# Beneficiation of a low-grade iron ore by combination of wet low-intensity magnetic separation and reverse flotation methods

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| Keywords | Abstract |
|---|---|
| | Beneficiation of a low-grade iron ore was investigated by combination of the low-intensity magnetic separation and reverse flotation methods. The main constituents of the representative sample were 36.86% Fe, 8.1% FeO, 14.2% CaO, 13.6% $SiO_2$, and 0.12% S based on the X-ray fluorescence, titration, and Leco analysis methods. The mineralogical studies by the X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray spectroscopy, electron probe micro-analyzer, and Fe/FeO titration methods showed that the ore minerals present in the representative sample were magnetite, hematite, and goethite, and the main gangue minerals were calcite and quartz. The effects of the operating parameters including the feed size, solid content, and drum rotation speed were investigated on the performance of the wet low-intensity magnetic separation (WLIMS). The optimum operating conditions of WLIMS were determined to be feed size = 135 μm, solid content = 40%, and drum rotation speed = 50 rpm. Under these conditions, a concentrate of 62.69% Fe grade and 55.99% recovery was produced. The tailing of WLIMS with an iron grade of 28.75% was upgraded by reverse flotation with fatty acids as the collector. The effects of five parameters on two levels were investigated using the $2^{5-1}$ fractional factorial design in 16 experiments. The optimum flotation conditions were determined to be pH = 12; dosage of collector, 1 kg/t; dosage of $Ca^{2+}$ as activator, 4 kg/t; and dosage of starch as depressant, 1 kg/t. Under these conditions, a concentrate of 53.4% Fe grade and 79.91% recovery was produced. |

## 1. Introduction

The beneficiation of low-grade iron ores has received many attentions in the recent years due to the depletion of high-grade iron ore reserves and the raising demand around the world for steel-making. The smelting of low-grade iron ores through the blast furnace or direct reduction method results in some problems such as increasing energy consumption, reducing furnace capacity, increasing iron loss, and improper operation of furnace as a result of high slag formation [1]. Hence, it must be upgraded to a concentrate with an iron grade more than 65%. The main difficulties in the upgradation of low-grade iron ores can be listed as the mineralogical and textural complexity, high silica

content, soft nature of some iron minerals, and variability of iron minerals [2, 3].

The upgradation of an iron ore (42.98% Fe) with wet low-intensity magnetic separation (WLIMS) was investigated by Dewari et al. [4, 5]. A concentrate of 66% Fe grade and 88% recovery was produced by WLIMS when the feed size was less than 0.5 mm [4, 5]. In another research work, a concentrate of 52.74% Fe grade and 66.35% recovery was produced by WLIMS from a low-grade iron ore (30.3% Fe) from Quadrilatero Ferrifero mineral province in Brazil [6].

WLIMS is the most economical way for upgradation of iron ores with a high magnetic susceptibility. The efficiency of WLIMS

decreases by decreasing the magnetic susceptibility of iron ores. A technique for upgradation of these iron ores is reduction roasting followed by WLIMS. In this technique, the reduction of hematite into magnetite is performed by a suitable reductant such as graphite and coal, and then its upgradation is performed by WLIMS [7, 8]. A concentrate of 65.22% Fe grade and 89.9% recovery was produced by upgradation of a low-grade iron ore (34.6% Fe) using this technique [9]. In another study, a low-grade iron ore (32% Fe) was upgraded to a concentrate of 52.95% Fe grade and 47.38% recovery using reduction roasting followed by WLIMS [7]. The main challenges encountered in implementing this technique are the high energy consumption, environmental concerns, and efficiency of the roasting process. An alternative technique available for upgrading iron ores with a low magnetic susceptibility is flotation. The reverse flotation of iron ores is the most widely used flotation route for the beneficiation of iron ores, and its efficiency has been proved in several research works [10-12]. In this flotation route, the depression of iron minerals is done by addition of a suitable depressant such as starch, and a cationic collector such as an amine or an ether amine is used to improve the hydrophobicity of gangue minerals [13, 14]. This process is very sensitive to the presence of slime-sized materials, and the de-sliming operation must be performed before the flotation process, which results in some iron loss [15].

The upgradation of a low-grade iron ore by combination of the WLIMS and flotation methods can take the benefits of both methods and fix their disadvantages. This technique has so far received a little attention and its performance has been investigated in more detail in this research work. Furthermore, the reverse flotation of the low-grade iron ore has been investigated by an anionic collector instead of a cationic collector. The reverse flotation of iron ores by anionic collectors has received much attention in the recent years due to the lower cost of anionic collectors in comparison with the cationic collectors and insensitivity of this flotation route to the presence of slime-sized materials.

## 2. Materials and methods
### 2.1. Studied area
The Sangan iron ore mine in NE Iran with 1.2 billion tons of geological reserve is one of the largest iron reserves in Iran and the Middle East. The ore has been distributed in a rectangular area

of $22 \times 10$ km$^2$. The Sangan mining area is usually divided into three zones: western, central, and eastern. There are several mining attempts in each zone. The western zone, as the main zone, consists of four mines A, B, $C_N$, and $C_S$ in a rectangular area of $2 \times 3$ km$^2$. The geographical position of the Sangan mining area is shown in Figure 1.

The extracted ore from mines B and $C_N$ with an iron grade more than 50% is transported to the iron ore processing plant for producing a concentrate with an iron grade more than 65%, and the extracted ores with an iron grade less than 50% are deposited in the mining site in terms of their iron grade in two dumps of 40-50% and 20-40% Fe.

### 2.2. Sampling procedure
The samples were collected from the lateral side of the first dump (20-40% Fe) of mine B (Figure 2a). The sampling grid was designed based on the standard hexagonal sampling grid. It consisted of 12 profiles and three sampling points in each one. The samples were taken using a blade and a cylindrical vessel (Figure 2b) after removing the weathered zone. The blade was placed on the upstream to prevent rock falling, and the sample was collected by the cylindrical vessel (Figure 2c). The minimum sample weight was determined based on the Gay's formula. The total weight of the sub-samples was 200 kg.

### 2.3. Sample preparation
The sample was dried at 105 °C in a laboratory oven. Crushing of the sample was carried out in four stages in a closed circuit with a 2-mm sieve using laboratory jaw crushers. Afterward, the sample homogenization and division was done to produce 2 kg sub-samples.

### 2.4. Characterization of low-grade iron ore
The mineralogical composition of the sample was determined using a Philips-Xpert Pro X-ray diffraction (XRD) analyzer. The chemical analysis of the sample was performed using a Philips PW1404 X-ray Fluorescence (XRF) spectrometer. In order to determine the total Fe content of the sample, it was dissolved in a hot concentrated hydrochloric acid solution and titration of the leached solution was performed by potassium dichromate in the presence of diphenylamine as an indicator after the tin(II) chloride reduction of ferric ions. The FeO content of the sample was determined by the same procedure as the total Fe, with the difference that tin(II) chloride was not

added to the leached solution for reduction of ferric ions. The sulfur content of the sample was determined by a Leco sulfur analyzer.

High-resolution compositional maps of the sample were obtained through imaging with back-scattered electrons (BSEs) using a scanning electron microscope (LEO, 1450 UP, Zeiss; Oberkochen, Germany). The qualitative and quantitative chemical composition of the particular phases was investigated by an EDS analyzer (INCA ENERGY 350 accessory) and EPMA (SX100, Cameca, France), respectively.
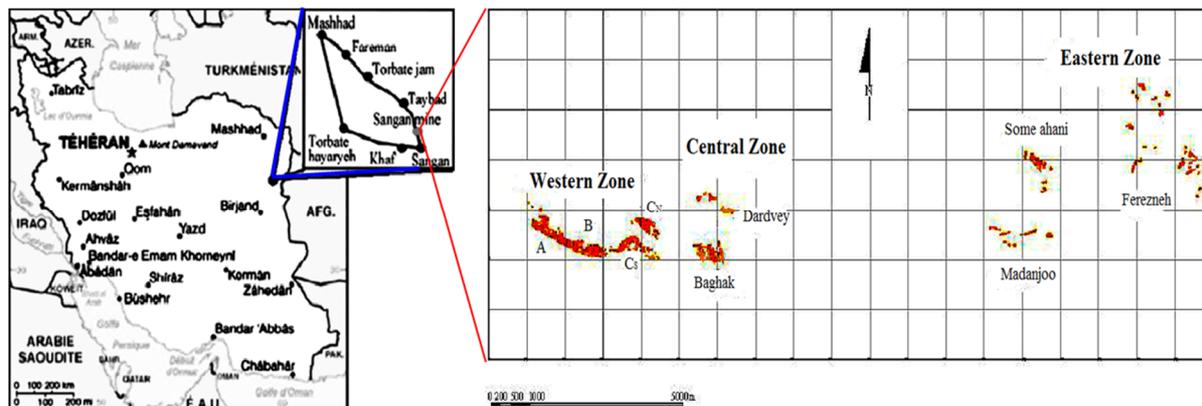


**Figure 1. The geographical position of Sangan iron ore mines.**
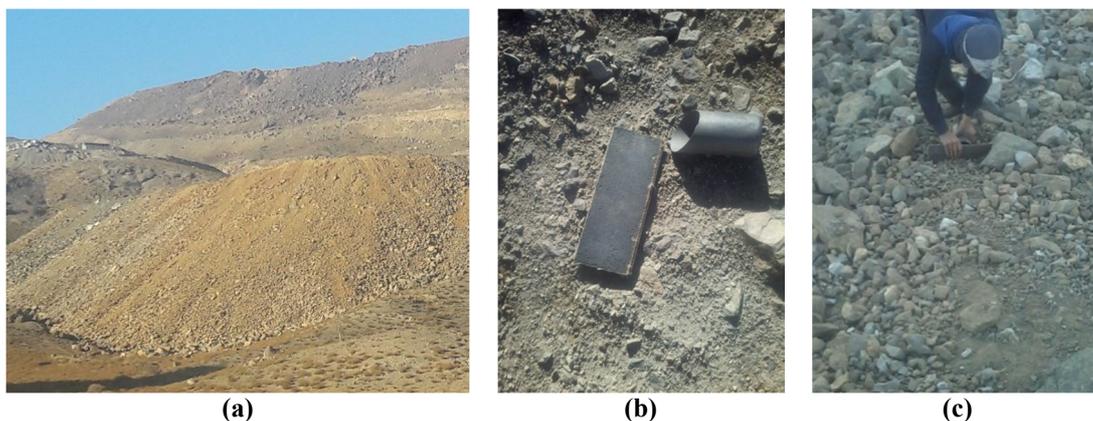


|  (a) | (b) | (c) |

**Figure 2a. The lateral side of the first dump (20-40% Fe) of mine B, b. The sampling tools, c. The sampling method.**

## 2.5. WLIMS

The magnetic separation tests were done using a wet low-intensity drum magnetic separator. The length and diameter of the magnetic separator were 50 and 30 cm, respectively. The intensity of the magnetic separator was determined to be 1000 Gauss by a TM-701 Gauss-meter, Japan. The effects of feed size, solid content, and drum rotation speed on the performance of the magnetic separator was studied.

In order to investigate the effect of feed size on the performance of the magnetic separator, initially, 2 kg of the sub-samples were ground by a laboratory ball mill at the four different grinding times of 15, 30, 45, and 60 min at a solid content of 40%. The d80 of the milled sub-samples at the grinding times of 15, 30, 45, and 60 min was determined to be 367, 230, 135, and 115 μm, respectively, from the graph of cumulative percent passing versus sieve size. Afterward, the magnetic separation was done at the solid content of 40% and the drum rotation speed of 50 rpm.

In order to investigate the effect of drum rotation speed on the magnetic separator performance, it was set at the three different values of 30, 40, and 50 rpm, and the other parameters including the solid content and feed size (d80) were kept constant at 40% and 135 μm, respectively.

In order to evaluate the effect of the solid content on the magnetic separator performance, it was set at the three different values of 30%, 40%, and 50%, and the other parameters including the drum rotation speed and feed size (d80) were kept constant at 50 rpm and 135 μm, respectively.

## 2.6. Flotation

At first, the five sub-samples were separately ground in a laboratory ball mill for 15 min at a

solid content of 40%. The milled sub-samples were separately subjected to WLIMS at the conditions of drum rotation speed = 50 rpm and solid content = 40%. The concentrate and tailing of the magnetic separation step were filtered, dried, homogenized, and analyzed for the iron grade by the titration method. The iron grade of the magnetic separation tailing was determined to be 28.75%. It was ground for 30 min by a laboratory ball mill at conditions of dried solid weight = 2 kg and solid content = 40%. The ground sample was filtered, dried, homogenized, and divided into 200 g sub-samples. Its particle size distribution curve was drawn and d80 was determined to be 162 μm.

A 200 g sub-sample plus 800 mL of water were added to a 1-L flotation cell. The flotation machine was turned on and allowed to mix the materials for a few minutes. The pH of the pulp was then adjusted to a desired value by adding a 2 M sodium hydroxide solution. In the next step, the depressant (corn starch or sodium silicate), the calcium ions as the activator were added to the flotation cell, and the conditioning was done for 5 minutes. The collector was then added to the flotation cell and conditioned for 5 minutes. Finally, the air valve was opened and the frothing was performed for 5 min. The flotation

concentrate and tailing were filtered, dried at 105 °C, and analyzed for total Fe by the titration method. The collector used in these experiments was a combination of two types of the Alke and Dirol collectors with a weight ratio of 50:50. The Alke and Dirol collectors were fatty acid collectors, the Dirol collector had also the frothing properties; they were obtained from a local producer. The effects of five factors on two levels were investigated on the reverse flotation. Table 1 shows the factors and their levels in the reverse flotation experiments.

The design of flotation experiments was carried out using the fractional factorial design. In the statistical science, the fractional factorial design is one type of experimental designs in which a number of tests of full factorial design are selected carefully based on the principles of design of experiments. The fractional factorial design is expressed using the symbol $I^{k-p}$, in which I is the number of factor levels, k is the number of factors, and p is the size of the fraction of the full factorial used. In this research work, the $2^{5-1}$ fractional factorial design was applied to study the reverse flotation of the magnetic separation tailing. Table 2 shows the conditions and run order of the tests.

**Table 1. Factors and their levels in the reverse flotation of magnetic separation tailing.**

| Factors | Low level (-) | High level (+) |
|---|---|---|
| pH | 8 | 12 |
| Collector conc. (kg/t) | 1 | 5 |
| $Ca^{2+}$ conc. (kg/t) | 1 | 4 |
| Depressant type | Corn starch | Sodium silicate |
| Depressant conc. (kg/t) | 1 | 5 |

**Table 2. The consequence and conditions of the reverse flotation tests.**

| Std. order | Run order | pH | Collector Conc. (kg/t) | $Ca^{2+}$ Conc. (kg/t) | Depressant type | Depressant conc. (kg/t) |
|---|---|---|---|---|---|---|
| 5 | 1 | 12 | 1 | 1 | Si | 5 |
| 1 | 2 | 12 | 1 | 1 | Starch | 1 |
| 2 | 3 | 8 | 5 | 1 | Starch | 1 |
| 4 | 4 | 8 | 5 | 4 | Starch | 5 |
| 7 | 5 | 12 | 1 | 4 | Si | 1 |
| 6 | 6 | 8 | 5 | 1 | Si | 5 |
| 3 | 7 | 12 | 1 | 4 | Starch | 5 |
| 8 | 8 | 8 | 5 | 4 | Si | 1 |
| 9 | 9 | 8 | 1 | 1 | Starch | 5 |
| 12 | 10 | 12 | 5 | 4 | Starch | 1 |
| 14 | 11 | 12 | 5 | 1 | Si | 1 |
| 13 | 12 | 8 | 1 | 1 | Si | 1 |
| 15 | 13 | 8 | 1 | 4 | Si | 5 |
| 10 | 14 | 12 | 5 | 1 | Starch | 5 |
| 16 | 15 | 12 | 5 | 4 | Si | 5 |
| 11 | 16 | 8 | 1 | 4 | Starch | 1 |

## 3. Results and discussion
### 3.1. Characterization of sample
The XRF analysis results showed that the low-grade iron ore consisted of 52.33% $Fe_2O_3$, 14.2% CaO, 13.6% $SiO_2$, 4.98% $Al_2O_3$, 0.93% $K_2O$, 0.66% MgO, 0.4% $SO_3$, 0.38% MnO, and 12.42% L.O.I.; the other oxides in the sample were less than 0.1%. The sulfur grade of the sample was determined to be 0.12%, which was below the standard limit of 0.2%.

The mineralogical composition of the sample was determined by the XRD analysis. Figure 3 shows the XRD spectrum for the sample. The main peak of the constituting minerals of the sample is specified in this spectrum. It can be seen that the ore minerals of the sample are magnetite and, to a lesser extent, hematite, along with a small amount of goethite. Furthermore, calcite and quartz are the main gangue minerals of the sample, and kaolinite, diopside, and dickite are the minor gangue minerals.
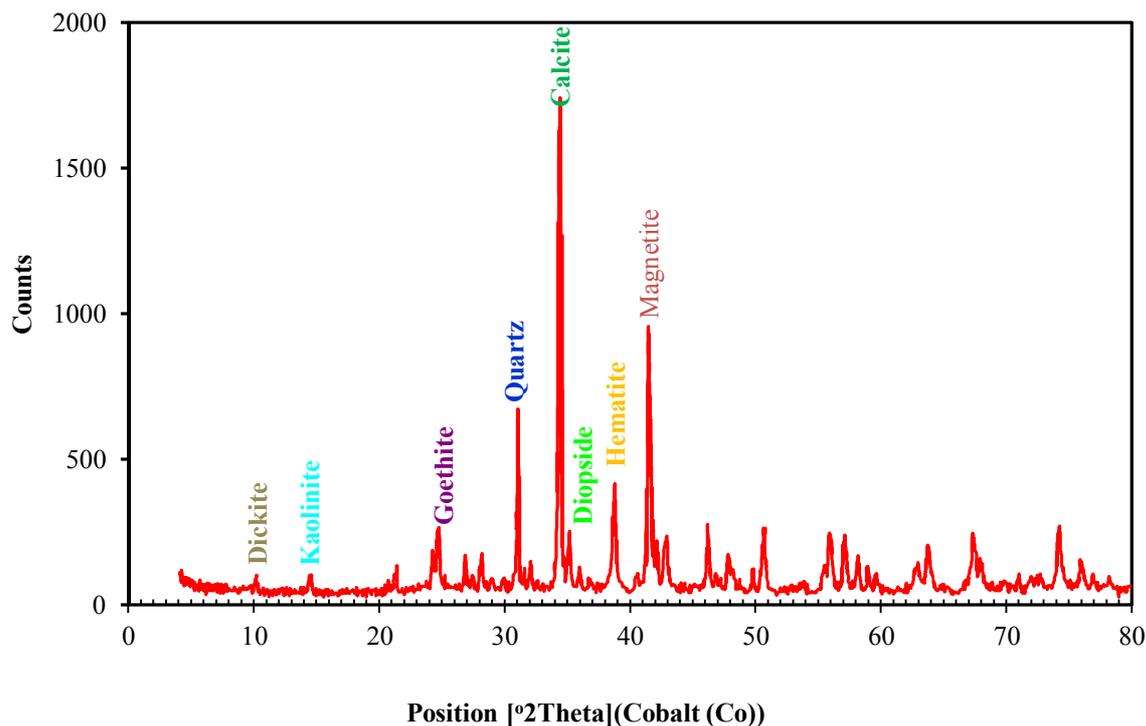


**Figure 3. The XRD graph of the low-grade iron ore sample.**

The total Fe content of the low-grade iron ores is usually considered to be less than 40%. It was determined to be 36.86% for the representative sample, which indicated that it was a low-grade iron ore. The FeO content of an iron ore is commonly used for determination of the magnetite content of the sample based on the following equation:

$$\%Fe_3O_4 = \frac{232}{72} \times \%FeO \tag{1}$$

The FeO content of the representative sample was determined to be 8.1%. Therefore, the magnetite content of the sample was determined to be 26.1% through Eq. 1. The Fe/FeO ratio is an indicator of the hematite or magnetite nature of an iron ore. The Fe/FeO ratio for a magnetite iron ore is less than 4; for a magnetite/hematite iron ore, it is in the range of 4-7, and for a hematite iron ore, it is

more than 7. The Fe/FeO ratio for the representative sample was determined to be 4.55%. It confirms that the sample is a magnetite/hematite iron ore.

The SEM image of the sample in the BSE imaging mode and its EDS analysis is shown in Figure 4. The SEM image in the BSE imaging mode is used to detect the contrast between areas with different chemical compositions since heavy elements (with high atomic numbers) backscatter electrons more strongly than light elements (with low atomic numbers), and thus appear brighter in the SEM image. It can be seen that different phases based on their brightness can be distinguished in the SEM image of the sample in the BSE imaging mode. In order to determine the chemical composition of different phases, the EDS analysis was performed at seven points and the results obtained were shown in Figure 4. The

EDS analysis cannot detect the lightest elements, typically below the atomic number for sodium (Na). Therefore, carbon (C), nitrogen (N), and oxygen (O) cannot be detected in the EDS analysis. Fe was detected in points 1, 2, and 4. Hence, the chemical composition of this phase, which appears with the highest brightness may be $Fe_2O_3$ or $Fe_3O_4$. Calcium was detected in the EDS analysis of point 3. Therefore, the chemical composition of this phase can be estimated as $CaCO_3$. The chemical composition of points 5 and 6 can be estimated as $SiO_2$ since Si has been detected in the EDS analysis. Si, Al, and K are observed in the EDS spectrum of point 7.
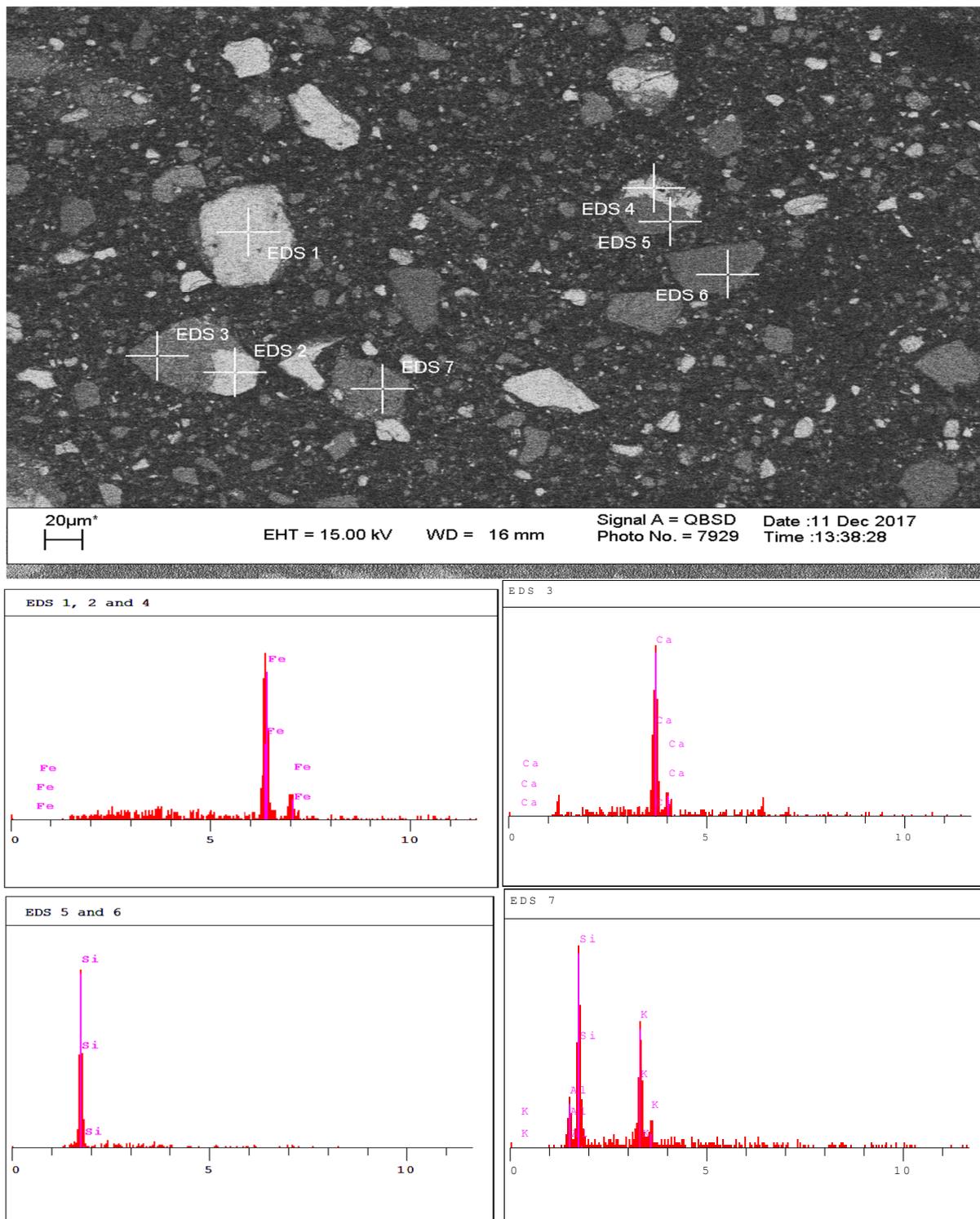


**Figure 4. SEM-EDS analysis of the low-grade iron ore sample.**

In order to determine the exact chemical composition of the various phases in the SEM image, the EPMA analysis was conducted, and the results obtained were presented in Figure 5a. The mineralogical phase of points 1 to 4, based on their chemical assay, was determined to be calcite, hematite, goethite, and quartz, respectively. Therefore, the main gangue minerals of the sample are calcite and quartz, and they appear in the SEM image with a lower brightness than the ore minerals. In order to determine different types of iron oxide minerals in the sample that appear with a higher brightness in the SEM image, a further EPMA analysis was conducted and the results obtained were presented in Figure 5b. The mineralogical phase of points 1 to 3 is goethite, points 4, 5, and 8 is magnetite, and points 6 and 7 is hematite, based on the quantitative chemical assay. Therefore, it can be concluded that the ore minerals of the sample are magnetite, hematite, and goethite, and the main gangue minerals of the sample are calcite and quartz. This is in accordance with the results of the XRD and Fe/FeO analysis methods.
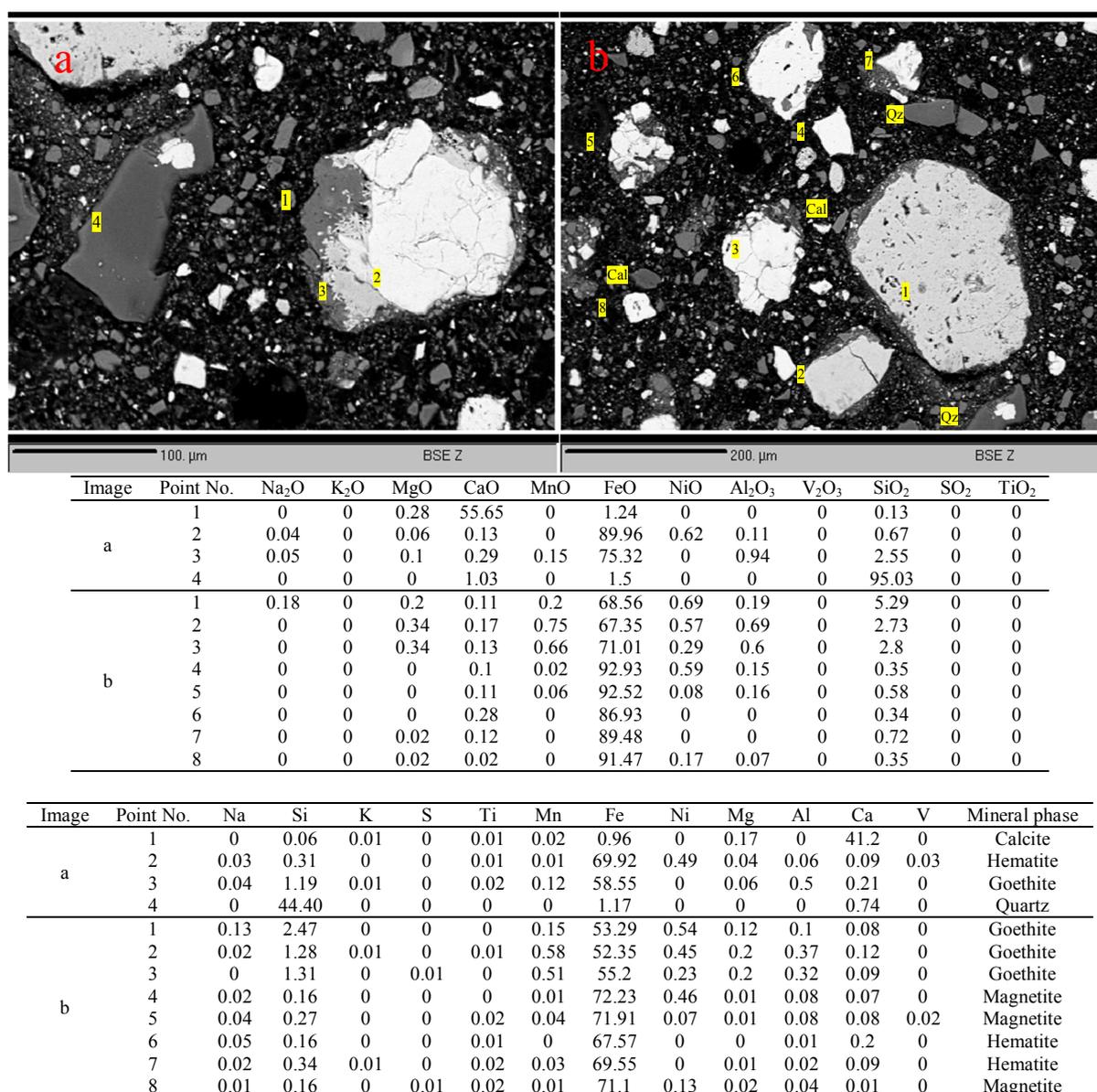


| Image | Point No. | Na$_2$O | K$_2$O | MgO | CaO | MnO | FeO | NiO | Al$_2$O$_3$ | V$_2$O$_3$ | SiO$_2$ | SO$_2$ | TiO$_2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| a | 1 | 0 | 0 | 0.28 | 55.65 | 0 | 1.24 | 0 | 0 | 0 | 0.13 | 0 | 0 |
|   | 2 | 0.04 | 0 | 0.06 | 0.13 | 0 | 89.96 | 0.62 | 0.11 | 0 | 0.67 | 0 | 0 |
|   | 3 | 0.05 | 0 | 0.1 | 0.29 | 0.15 | 75.32 | 0 | 0.94 | 0 | 2.55 | 0 | 0 |
|   | 4 | 0 | 0 | 0 | 1.03 | 0 | 1.5 | 0 | 0 | 0 | 95.03 | 0 | 0 |
| b | 1 | 0.18 | 0 | 0.2 | 0.11 | 0.2 | 68.56 | 0.69 | 0.19 | 0 | 5.29 | 0 | 0 |
|   | 2 | 0 | 0 | 0.34 | 0.17 | 0.75 | 67.35 | 0.57 | 0.69 | 0 | 2.73 | 0 | 0 |
|   | 3 | 0 | 0 | 0.34 | 0.13 | 0.66 | 71.01 | 0.29 | 0.6 | 0 | 2.8 | 0 | 0 |
|   | 4 | 0 | 0 | 0 | 0.1 | 0.02 | 92.93 | 0.59 | 0.15 | 0 | 0.35 | 0 | 0 |
|   | 5 | 0 | 0 | 0 | 0.11 | 0.06 | 92.52 | 0.08 | 0.16 | 0 | 0.58 | 0 | 0 |
|   | 6 | 0 | 0 | 0 | 0.28 | 0 | 86.93 | 0 | 0 | 0 | 0.34 | 0 | 0 |
|   | 7 | 0 | 0 | 0.02 | 0.12 | 0 | 89.48 | 0 | 0 | 0 | 0.72 | 0 | 0 |
|   | 8 | 0 | 0 | 0.02 | 0.02 | 0 | 91.47 | 0.17 | 0.07 | 0 | 0.35 | 0 | 0 |

| Image | Point No. | Na | Si | K | S | Ti | Mn | Fe | Ni | Mg | Al | Ca | V | Mineral phase |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| a | 1 | 0 | 0.06 | 0.01 | 0 | 0.01 | 0.02 | 0.96 | 0 | 0.17 | 0 | 41.2 | 0 | Calcite |
|   | 2 | 0.03 | 0.31 | 0 | 0 | 0.01 | 0.01 | 69.92 | 0.49 | 0.04 | 0.06 | 0.09 | 0.03 | Hematite |
|   | 3 | 0.04 | 1.19 | 0.01 | 0 | 0.02 | 0.12 | 58.55 | 0 | 0.06 | 0.5 | 0.21 | 0 | Goethite |
|   | 4 | 0 | 44.40 | 0 | 0 | 0 | 0 | 1.17 | 0 | 0 | 0 | 0.74 | 0 | Quartz |
| b | 1 | 0.13 | 2.47 | 0 | 0 | 0 | 0.15 | 53.29 | 0.54 | 0.12 | 0.1 | 0.08 | 0 | Goethite |
|   | 2 | 0.02 | 1.28 | 0.01 | 0 | 0.01 | 0.58 | 52.35 | 0.45 | 0.2 | 0.37 | 0.12 | 0 | Goethite |
|   | 3 | 0 | 1.31 | 0 | 0.01 | 0 | 0.51 | 55.2 | 0.23 | 0.2 | 0.32 | 0.09 | 0 | Goethite |
|   | 4 | 0.02 | 0.16 | 0 | 0 | 0 | 0.01 | 72.23 | 0.46 | 0.01 | 0.08 | 0.07 | 0 | Magnetite |
|   | 5 | 0.04 | 0.27 | 0 | 0 | 0.02 | 0.04 | 71.91 | 0.07 | 0.01 | 0.08 | 0.08 | 0.02 | Magnetite |
|   | 6 | 0.05 | 0.16 | 0 | 0 | 0.01 | 0 | 67.57 | 0 | 0 | 0.01 | 0.2 | 0 | Hematite |
|   | 7 | 0.02 | 0.34 | 0.01 | 0 | 0.02 | 0.03 | 69.55 | 0 | 0.01 | 0.02 | 0.09 | 0 | Hematite |
|   | 8 | 0.01 | 0.16 | 0 | 0.01 | 0.02 | 0.01 | 71.1 | 0.13 | 0.02 | 0.04 | 0.01 | 0 | Magnetite |

**Figure 5. EPMA analysis of the sample.**

## 3.2. Beneficiation of low-grade iron ore by WLIMS

### 3.2.1. Effect of feed size

Figure 6 illustrates the effect of feed size on the performance of the wet low-intensity magnetic separator. As it can be seen, the iron grade of the concentrate increased from 58.88% to 62.69% and the iron recovery increased from 44.85% to 55.99% with decrease in the feed size (d80) from 367 to 135 μm; it is due to the increase in the

degrees of freedom. With a further reduction in the feed size (d80 = 115 μm), the iron recovery increased but the iron grade of the concentrate decreased significantly. This shows that the performance of the magnetic separator decreases with a decrease in the feed size. It may be due to increasing the amount of slime-sized materials. The optimum feed size (d80) was determined to be 135 μm, and thus it was considered as a constant value in the evaluation of the other parameters.

### 3.2.2. Effect of drum rotation speed

Figure 7 shows the effect of drum rotation speed on the performance of the magnetic separator. As it can be seen, with increase in the drum rotation speed from 30 to 40 rpm, the iron grade of the concentrate decreased from 57.63% to 54.29% but the iron recovery increased from 31.27% to 58.91%. With more increase in the drum rotation speed to 50 rpm, the iron recovery reduced to 55.99% but the iron grade of the concentrate increased to 62.69%. Therefore, the optimum drum rotation speed was determined to be 50 rpm, and it was kept constant in the next experiments. In the magnetic drum separator, several forces act on the flowing particles, namely the centrifugal, gravitational, and magnetic forces [2, 3]. The induced centrifugal force to the flowing particles increases with increase in the drum rotation speed. It facilitates the transfer of gangue minerals to the tailing sector.
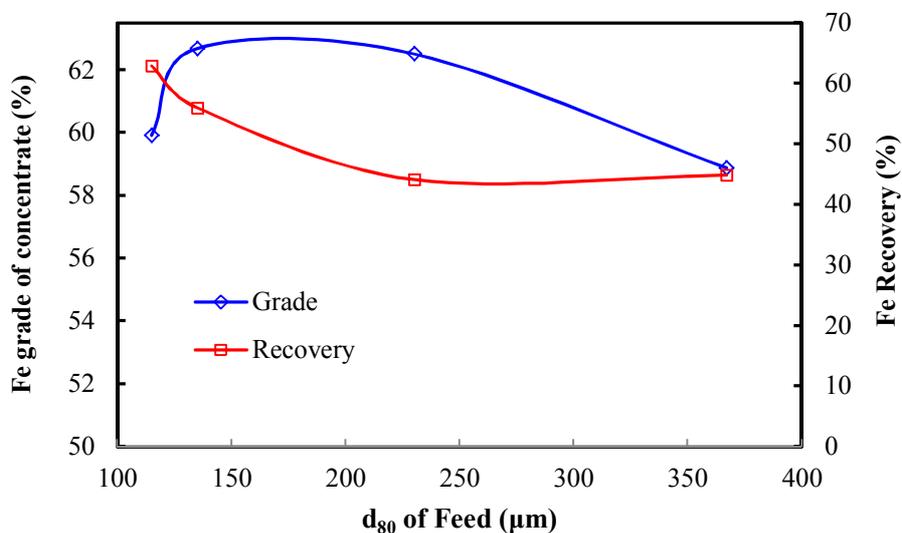


**Figure 6. The effect of feed size on the performance of wet low-intensity magnetic separator.**
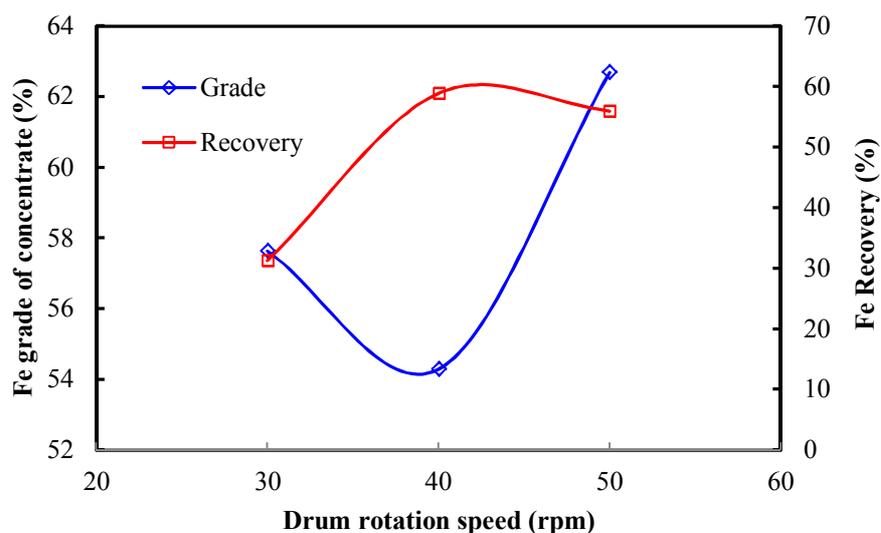


**Figure 7. The effect of drum rotation speed on the performance of wet low-intensity magnetic separator.**

### 3.2.3. Effect of solid content

Figure 8 shows the effect of solid content on the performance of the magnetic separator. As it could be seen, increasing the solid content from 30% to 40% increased the iron grade of the concentrate from 54.19% to 62.69%. However, the iron recovery decreased from 77.79% to 55.99%. By increasing the solid content up to 50%, the iron grade of the concentrate decreased to 56.2% and the iron recovery reduced to 43%. The viscosity of the pulp increased with increase in the solid content, which resulted in minimizing the fluid drag to assist the separation process. Therefore, the optimum solid content was considered to be 40%.

### 3.3. Beneficiation of magnetic separation tailing by reverse flotation

Figure 9 shows the effect of solution pH, collector concentration, type and concentration of the depressant, and concentration of $Ca^{2+}$ ions as the activator on the iron grade of the flotation concentrate. As it can be seen, with raising the solution pH from 8 to 12, the iron grade of flotation concentrate increased from 40.33% to 42.23%. This can be attributed to the surface activation of silicate minerals by $Ca^{2+}$ ions. When the solution pH is more than the pH of zero charge of quartz ($pH_{PZC} = 1.8$), $Ca(OH)^+$ will be adsorbed onto the quartz surface through electrostatic forces and $–Si–O–Ca^+$ will be generated that makes the zeta potential of quartz more positive. At pH > 9.5, the zeta potential of quartz will increase sharply because of the excessive adsorption of $Ca(OH)^+$ ions on the quartz surface. By this means, quartz is activated by calcium ions in the flotation process [16]. The iron grade of the concentrate decreased from 43.01% to 39.56% by increasing the collector concentration from 1 to 5

kg/t. It is due to the tendency of the collector to micelle formation at higher concentrations [17]. An increase in the $Ca^{2+}$ ion concentration from 1 to 4 kg/t improves the iron grade of concentrate from 40.32% to 42.25% due to a better surface activation of the silicate minerals at higher $Ca^{2+}$ ion concentrations [16]. The use of sodium silicate in comparison with corn starch increased the iron grade of concentrate from 41.08% to 41.49%. The iron grade of the concentrate increased from 40.34% to 42.22% by increasing the depressant concentration from 1 to 5 kg/t. Sodium silicate and starch in the reverse flotation of iron ores play the role of the depressant, and increasing their concentration increases the depression of iron minerals.

Figure 10 shows the two-way interaction plots of the parameters studied on the iron grade of the reverse flotation concentrate. A significant interaction is observed between pH and the depressant type so that starch has a better performance in pH 8, while sodium silicate has a better performance in pH 12. The interaction between the collector concentration and depressant type shows that the iron grade of the concentrate increases by increasing the collector concentration when the depressant type is sodium silicate and it decreases when the depressant type is starch. The interaction between the $Ca^{2+}$ ions and depressant concentrations indicates that at a low concentration of the depressant (i.e. 1 kg/t), increasing the collector concentration from 1 to 4 kg/t increases the iron grade of the concentrate from 36.93% to 43.76%. A revers trend is observed at a high concentration of the depressant (i.e. 5 kg/t) so that the iron grade of the concentrate decreases from 43.7% to 40.74%.
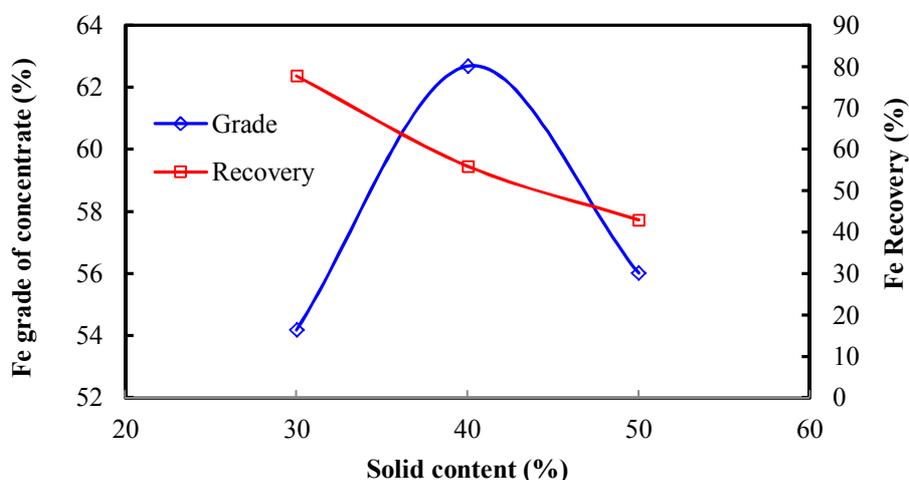


**Figure 8. The effect of solid content on the performance of wet low-intensity magnetic separator.**
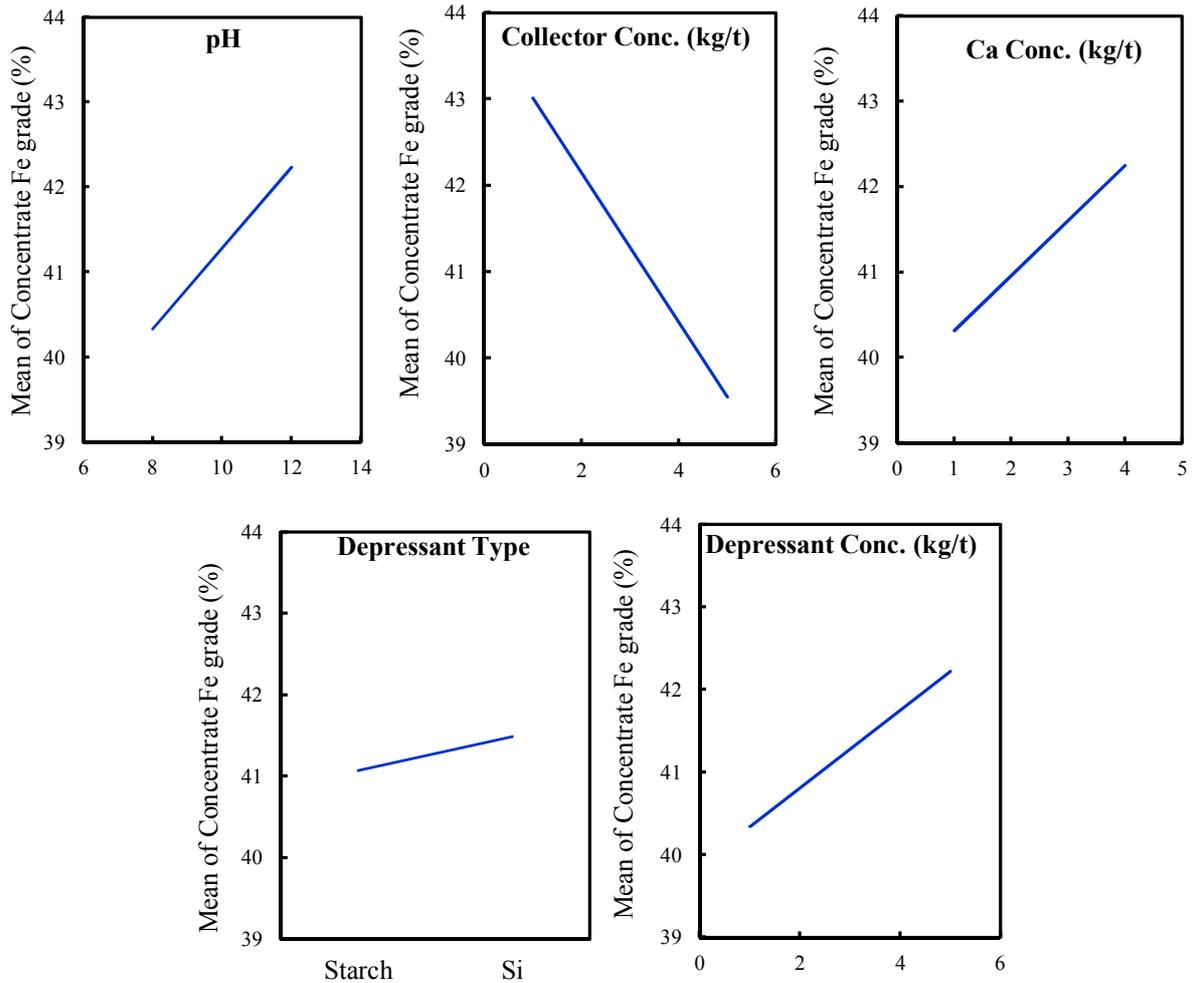
**Figure 9. The effect of process parameters on the iron grade of reverse flotation concentrate.**
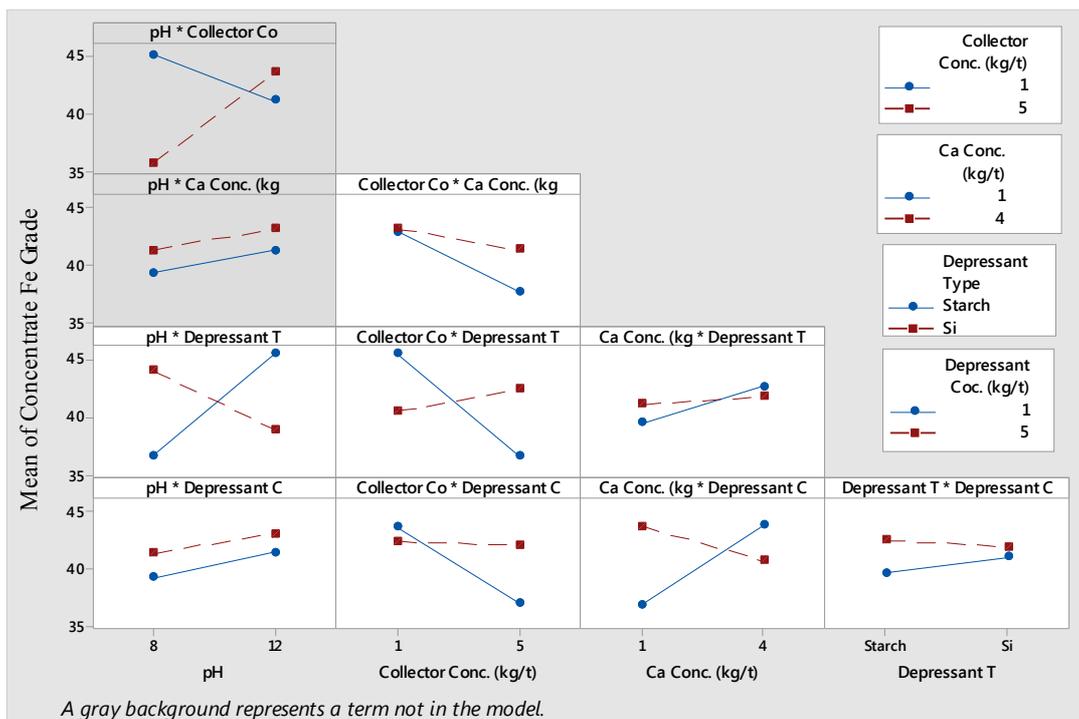


**Figure 10. The two-way interactions between the factors investigated on the iron grade of reverse flotation concentrate.**

Figure 11 shows the half-normal plot of the main effects and their two-way interactions investigated on the iron grade of the reverse flotation concentrate. The important parameters with the red solid square symbol and the unimportant parameters with blue dot symbol are shown in this plot. As it can be seen, all the main effects and two-way interactions are the important parameters involved. In this plot, any symbol farther from the red line will have a greater effect on the iron grade of the concentrate. Therefore, the most important parameter on the iron grade of the concentrate is the interaction between pH and the depressant type followed by the interaction between the collector concentration and the depressant type and the interaction between the $Ca^{2+}$ ion and depressant concentrations. Among the main effects involved, the most significant parameter is the collector concentration followed by $Ca^{2+}$ ion concentrations, pH, and concentration and type of the depressant.

Figure 12 shows a normal plot of the main effects and their two-way interactions on the iron grade of the reverse flotation concentrate. In this plot, the parameters having positive effects on the process are placed on the right hand side of the red line, and vice versa. Furthermore, any symbol farther from red will have a greater effect on the process. As it can be seen, the interaction between the collector concentration and the depressant type has the greatest positive effect and the interaction between the pH and the depressant type has the most negative effect on the iron grade of flotation concentrate. Among the main effects, $Ca^{2+}$ concentration, pH, and concentration and type of the depressant have positive effects on the process, respectively.

Figure 13 shows the effects of the process parameters on the recovery of iron in the reverse flotation of the magnetic separation tailing. As it can be seen, the iron recovery increases from 61.30% to 66.85% by raising the solution pH from 8 to 12. It can be due to the better performance of the depressants at higher pH values. By increasing the collector concentration from 1 to 5 kg/t, iron recovery decreased from 82.20% to 45.95%. It can be due to this fact that at higher concentrations of the collector, some iron minerals will have the opportunity to float along with the gangue minerals, which leads to some iron loss [18]. The iron recovery increased from 60.29% to 67.86% by increasing the $Ca^{2+}$ concentration from 1 to 4 kg/t. A better surface activation of silicate minerals occurs at higher concentrations of $Ca^{2+}$ ions [16]. It results in more collector adsorption on silicate minerals, which reduces the collector adsorption on iron minerals. The iron recoveries were 50.43% and 77.72% using starch and sodium silicate as the depressant, respectively. It shows a better performance of sodium silicate in depression of iron minerals in comparison with corn starch. Iron recovery increases from 60.35 to 67.85% with increasing depressant concentration from 1 to 5 kg/t. A better depression of iron minerals will occur at higher depressant concentrations, which will result in increasing the iron recovery in the reverse flotation of low-grade iron ores [18].

Figure 14 shows the two-way interactions of the parameters studied on the iron recovery in the reverse flotation process. As it can be seen, the interaction between the depressant type and pH is significant so that increasing the solution pH has a positive effect on the iron recovery when the depressant type is starch and it has a negative effect using sodium silicate as the depressant. The interaction between the $Ca^{2+}$ ion and depressant concentrations is also significant so that at a low concentration of the depressant (i.e. 1 kg/t), increasing the concentration of $Ca^{2+}$ ions from 1 to 4 kg/t leads to an increase in the iron recovery from 49.13 to 71.48% but at a high concentration of the depressant (i.e. 5 kg/t), it leads to a decrease in the iron recovery from 71.46% to 64.24%.

Figure 15 shows the half-normal plot of the standardized effects investigated on the iron recovery in the reverse flotation process. This figure shows that all the main effects and two-way interactions are shown with the red solid square symbol, and there is no blue dot symbol in this chart that indicates that all the parameters affect the iron recovery in the reverse flotation process. As it can be seen, the collector concentration has the greatest effect on the iron recovery. Afterward, the type of depressant, interaction between the pH and the depressant type, interaction between the $Ca^{2+}$ ion and depressant concentrations, interaction between the depressant type and its concentration, interaction between the collector concentration and the depressant type, main effects of $Ca^{2+}$ ion concentration, the depressant concentration, pH, interaction between the collector and depressant concentrations, and interaction between the collector and $Ca^{2+}$ ion concentrations affect the iron recovery in the reverse flotation process, respectively.

Figure 16 shows the normal plot of the standardized effects investigated on the iron recovery in the reverse flotation process. As it can be seen, the type of depressant has the most

positive effect and the collector concentration has the most negative effect on the iron recovery. The other parameters that have positive effects on the iron recovery are the concentration of $Ca^{2+}$ ions,

depressant concentration, and solution pH, respectively. Among the main effects, the collector concentration has a negative effect on the iron recovery in the reverse flotation process.
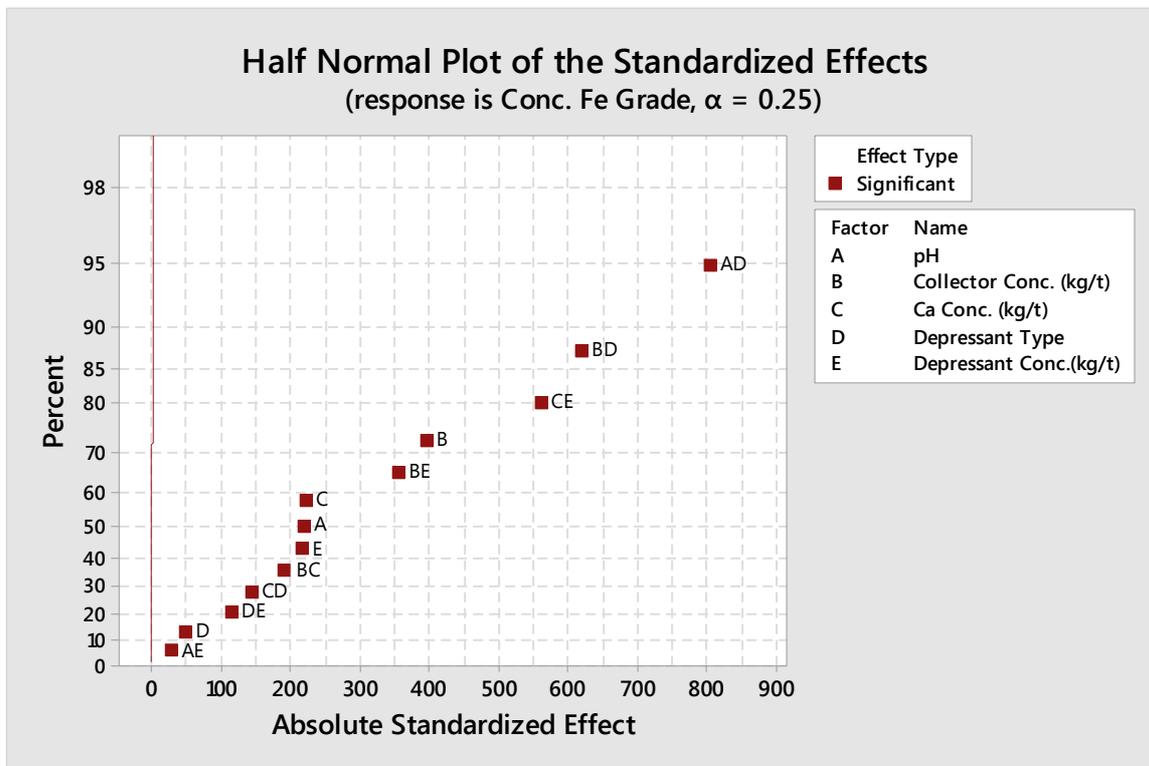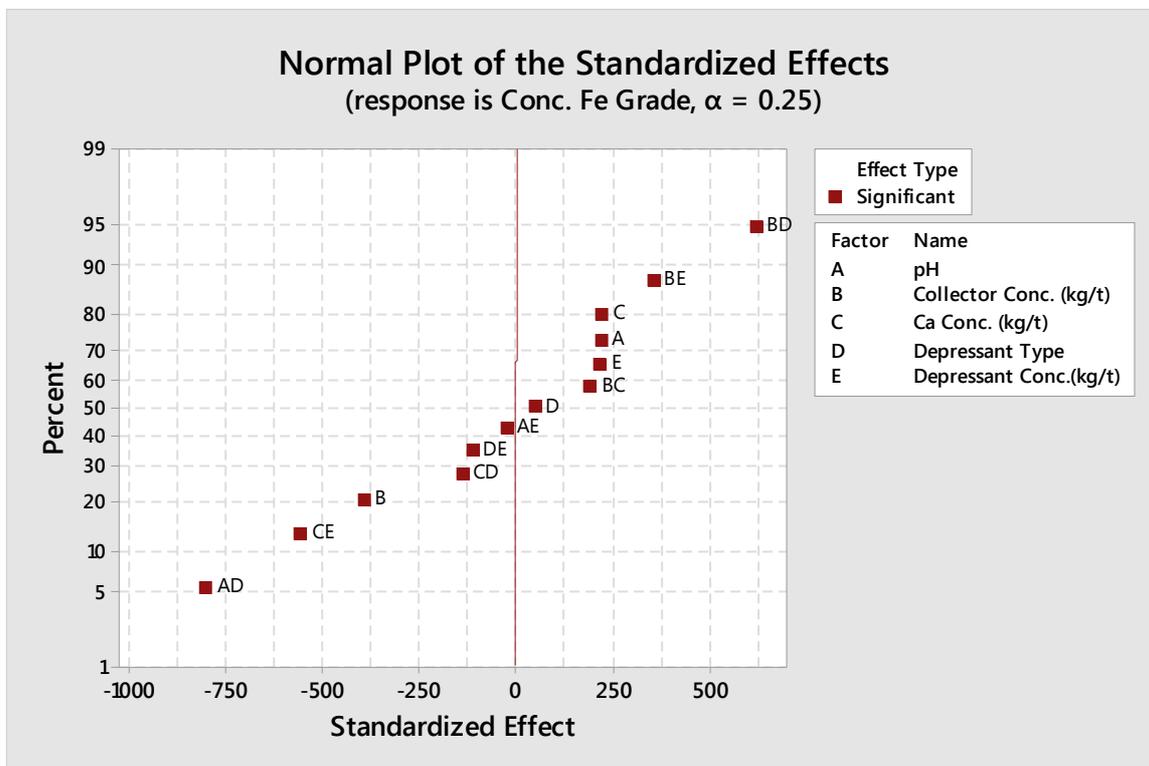


**Figure 11. The half-normal plot for the main effects and their two-way interactions investigated on the iron grade of reverse flotation concentrate.**



**Figure 12. The normal plot for the main effects and their two-way interactions investigated on the iron grade of reverse flotation concentrate.**
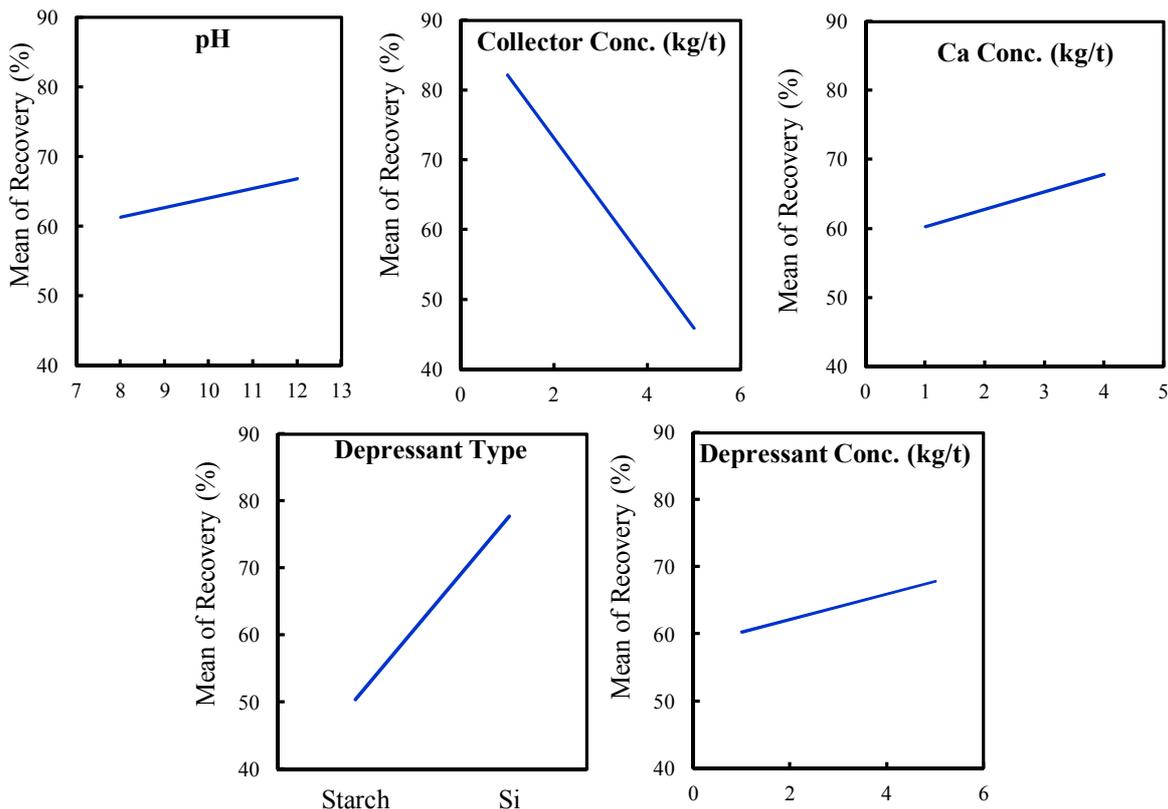
**Figure 13. The effect of process parameters on the iron recovery in the reverse flotation process.**



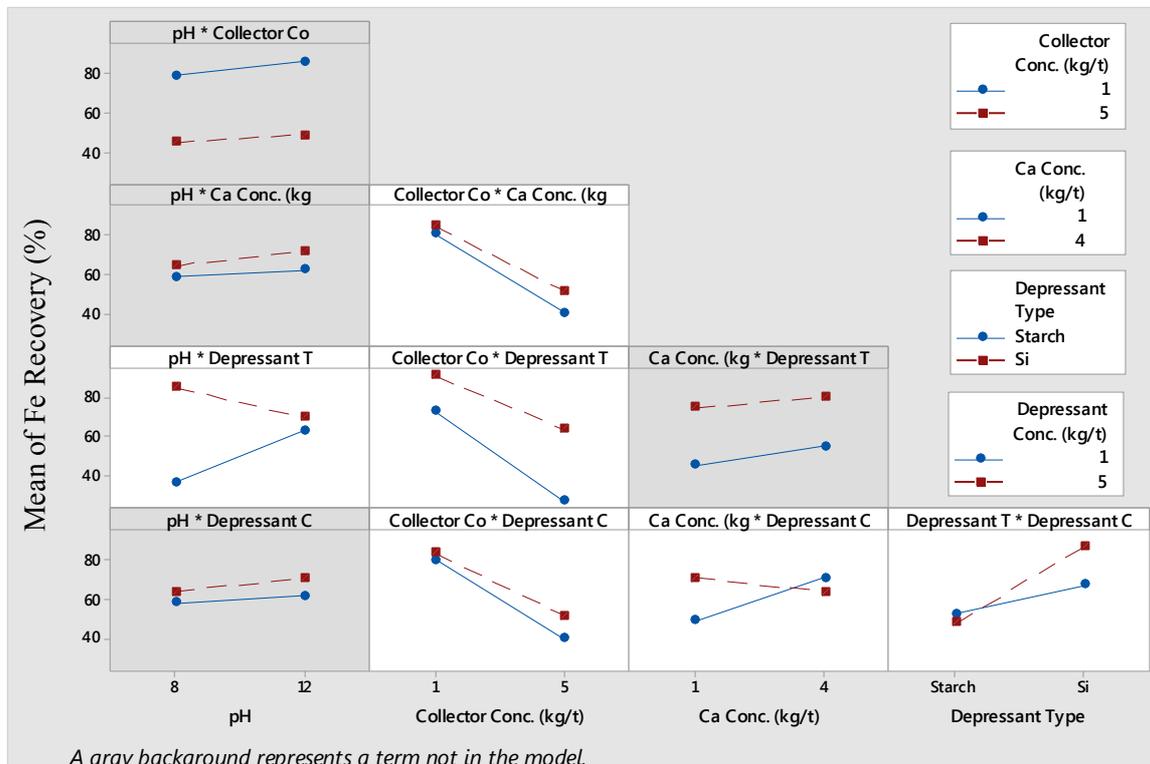*A gray background represents a term not in the model.*

**Figure 14. The two-way interactions between the factors investigated on the iron recovery in the reverse flotation process.**
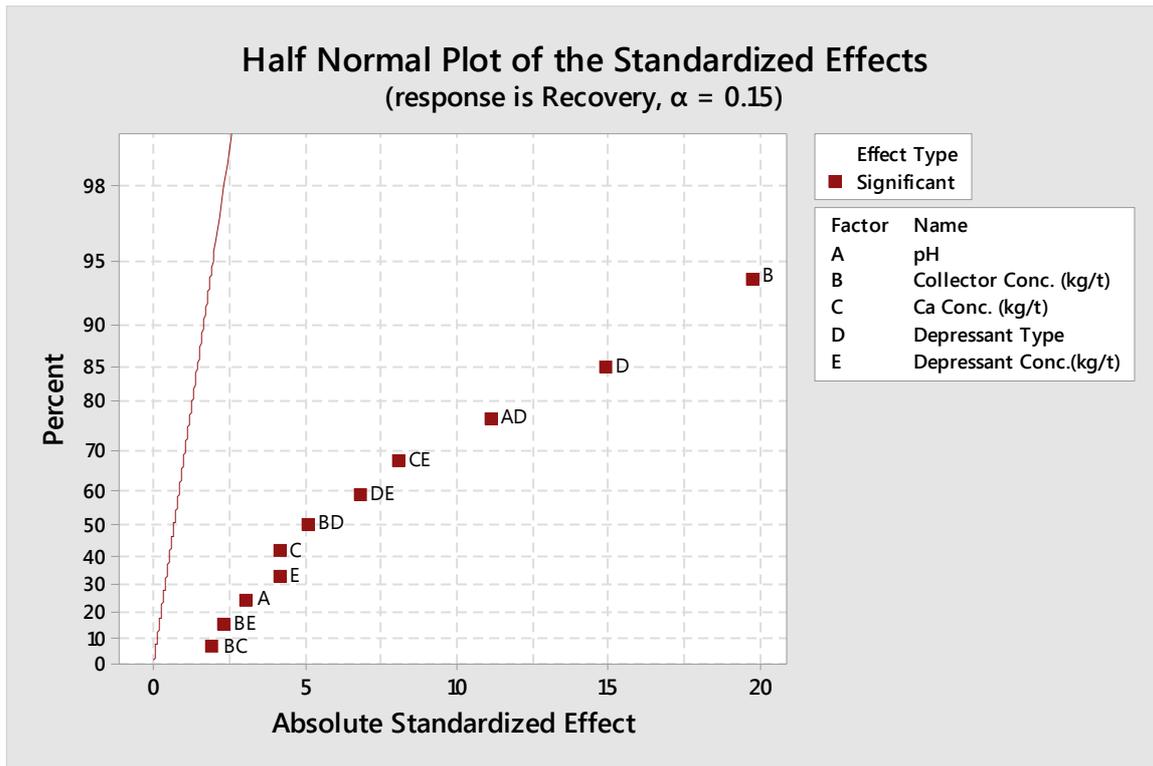
**Figure 15. The half-normal plot for the main effects and their two-way interactions investigated on the iron recovery in the reverse flotation process.**
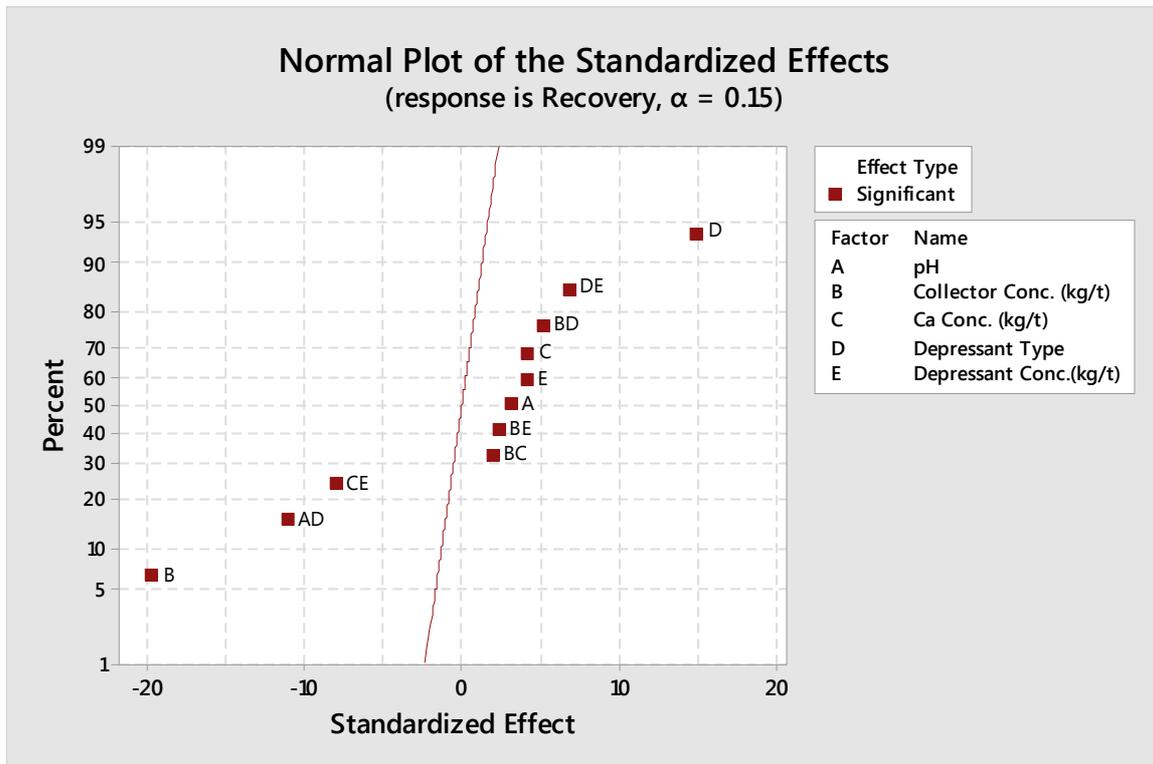


**Figure 16. The normal plot for the main effects and their two-way interactions investigated on the iron recovery in the reverse flotation process.**

## 4. Conclusions

This research work showed that a low-grade iron ore could be upgraded using both the WLIMS and reverse flotation methods. It is possible to produce a high-grade magnetite concentrate by WLIMS and then produce a high-grade hematite concentrate by upgradation of the tailing of magnetic separation by reverse flotation.

The effects of feed size, solid content, and drum rotation speed on the performance of wet low-intensity magnetic separator were investigated. The results obtained showed that the iron grade of the concentrate increased from 58.88% to 62.69%, and the iron recovery increased from 44.85% to 55.99% by decreasing the feed size from d80 = 367 μm to d80 = 135 μm. Any further decrease in the feed size results in a drop in the Fe grade of the concentrate. It is due to decreasing the separator performance by increasing the amount of slime sized-materials in the feed. The iron grade of the concentrate increased from 54.19% to 62.69% and the iron recovery decreased from 77.79% to 55.99% by increasing the solid content from 30% to 40%. Any further increase in the solid content had a significant negative effect on the iron grade and iron recovery. With increase in the drum rotation speed from 30 to 40 and then 50 rpm, the iron grade of the concentrate was first reduced and then increased, and a reverse trend was observed for the iron recovery. The optimum operating conditions for WLIMS were determined as d80 = 135 μm, 40% solid content, and 50 rpm drum rotation speed. Under these conditions, the iron grade of the concentrate and the iron recovery were 62.69% and 55.99%, respectively.

The tailing of WLIMS with an iron grade of 25.96% was upgraded by the reverse flotation. The effects of the process parameters including pH, collector concentration, concentration of $Ca^{2+}$ ions as the activator, and type and concentration of the depressant were investigated using the $2^{5-1}$ fractional factorial design. The experimental results obtained showed that the iron grade of the concentrate and the iron recovery increased by increasing the solution pH from 8 to 12, the $Ca^{2+}$ concentration from 1 to 4 kg/t, and the depressant concentration from 1 to 5 kg/t; they decreased by increasing the collector concentration from 1 to 5 kg/t. The use of sodium silicate in comparison with corn starch results in increasing the iron grade of the concentrate and the iron recovery. The optimum flotation conditions were determined using the Minitab 17 software including pH = 12, the collector concentration = 1 kg/t, the concentration of $Ca^{2+}$ ions as the activator = 4 kg/t, and the concentration of starch as the depressant = 1 kg/t. Under these conditions, the iron grade of the concentrate and iron recovery were determined to be 53.4% and 79.91%, respectively.

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# پرعیارسازی یک کانسنگ آهن کم‌عیار با ترکیب روش‌های جدایش مغناطیسی تر شدت پایین و فلوتاسیون معکوس

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**چکیده:**

در این پژوهش پرعیارسازی یک کانسنگ آهن کم‌عیار با ترکیب روش‌های جدایش مغناطیسی تر شدت پایین و فلوتاسیون معکوس تحقیـق شـد. اجـزای اصلـی تشکیل‌دهنده نمونه معرف Fe ۳۶/۸۶٪، FeO ۸/۱٪، CaO ۱۴/۲٪، SiO$_2$ ۱۳/۶٪ و S ۰/۱۲٪ بر مبنای روش‌های آنالیز فلوئورسانس اشعه ایکس، تیتراسیون و Leco تعیین شدند. مطالعات کانی‌شناسی به وسیله روش‌های آنالیز پراش اشعه ایکس، میکروسکوپی الکترونی روبشی، طیف‌سنجی پـراش انـرژی اشـعه ایکـس، میکرو آنالیزور پویشی الکترون و تیتراسیون Fe/FeO نشان دادند که کانی‌های با ارزش حاضر در نمونه معرف مگنتیت، هماتیت و گوتیت هسـتند و کـانی‌هـای گانگ اصلی نمونه معرف کلسیت و کوارتز می‌باشند. تأثیر پارامترهای عملیاتی شامل اندازه ابعادی خوراک، درصد جامد و سرعت چرخش درام بر کارایی جدایش مغناطیسی تر شدت پایین (WLIMS) تحقیق شد. شرایط بهینه عملیاتی WLIMS به صورت اندازه ابعادی خـوراک ۱۳۵µm، درصـد جامـد ۴۰٪ و سـرعت چرخش درام ۵۰rpm تعیین شدند. تحت این شرایط، کنسانتره‌ای با عیار آهن ۶۲/۶۹٪ و بازیابی ۵۵/۹۹٪ تولید شد. باطله جدایش مغناطیسی تر شدت پایین با عیار آهن ۲۸/۷۵٪ به وسیله فلوتاسیون معکوس توسط اسیدهای چرب به عنوان کلکتور پرعیارسازی شد. تأثیـر پـنج پـارامتر در دو سـطح بـا اسـتفاده از طـرح آزمایشی فاکتوریل جزئی ۲$^{۵-۱}$ در ۱۶ آزمایش بررسی شد. شرایط بهینه فلوتاسیون معکوس به صورت pH برابر با ۱۲، غلظت کلکتور ۱kg/t، غلظـت Ca$^{۲+}$ بـه عنوان فعال‌کننده ۴kg/t و غلظت نشاسته به عنوان بازداشت کننده kg/t ۱ به دست آمد. تحت این شرایط کنسانتره‌ای با عیار آهـن ۵۳/۴٪ و بازیابی ۷۹/۹۱٪ حاصل شد.

**کلمات کلیدی:** کانسنگ آهن کم‌عیار، پرعیارسازی، جدایش مغناطیسی تر شدت پایین، فلوتاسیون معکوس.