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Effective Processing of Specularite Ore by Wet Magnetic Separation and Reverse Flotation Techniques

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ABSTRACT

he aim of this study was to obtain a high grade and yield percentage of iron concentrate from a specularite ore by using wet magnetic separation and reverse flotation techniques. The processing a specularite sample using wet magnetic separation and reverse flotation method was studied. During the magnetic separation process, particle size was -2000 µm and magnetic field applied at 0.25 T and 0.85 T which were performed at a solid-liquid ratio of 10% and 20% by weight, respectively. In the reverse flotation tests, experimental design (DOE) was applied, statistically. Depressant dosage, collector dosage and flotation time were selected as main parameters. PH value, frother dosage (MIBC) and particle size were constant parameters. The results showed that particle size and magnetic field intensity had a significant effect on the iron concentrate grade and yield for wet magnetic separation. The optimum value of iron concentrate grade was 98.75% at 0.25 T and particle size of -150 μm while the highest value of iron concentrate yield was 67% at 0.75 T and particle size of -74 µm. In the flotation tests, depressant dosage had the greatest influence on the iron concentrate grade while the effect of the collector dosage and froth collection time were less. Froth collection time had the greatest effect on iron concentrate yield. The maximum iron concentrate grade was 90.13% for the following conditions: 5250 g/ton depressant, 1000 g/ton collector and 2-minute froth collection time. The maximum iron concentrate yield was 98.96% for the following conditions: 5250 g/ton depressant, 1500 g/ton collector and 1 min froth collection time under fixed conditions.

Keywords:

Magnetic separation, Reverse flotation, Particle size, Upgrading, Specularite, Experimental design.

INTRODUCTION

In recent years, the world steelmaking industry has Leben significantly developed. This led to a rapid increase in iron ore production and demand [1]. Iron, which is one of the most useful elements in the world, has plentiful mineral forms. The primary industrial types of iron minerals include oxides and hydroxides and carbonates. Magnetite [Fe₂O₄], hematite [Fe₂O₂], goethite [FeO (OH)], limonite [FeOOH×nH2O] and siderite [FeCO₂] are the most common iron minerals [2]. Most of the iron ores in the world are in the form either hematite (Fe₂O₂) or magnetite (Fe₂O₄) [3]. Gravity and magnetic separation methods are basic ore processing techniques for obtaining the iron concentrates [4]. Wet low and high intensity magnetic separation methods are appropriate methods to process these ores with high iron content and a high degree

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of liberation of iron oxides from gangue [2]. The magnetic separation method is a dominant technique in beneficiation of low-grade iron ores since magnetic separator process has benefits of low operating cost, large capacity and being environmentally friendly [1]. Magnetic separation method depends on magnetic susceptibility of the materials. The magnetic fraction with higher magnetic susceptibility may be valuable or gangue depending upon its end use in a particular process and also the non-magnetic fraction, e.g., separation of magnetite (magnetic) from quartz (non-magnetic), etc. [5].

Also, flotation can also be used for the beneficiation of iron oxides minerals (hematite and magnetite) if the major impurity is quartz [6]. Flotation is a physicoche-

mical separation method based on chemical differences in the surface characteristics of the mineral species present in pulp [6] and the efficiency of the process directly proportional to the characteristics of the bubbles produced [7]. This separation procedure has been applied to iron ores since 1931[8-10]. There are 2 types of flotation have been carried out from that period to upgrade the iron ores by froth flotation process including both direct and reverse flotation. The formation processes and the mineralogical composition of iron ores have a significant influence on these separation processes [11]. The market necessities for the higher-grade iron concentrate to improve the productivity of the steelmaking industry have increased the significance of the flotation process concerning the classic pre-concentration of iron ores by gravity or magnetic separation [9]. Today, the cationic reverse flotation of quartz is the most commonly used route, which depends on floatability of the quartz with amines partially neutralized with acetic acid, whilst iron ore is depressed by starches [12]. Besides, reverse flotation of quartz has been successfully used for particle size below 150 μm for iron ore separation [13].

The objective of this study is to separate iron concentrate from gangues by using wet magnetic separation and reverse flotation techniques and to investigate the effect of the effective separation parameters (particle size and magnetic field intensity in magnetic separation, corn starch depressant, Aero 343 collector additions and froth collection time in reverse flotation) on the grades and recovery yields of specularite ore.

EXPERIMENTAL

Materials

Specularite sample was obtained from Feke district of Adana in Turkey. The sample contents have been determined by XRF (X-ray fluorescence) analysis using Minipal-4 Panalytical device. As shown in Table 1, the sample contains a high ratio of ${\rm Fe_2O_3}$. In addition, the results of the analysis showed high content of both silica and calcium oxide. The mineral phases of the sample have been revealed by XRD (X-ray diffraction) analysis. Rigaku Minflex XRD device has been used for this purpose. XRD patterns have been interpreted with the PDXL software with current database (Fig. 1).

The Fig. 2 was showed the microscope images of specularite ore sample under reflected light microscope (Nikon-YS100). The microscope images were taken by particle size and they were classified (a: -38 μ m, b: -74+38 μ m, c: -150+74 μ m, d: -500+150 μ m, e: -1000+500 μ m and f: -2000+1000

μm). In the microscopic images, white colored particles are quartz minerals while red, black and dark gray colored particles are iron minerals such as goethite, hematite and specularite. The sample, which had the maximum particle size of 90 mm, was crushed by a laboratory-type jaw crusher (<10 mm) and ground for 12 minutes using a ball mill (the charge volume was 45-50 % of the internal volume of the mill) at 60 rpm drum speed as the critical value in dry conditions. Then samples were sieved into different degrees of fineness: -2000+1000 μm, -1000+500 μm, -500+150 μm, -150+74 μm, -150 μm, -74 μm and -106 + 38 μm. In flotation tests, -38+0 μm sized materials were discharged by desliming. The particle size distribution is shown in Fig. 3.

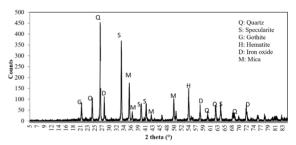


Figure 1. XRD analysis of the sample

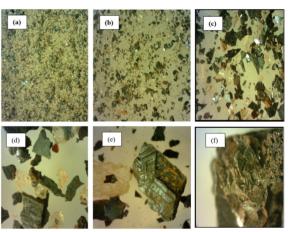
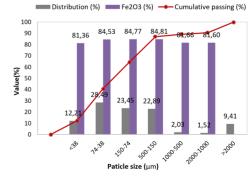


Figure 2. Microscope images of the different sized samples



 $\textbf{Figure 3.} \ \ \text{The particle size distribution of the sample}$

Table 1. "XRF analysis of the specularite sample.

Content	Fe ₂ O ₃	SiO ₂	CaO	SO ₃	La ₂ O ₃	MnO	HgO	Cr ₂ O ₃	CuO	Rb₂O	MoO ₃	Y ₂ O ₃	Eu ₂ O ₃	V ₂ O ₅
%	82.78	8.11	8.46	0.3	0.11	0.02	0.025	0.02	0.04	0.03	0.01	0.003	0.07	0.02

Methods

Wet Magnetic Separation Tests

Magnetic separation experiments were carried out using laboratory type high intensity wet magnetic separator (Eriez L4-20). Beneficiation of specularite sample using this separation method divided into two stages. The first stage, samples were prepared according to particle size at five different sizes:-2000+1000 µm, -1000+500 µm, $-500+150 \mu m$, $-150+74 \mu m$, $-74 \mu m$ and separation tests carried out at a fixed magnetic field intensity of 0.75 T. All the tests performed at 10% solid ratio by weight (25 g solid/225 ml water). The second stage was performed at 7 different magnetic field intensity; 0.25 T, 0.35 T, 0.45 T, 0.55 T, 0.65 T, 0.75 T and 0.85 T using optimum particle sized sample (-150 µm). This size was selected based on the results of the experiments of the first stage and the experiments in 2nd stage were performed at 20% solid ratio by weight (40 g solid/160 ml water).

Reverse Flotation Tests

The reverse flotation tests were carried out in a batch Denver type flotation machine equipped with a volume of 1 L cell and operating at a fixed impeller speed of 1200 rpm using tap water. The solid/liquid ratio was 10% by weight at the constant value of pH 12.5 in all experiments using sodium hydroxide granulated NaOH (grade>99.0%). Comparing to acidic medium, the flotation of iron oxides and silica was stronger in alkaline medium. As pH increases, the surface charge of oxides increases. The surface charges aim to become more negative, according to previous studies [14-18]. Methyl isobutyl carbinol (MIBC) was used in the experiments as frother and added to the flotation cell at a fixed dosage of 600 g/ton for all experiments. MIBC was added to the cell one minute before froth collection. The froth product was collected at certain time intervals. The particle size was -106+38 μm. MIBC dosage and the particle size were selected based on the preliminary tests. Corn starch is widely preferred in flotation of metallic ores due to its good depressant properties and their low cost [3, 19, 20]. The dosages of corn starch were between 500 - 10000 g/ton. The collector in these experiments was Aero 343. Collector dosages ranged from 500-1500 g/ton. Conditioning time for the depressant was 2 min and was 4 min for collector reagent. Then, it was mixed in 1200 rpm with adjusted pH.

Box Behnken test design, one of the DOE method for response surface, was used to evaluate flotation results. It was preferred due to the presence of more variables in reverse flotation experiments compared to magnetic separation. It is also possible to determine the active parameters more quickly with fewer experiments in this design. The models were created by using Design Expert 11 software shown in Table 2.

Table 2. Box Behnken test design parameters.

Run	A: Collector g/ton (x1)	B: Depressant g/ton (x2)	C: Time min. (x3)
1	1500	10000	2
2	1000	5250	2
3	1500	5250	1
4	1000	500	1
5	1000	10000	3
6	1500	500	2
7	1000	10000	1
8	1500	5250	3
9	1000	500	3
10	1000	5250	2
11	1000	5250	2
12	500	5250	1
13	500	500	2
14	500	10000	2
15	500	5250	3

After each experiment, both concentrate and tailings were dried over $105\,^{\circ}\text{C}$ and then weighed to calculate the recovery of the Fe_2O_3 by using the following equation:Where R is the recovery rate of iron concentrate, F is the weight of the feed solids (%), C is the weight of the concentrate (%), f is metal grade in the feed (%), c is the metal grade in the concentrate (%) [21, 22].

$$\%Recovery = R = \frac{Cc}{Ff}x100$$
 (1)

RESULTS AND DISCUSSION

In general, there is an inverse relationship between upgrading and recovery yield, so that when one of them increases, the other decreases. In this study, we tried as much as possible compatibility between them so that the results of the grade and recovery yield are high for both.

Wet Magnetic Separation

Parameters affecting the performance of the wet intensity magnetic separator are particle size and magnetic field intensity, mostly. The effect of each operating parameters on grade and recovery yield of iron concentrate was discussed in the following subtitles.

Effect of Feed Particle Size on Grade and Yield of Iron Concentrate

Wet magnetic separation experiments were carried out to investigate the effect of particle size on the specularite upgrading in the wet magnetic separation as shown in Fig. 4. The results showed that, at a fixed magnetic field intensity of 0.75 T and solid/liquid rate of 10% by weight, the particle size had a significant effect on both grade and yield of iron concentrate. Generally, when the

particle size of the specularite sample was reduced, iron concentrate grade gradually increased and reached to highest value (97,01%) at the particle size of 150 μm. Then, iron concentrate began decreasing lightly when the particle size was decreased. The particle size of the samples also had a particular effect on the iron concentrate yield as illustrated in Fig. 3. At particle size of 2000 µm, iron concentrate yield was at its lowest value as 36.26%. When particle size reduced from 2000 µm, iron concentrate yield significantly increased. A maximum recovery yield (67%) achieved at the particle size of -74 μm. In these experiments, the efficiency of the wet magnetic separator to attract concentrations of iron in large quantities and selectivity was increased by decreasing the particle size at magnetic field strength 0.75 Tesla. Therefore, both the grade and yield of the iron concentrate were increased by decreasing the particle size of the sample.

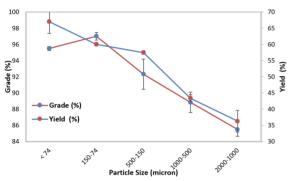


Figure 4. Concentrate grades and yields at different particle sizes in the magnetic separation

Effect of Magnetic Field Intensity on Grade and Yield of Iron Concentrate

Results of wet magnetic separation experiments were seen in Fig. 5. After the optimum particle size range was determined as -150+0 μ m, the low intensity wet magnetic separation tests were performed at 0.25 T, 0.35 T, 0.45 T, 0.55 T, 0.65 T, 0.75 T, and 0.85 T magnetic field strengths.

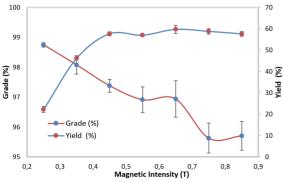


Figure 5. Concentrate grades and yields at different magnetic field intensities.

In Fig. 4, it was concluded that there was a significant effect of the magnetic field intensity on both iron concentrate grade and recovery yield by using wet magnetic separation. In general, iron concentrate grades decreased as the magnetic field intensities increased. When magnetic field intensity was low, the ability of the separator to attract concentrations containing iron-quartz reduced. Only liberated iron particles were attracted at low magnetic fields and this caused increasing ability of the separator to upgrading the sample but in return, yield decreased. At a magnetic field intensity of 0.25 T, iron concentrate grade was at its maximum value of 98.75%. After this point, iron concentrate grade began to decrease slightly with increasing magnetic field intensity due to the attraction of more quartz-iron bearing (non-liberated particles) particulates. At a magnetic field intensity of 0.55 T, iron concentrate grade was increased so slightly by increasing the magnetic field intensity from 96.91% to 96.94% at magnetic field intensity of 0.65 T. iron concentrate grade began to decrease slightly to 95.63 % at magnetic field intensity of 0.75 T which are considered at the lowest value of iron concentrate grade. Iron concentrate yield was directly proportional to the magnetic field intensity except in the intensities of the magnetic fields between 0.45 and 0.85 T, there was a slight variation of iron concentrate yield increases and decreases. The highest value of iron concentrate yield was 59.62 % at a magnetic field intensity of 0.65 T.

Reverse Flotation

Reverse flotation experiments were carried out to reveal the role of depressant dosage, collector dosage and froth collection time in the flotation separation of quartz from specularite. Table 3 shows the parameters (collector, depressant and froth collection time) as factors while both grade and yield of the iron concentrate as the response in reverse flotation test for all experiments, which were analyzed in Box-Behnken test design.

Effect of Depressant Dosage on Grade and Yield of the Iron Concentrate

The dosage of the corn starch had a significant and positive effect on iron concentrate grade. Generally, the grade of the iron concentrate was slightly increased with increasing depressant dosages. Fig. 6 shows the effect of the depressant on iron concentrate grade at 500 g/ton, 5250 g/ton and 10000 g/ton conditions, respectively. Using 500 g/ton depressant, iron concentrate grade was decreased at increasing collector and froth collection times. At a depressant dosages of 5250 g/ton, iron concentrate grade was slightly increased by increasing collector and froth collection times. The highest grade of iron concentrate (90.13%) was obtained the medium values of collector and froth collection time. At depressant dosage of 10000

Table 3. The factors and responses which were analyzed in Box Behnken test design in reverse flotation tests.

Run		Depressant g/ton (x2)	Time min. (x3)	Iron	Iron Concentrate Yield (%)	SiO2 (%)	CaO (%)
1	1500	10000	2	89.78	95.74	6.01	3.91
2	1000	5250	2	90.13	96.85	6.00	4.05
3	1500	5250	1	89.02	98.96	7.03	4.18
4	1000	500	1	88.67	98.65	7.00	4.06
5	1000	10000	3	89.98	93.80	6.01	3.75
6	1500	500	2	87.21	96.24	7.80	4.50
7	1000	10000	1	87.27	96.57	8.20	4.11
8	1500	5250	3	90.05	92.93	6.00	3.65
9	1000	500	3	88.03	93.15	7.50	4.24
10	1000	5250	2	89.09	95.05	7.01	3.98
11	1000	5250	2	89.06	95.04	7.03	4.14
12	500	5250	1	89.43	94.76	6.03	4.16
13	500	500	2	88.06	91.09	7.01	4.38
14	500	10000	2	88.54	93.23	7.10	4.08
15	500	5250	3	89.23	92.40	6.02	4.05

g/ton, iron concentrate grade was continue to increase with increasing both collector and froth collection time and the general effect of the depressant dosage on the iron concentrate grade at this dosage was more positively comparing at lower dosages of depressant. High pH values have the effect of increasing the effect of corn starch [3]. Experiments in this study conducted at pH 12.5 also resulted in >90% iron concentration grades (Fig. 6). Effect of the depressant dosages on speculariteat 500 g/ton, 5250 g/ton and 10000 g/ton were shown in Fig. 7.

As shown, the effect of the depressant on the iron concentrate yield was negative and the effect was almost the same for all experiments. At different dosages of depressant, the iron concentrate yield was increased with increasing collector dosage and decreasing froth collection time. The

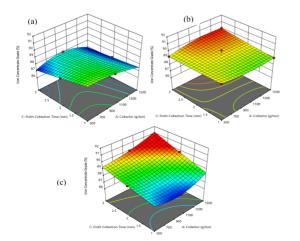


Figure 6. Effect of depressant dosage: 500 g/ton (a), 5250 g/ton (b), and 10000 g/ton (c) on iron grade (%)

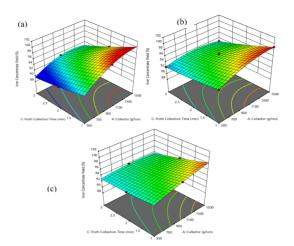


Figure 7. Effect of depressant dosage: 500 g/ton (a), 5250 g/ton (b) and 10000 g/ton (c) on iron yield (%)

highest value of iron concentrate recovery (98.96%) was achieved by 5252 g/ton of depressant usage at 1500 g/ton collector and 1 min froth collection time.

Effect of Collector Dosage on Grade and Yield of the Iron Concentrate

As seen in Fig. 8, under the highest depressant and froth collection time conditions, Fe,O3 grade increased according to the increase in the Aero 343 collector dosage. When 500 g/ton collector was used, iron concentrate grade was slightly increased with the increase of the depressant. Then, iron concentrate grade was slightly decreased with increased depressant values but iron concentrate grade continued to increase by increasing the depressant at froth collection time of 3 minutes. When collector was used more than 1000 g/ton and both of the depressant and froth collection time were at the highest values, the effect of Aero 343 on iron concentrate grade was positive. While the collector dosage was the lowest, more flotation time and highest values of depressant were needed to obtain high-grade concentrate. No significant increase was observed in the iron grades after 5000 g/ton of depressant and 2 min of froth collection time. At collector dosage of 1500 g/ton, the effect of the froth collection time on iron concentrate grade remained the same at the lower froth collection times (1-2 min). The maximum iron content was obtained with the depressant dosage (5252 g/ton) for 2 min (Fig. 8).

The effect of the 500 g/ton, 1000g/ton and 1500 g/ton collector dosages on iron concentrate yield were shown in Fig. 9. The collector dosage had a positive effect on iron concentrate yield. However, the iron concentrate yield increased according to the increase in the collector dosage and the decrease in both depressant dosage and froth collection time. iron concentrate yield reached its highest value at 1500 g/ton collector dosage. At this test, depressant and froth collection time were 500 g/ton and 1 min, respectively.

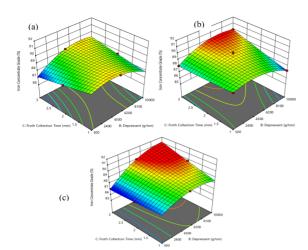


Figure 8. Effect of collector dosage: 500 g/ton (a), 1000 g/ton (b) and 1500 g/ton (c) on iron grade (%)

Effect of Froth Collection Time on Grade and Yield of the Iron Concentrate

The effect of froth collection time on iron concentrate grade was observed and the results obtained as shown in Fig. 10 at froth collection times of 1, 2 and 3 min, respectively. Generally, the effect of froth collection time on the iron concentrate grade was not stable. However, the effect was positive at 3 min and the highest values of depressant and collector. For 1 min collection time, iron concentrate grade was slightly increased with the increase in the depressant adding and then iron concentrate grade began to decrease slightly with the increase in the depressant dosage.

When 3350 g/ton depressant and 500 g/ton collector were used, the iron concentrate grade began to decrease slightly. Under the 1500 g/ton collector usage condition, a significant increase in ${\rm Fe_2O_3}$ grade seems to be obtained according to the increase in depressant dosage. While the other variables were at the highest value, the iron grade was obtained at the highest value for 3 min froth collection time.

Froth collection time had the greatest effect on the iron concentrate yield as shown in Fig. 11. When froth collection time was 1 min, where 5250 g/ton depressant and 1500 g/ton collector were used, iron concentrate yield reached 98.96%. In standard flotation tests, recovery yields increase with increasing froth collection times. Nevertheless, this phenomenon is vice versa in reverse flotation method which floats the gangue minerals. When the froth collection times were increased, the iron concentrate yields were decreasing, gradually. The yield reached its lowest value at the froth collection time of 3 min. iron concentrate yield was in its lowest value when the values of depressant and collector were at their lowest points. The grade values in Fig. 11 confirm the recovery yield values in figure.

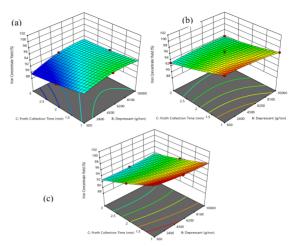


Figure 9. Effect of collector dosage: 500 g/ton (a), 1000 g/ton (b) and 1500 g/ton (c) on iron recovery yield (%)

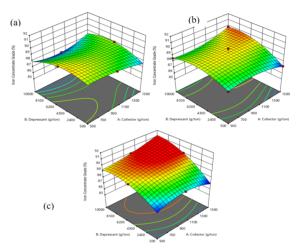


Figure 10. Effect of froth collection time (minute) on iron grade(%): 1 (a), 2 (b) and 3 (c)

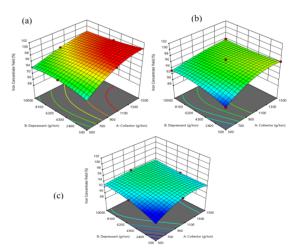


Figure 11. Effect of froth collection time (minute) on iron yield(%): 1 (a), 2 (b) and 3 (c)

ANOVA tests revealed that iron concentrate grade and recovery yields could be explained by cubic models using DOE. The correlation coefficients (R2) for iron concentrate

grade and yield were calculated as 0.94 and 0.97, respectively. It was shown a good correlation for parameters. The equations 2 and 3 show the obtained models:

Fe₂O₃grade (%) = 89.43 + 0.103
$$X_1$$

+ 0.138 X_2 + 0.518 X_3 + 0.523 X_1X_2
+ 0.30 X_1X_3 + 0.838 X_2X_3 - 0.042 X_1^2
-0.9887 X_2^2 + 0.048 X_3^2 + 0.625 $X_1^2X_2$
- 0.31 $X_1^2X_3$ - 0.005 $X_1X_2^2$
Fe₂O₃yield (%) = 95.65 + 1.18 X_1
- 0.358 X_2 - 20.7 X_3 - 0.66 X_1X_2
- 0.918 X_1X_3 + 0.683 X_2X_3 - 1.18 X_1^2
- 0.396 X_2^2 + 0.292 X_3^2 + 0.768 $X_1^2X_2$
- 0.03 $X_1^2X_3$ - 0.733 $X_1X_2^2$

Where; X_1 : collector dosage (g/ton), X_2 : depressant dosage (g/ton) and X_2 : froth collection time (min).

CONCLUSION

Upgraded iron ores are indispensable raw materials that can be used in iron-steel and paint industries. As a result, it was possible to obtain Fe₂O₂ concentrates in the grades that can be used by the industry (>85% Fe₂O₂) by separating the specularite mineral from the gangues using physical (magnetic) and physicochemical (flotation) separation techniques. The wet magnetic separation method was selected to beneficiate specularite ore in terms of iron concentrate grade. The optimum conditions of tests were determined as follows: 0.75 T magnetic field intensity, -74+0 μm particle size and 20% solid/liquid ratio. In the wet magnetic separation process, both particle size and magnetic field intensity had a significant effect on grade and recovery yield of iron concentrate. At constant magnetic field intensity of 0.75 T and solid/liquid ratio of 20% by weight, when the particle size of the specularite sample was reduced, iron concentrate grade gradually increased. At fixed particle size of -150+0 µm and solid/liquid ratio of 20%, iron concentrate grade was increased with decreasing magnetic field intensities. At 0.25 T magnetic field intensity, iron concentrate grade was at the highest value. In low intensity wet magnetic separation tests, when the magnetic field intensity was increased, the grade and yield were inversely proportional to each other. The maximum value of iron concentrate grade was 98.75% (with 22.23% yield) at 0.25 T, -150+0 μm particle size and 20% solid/liquid ratio while the highest value of iron concentrate recovery yield was 67% (with 95.49% iron concentrate grade) at 0.75 T and particle size of -74+0 µm and 20% solid/liquid ratio.

Flotation method in a magnetic field were used recently in numerous studies [23, 24]. In the next stage of the study,

it is planned to investigate the effect of the combination of these two methods by applying flotation in the magnetic field. In the reverse flotation method the optimum conditions were as follows: 5250 g/ton corn starch, 1000 g/ton Aero 343 collector, 600 g/ton MIBC, 2 min froth collection time at pH 12.5 and -106+38 micron particle size. The tests indicated that iron concentrate grade especially increased with the increase in corn starch dosages. Because the iron depression ability of the corn starch was positively related to the depressing of specularite ore and to its optimum solubility at high PH condition. Froth collection time also had a crucial effect on the iron concentrate grades. While the froth collection time was 1 min, the iron yield was the highest. When the froth collection time was 3 min, the iron content was the highest but the yield was low. The reverse flotation of quartz caused this result. As noted, collector dosage and froth collection time had almost the same effect on iron concentrate grade. Optimum flotation test conditions for the best iron concentrate grade (90.13% with 96.85% recovery yield) were as follows: 5250 g/ton corn starch, 1000 g/ton Aero 343 collector, 600 g/ton MIBC, 2 min froth collection time at pH 12.5 and -106+38 particle size. In terms of iron concentrate yield, the depressant had a negative effect, whereas the effect of the collector dosage was significant. Also, iron concentrate yields were increased by Aero 343 collector additions. The froth collection time had the greatest effect on the iron concentrate yield. The highest iron concentrate yield (98.96% with 89.02% iron concentrate grade) was obtained at optimum conditions: 5250 g/ton corn starch, 1500 g/ton Aero 343, 600 g/ton MIBC, 1 min froth collection time and -106+38 micron particle size.

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