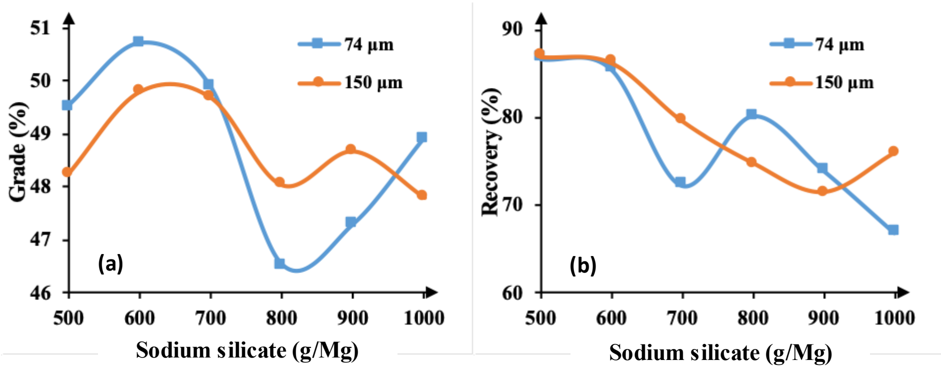


Fig. 7. Effect of sodium silicate dosage on (a) grade (b) recovery

#### 3.1.2.3. Effect of collector dosage

The effect of collectors of Alke 724 and Dirol was investigated at different dosages. At the lowest dosage of Alke 724 and Dirol, the collectors were more selective in separating iron minerals. The grade decreased by increasing the total quantity of both collectors to 400 g/Mg (Fig. 8 (a)). For both collectors, particles with size ranges of 0-74 µm showed higher grades than particles with size ranges of 0-150 µm. The results indicated that an increase in particle size decreased the ability of the heavier particles to be carried by froth. The effect of collectors' dosages on iron recovery is shown in Fig. 8 (b). It can be seen from Fig. 8 (b) that the increase in the dosage of Dirol from 200 g/Mg decreased the recovery, while no noticeable change occurred when more than 200 g/Mg of Alke 274 was used. Therefore, the combination of Alke 274 and Dirol was used to achieve the highest flotation performance at the dosage of 200 g/Mg.

### 3.1.3. Concentration of magnetic separation followed by reverse flotation

The final iron grade in magnetic separation was achieved at 60.85%. Three reverse flotation stages were carried out to increase the grade and decrease its sulfur content including one rougher and two cleaner

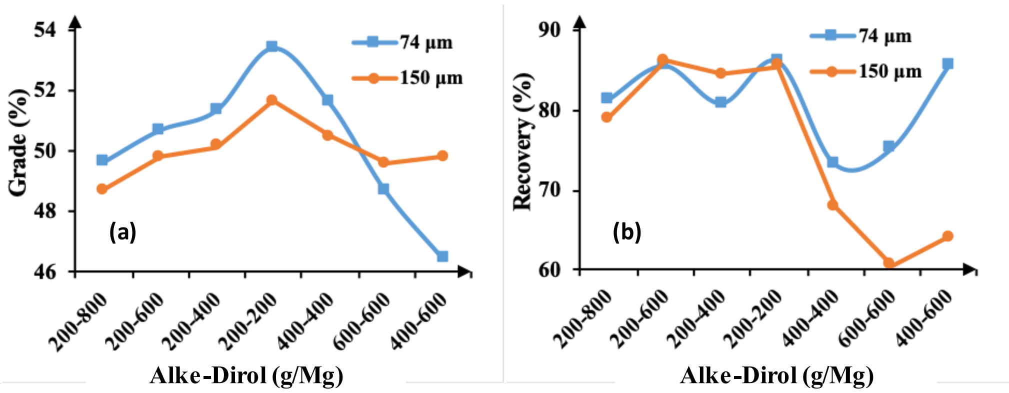


Fig. 8. Effect of collector type and dosage on flotation (a) grade (b) recovery

steps. To remove the sulfur-containing minerals, the particle size of the magnetic concentrate was reduced to less than 74 µm. The final grade of iron reached 67.9% after three flotation stages and the sulfur content decreased to 0.48% (Fig. 9). 76.38% was determined to be the final recovery of both magnetic separation and reverse flotation.

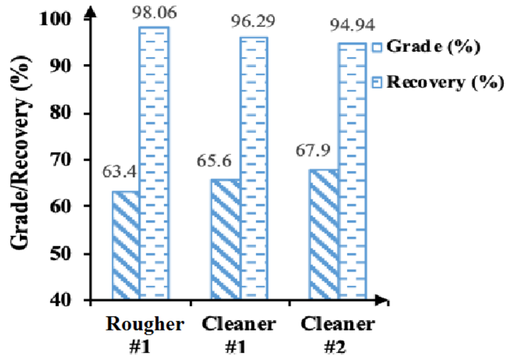


Fig. 9. Improving iron grade and recovery using flotation

### 3.2. Flotation-magnetic separation flowsheet

In the first flowsheet, the magnetic separation was followed by flotation, another flowsheet was designed as a flotation process that could be done before the magnetic separation steps. Then, the results of two different flowsheets were compared and selected the better one. After the crushing and grinding, five-stage reverse flotation was performed to purify the mineral, followed by a three-stage magnetite separation. A flowsheet for this process is shown in Fig. 10.

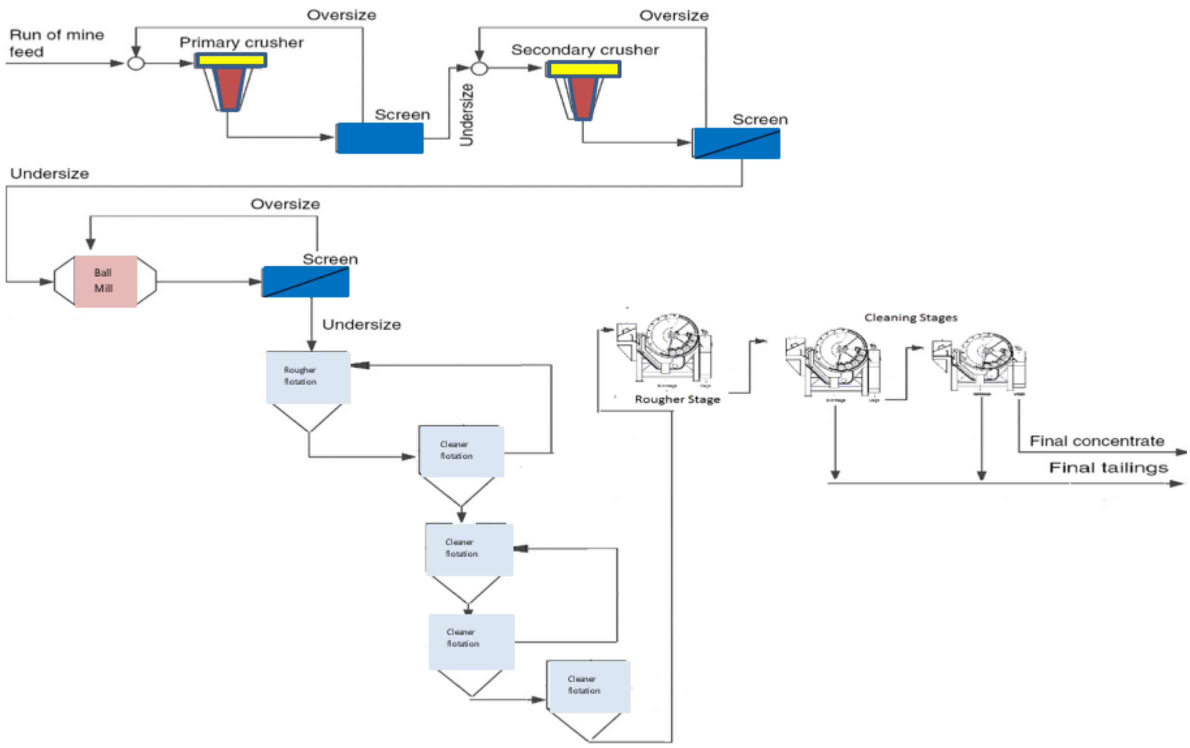


Fig. 10. Flowsheet of five-stage reverse flotation followed by three-stage magnetite

#### 3.2.1. Flotation

The flotation parameters were optimized. A rougher and four cleaning reverse flotation stages were conducted to improve the grade of iron ore products with a size of < 74 µm. In this step, the reduction of the flotation reagents dosage (e.g. collectors and depressant) were aimed. Therefore, 100 g/Mg of each collector and 600 g/Mg depressant were used in the rougher, and for the cleaning steps the dosage of each collector was reduced to 60 g/Mg and the dosage of depressant was 400 g/Mg. The results of grade and recovery were illustrated in Fig. 11. Recovery of more than 97% was obtained in each cleaning stage. The total recovery of the cleaning stages was calculated to be 93.38%. The purity of iron improved gradually from 53.42% rougher to more than 60% at the cleaner, indicating a good product for the market. Also, sulfur content was reduced to 0.95%.

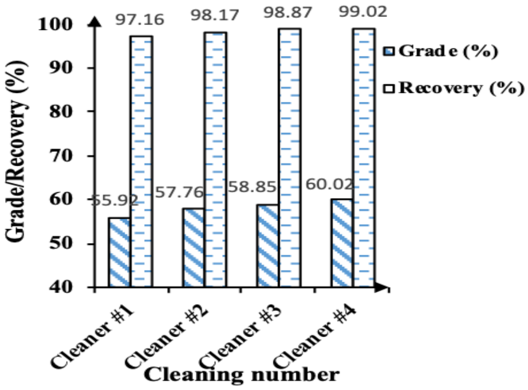


Fig. 11. Effect of consecutive cleaning flotation on iron grade and recovery

### 3.2.2. Concentration of flotation product with magnetic separation

In this process, magnetic separation was used to improve the quality of iron ore concentrate obtained by reverse flotation as a cleaning tool. To increase the iron grade with a particle size of < 74 µm, three magnetic field intensities of 1000, 1500, and 2000 G were used. The results showed that there was no significant improvement in both grade and recovery of products by increasing magnetic field intensities. Therefore, the lowest magnetic field strength, e.g. 1000 G, was chosen. In this condition, the iron grade increased to 62.9% with a corresponding recovery of 98.93%.

Three magnetic separation steps were performed to obtain more than 64 percent iron grade and decrease the sulfur content. Final iron recovery and grade of 95.5% and 64.3% were obtained, respectively, by the application of magnetic field strength of 1000 G. The final sulfur content was analyzed to be 0.74%. Also, the total recovery of the process including consecutive flotation and magnetic separation was 77.15%.

## 4. Conclusions

The type and complexity of ore, impact the selection of the method to process the ore. In addition, the factors such as the simplicity, cost-effectiveness, speed, and level of environmental pollution of the various processes must be taken into account for selecting the processing method. In this study, the combination of magnetic separation and reverse flotation was used to concentrate the iron ore from the Band Narges mine. It was intended to produce a marketable concentrate from fine iron with minimum sulfur content. Mineralogical studies and phase characterization showed that the primary mineral in the samples was magnetite. Its degree of freedom was approximately 150 µm.

Magnetic separation experiments were performed at different magnetic field intensities in both rougher and cleaning steps. The best iron recovery and grade obtained were 85.22% and 60.85%, respectively, and the sulfur content was 1.08%. A three-stage reverse flotation process was carried out to increase the iron grade and decrease the sulfur content of the magnetic product. The final grade of the product obtained was 67.9%, and the sulfur content was 0.48%. 76.38% was the overall recovery.

Reverse flotation variables were optimized. They included the particle size, pH, collector and depressant dosages. Due to the complexity of the ore mineralogy, one step of rougher flotation and four cleaning steps were performed to obtain a concentrate with a grade of 60.02% and 79.91% recovery. For the reverse flotation method, the final sulfur content was 0.95%. A three-stage low-intensity magnetic separation (1000 G) was employed to further improve the quality of the product. The results showed that the grade increased to 64.3% and the sulfur content decreased to 0.74%. The overall recovery was estimated at 77.15%.

In conclusion, the use of the magnetic separation method as a pre-concentration method for flotation was more appropriate than using the flotation method as a pre-concentration method for magnetic separation. In the magnetic separation, the particles sizes of < 150 µm were suitable for the process, while the proper particles sizes for flotation are < 74 µm. Using magnetic separation before the flotation could decrease the cost of grinding. On the other hand, the use of the flotation method as pre-concentration for magnetic separation required a total of five stages. It could increase the cost of chemicals consumed, the difficulty of the process, and the amount of environmental pollution produced. Finally, when magnetic separation is used as pre-concentration, its product has a higher grade and lower sulfur content. Overall, the first flowsheet is a better option to process iron fine particles with high sulfur content to obtain desired products for the marketplace.

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