Investigation of Efficiency of Magnetic Separation Methods for Processing of Low-Grade Iron Pigments Ore (RED Ochre)

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Abstract: In this research, the efficiency of magnetic separation methods for processing of a low-grade iron pigments ore (red ochre) has been studied. Based on the mineralogical analyses (XRD), thin section and polish studies, the reserve is an iron sedimentary deposit with an average Fe grade of %31.3. The most valuable minerals are Hematite and Goethite and main gangue minerals are Calcite and Quartz. Wet and dry high-intensity magnetic separation methods were applied for processing. The full factorial design was implemented for all of the wet high-intensity magnetic separation (WHIMS) experiments. Factors of magnetic field intensity, rotor speed and feed water flowrate were considered for design. In optimal conditions in rougher stage, WHIMS produced a concentrate with grade %42.92 Fe and recovery %62.23 in magnetic field intensity 1.7 Tesla, feed water flowrate 5 liter per minute and speed of rotor 3 rounds per minute; also after two stage cleaning WHIMS produced a concentrate with Fe grade and Fe recovery of %56.12 and %38.56, respectively. The results of the dry high-intensity magnetic separation for the two coarser fractions showed that size fraction of coarser than 1000 micron produced a concentrate with Fe grade %40.32 and relative Fe recovery %95.11 and size fraction of -1000+150 micron with Fe grade %45. 04 and relative Fe recovery %75.14. The results of experiments show that there is a low capability for improving quality and grade of this ore. Achieved concentrate from experiments could be used as an initial feed of producing pigment.

Keywords: Red Ochre, Iron, High-Intensity Magnetic Separation, Full Factorial Design

1. Introduction

Red ochre is one of the most important iron oxide forms. The main mineral of red ochre is Hematite (Fe_2O_3) and it uses in many industries such as paint, cement, chemical, cosmetics, plastic, paper, glass, ceramic and etc. [1-2]. There are many red ochre reserves in Iran which are mainly located in southern and central parts [3]. The majority of these deposits has a low grade and they should be processed to use in various industries [3-4]. Different separation methods such as Gravity, magnetic, flotation are mainly used for processing of these kinds of deposits [5-9]. There are a great number of researches on the separation principles [10], while a few applications have been accomplished in processing of low-grade ochre. In this research dry and wet high-intensity magnetic separation methods were applied in processing of red ochre.

2. Materials and Methods

2.1. Low Grade Ore Characteristics

A sample was taken from an iron sedimentary deposit which is located in the southern part of Iran. The sample had a size distribution finer than 5 mm. Total grade of iron in the sample was %31.3 and it is mainly in Hematite phase also a little in Goethite and Magnetite phases. Table 1 and figure 1 are shown Distribution of iron in various size fractions. XRD analysis, polish and thin section studies were done in order to complete mineralogical investigations of ore. Table 2 presents the results of XRD analysis. Figure 2 and 3 also shows two images of polish and thin sections which prepared to sample.

*Table 1. Distribution of iron in different size fractions.*

Size fraction (micron)	Iron grade (%)	Iron distribution (%)
+2000	30.6	4.52
-2000+1000	32.73	19.11
-1000+850	34.44	3.27
-850+600	34.38	7.14
-600+300	34.54	8.01
-300+212	31.59	6.62
-212+150	28.08	4.84
-150+75	27.88	4.76
-75	34.45	41.75
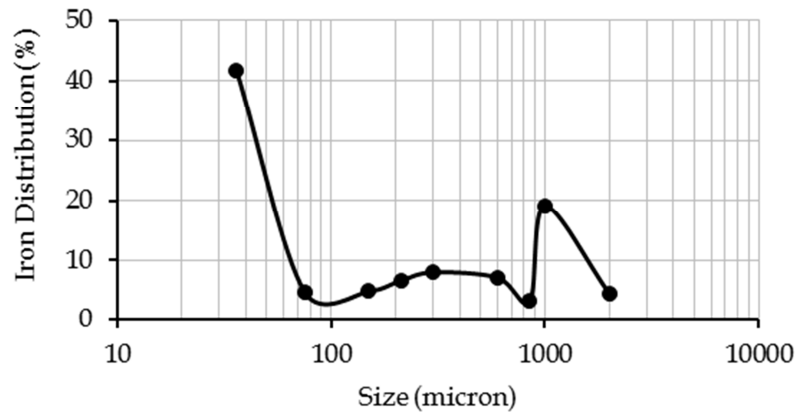
Figure 1. Iron distribution in different size fractions.

As shown in figure 1, distribution of iron in size fraction finer than 75 microns rather than larger size fractions has considerably increased. This is due to concentration of earthy Hematite in this size fraction.

Table 2. Mineralogical composition of the primary low-grade ore using XRD analysis.

Mineral	Chemical component	Value (%)
Hematite	Fe_2O_3	29.3
Quartz	SiO_2	20.4
Calcite	$CaCO_3$	15.5
Goethite	$FeO(OH)$	6.4
Halite	$NaCl$	2.5
Jarosite	$(K,H_3O)Fe_3(SO_4)_2(OH)_6$	5.5
Anorthite	$CaAl_2Si_2O_8$	4.2
Orthoclase	$KAlSi_3O_8$	3.6
Biotite	$KMg_3(Si_3Al)O_{10}(OH)_2$	3.8
Gypsum	$CaSO_4 \cdot 2H_2O$	3.5
Albite	$NaAlSi_3O_8$	1.6
Magnetite	Fe_3O_4	3.7
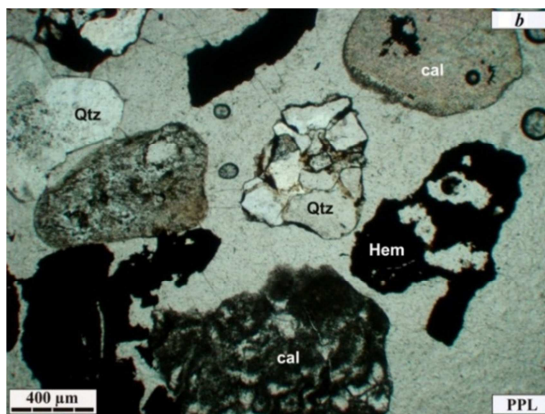
Figure 2. Thin section image of low-grade ochre ore, Hem: Hematite, Geo: Goethite, Qtz: Quartz, Cal: Calcite.
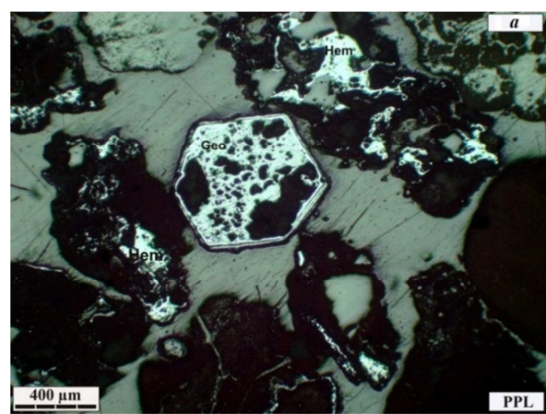
Figure 3. Polish section image of low-grade ochre ore, Hem: Hematite, Geo: Goethite, Qtz: Quartz, Cal: Calcite.

2.2. Procedure of Wet High-Intensity Magnetic Separation (WHIMS) Experiments

WHIMS experiments were done using high-intensity magnetic separator model Boxmag that was available in the mineral processing laboratory of college. Magnetic field intensity, feed water flowrate and speed of drum are adjustable factors of this separator.

2.3. Full Factorial Design

Many experiments should be done for achieving to optimized conditions in different processes which caused a lot of costs. So many researchers have focused on selecting the best conditions for effective factors on processes. Full factorial design is one of the experiment design methods which increased using it in recent years [11] and it was used to design WHIMS experiments in this research. Table 3 shows selected levels of each factor in magnetic separation rougher stage. Each factor was varied three levels.

Table 3. The level of factors in the full factorial design of high-intensity magnetic separation experiments.

Factor	Low level	Center level	High level
A: Field intensity (Ampere & Tesla)	1.2	1.45	1.7
B: Feed water flowrate (lpm)	2	3.5	5
C: Speed of drum (rpm)	1	2	3

2.4. Magnetic Experiments

Twelve magnetic experiments were designed using full factorial design. The experimental conditions and their responses are shown in Table 4. The results were added to "Design Expert (DX)" software and a quadratic model was chosen and fitted to them. Two models were fitted to Fe recovery and Fe grade.

Quadratic models found to be adequate for the prediction of the response variables are given by the following equations:

$$\text{Grade (\%)} = 41.14 + 2.25B \qquad (1)$$

$$\text{Recovery (\%)} = 60.26 + 1.95A + 4.22C - 3.1BC \qquad (2)$$

In these models all factors are in coded values and A, B and C are magnetic field intensity, feed water flowrate and Speed of drum, respectively. BC is the interaction of between B and C factors. Table 5 shows the analysis of variance (ANOVA) of the developed models. It demonstrates that all fitted models are significant at 95% confidence level (p-value < 0.05). Figures 4 and 5 represent predicted against actual values for Fe recovery and Fe grade, respectively. R-square Values of models are shown in these figures. The high R-square value indicates that the quadratic equation is capable of representing the system under the given experimental domain [12].

Table 4. Experimental scheme of magnetic tests and achieved results.

| Test No.	A Coded	A Actual (Ampere)	B Coded	B Actual (lpm)	C Coded	C Actual (rpm)	Concentrate Weight (%)	Fe recovery (%)	Fe grade (%)
1	−1	20	−1	2	−1	1	39.56	50.09	40.20
2	+1	40	−1	2	−1	1	44.59	57.05	39.20
3	−1	20	+1	5	−1	1	41.04	56.78	43.40
4	+1	40	+1	5	−1	1	43.30	60.24	43.17
5	−1	20	−1	2	+1	3	54.78	67.12	37.78
6	+1	40	−1	2	+1	3	57.64	69.31	38.38
7	−1	20	+1	5	+1	3	42.53	59.24	44.10
8	+1	40	+1	5	+1	3	45.54	62.23	42.92
9	0	30	0	3.5	0	2	42.46	58.37	43.20
10	0	30	0	3.5	0	2	41.02	57.06	43.35
11	0	30	0	3.5	0	2	41.50	58.89	43.06
12	0	30	0	3.5	0	2	41.45	57.93	43.95
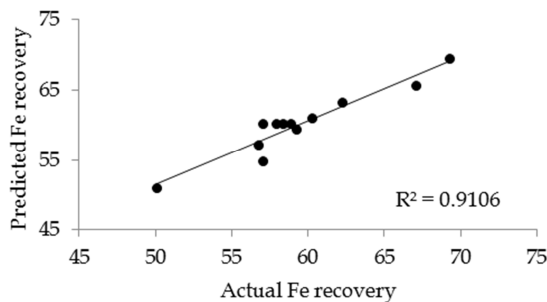
*Figure 4. Predicted vs. actual values of Fe recovery %.*
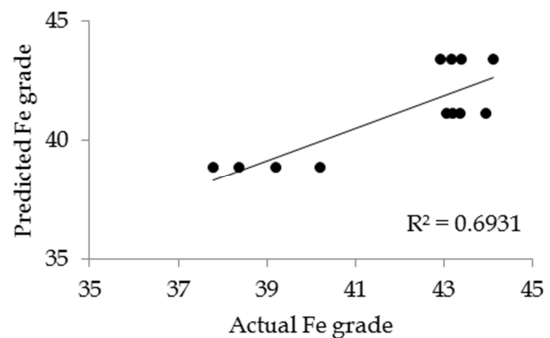
Figure 5. Predicted vs. actual values of Fe grade %.

Table 5. *The ANOVA results of selected factorial models.*

Source	Sum of Squares	df	Mean Square	F Value	p-value
Fe Grade	40.64	1	40.64	71.94	< 0.0001
Fe Recovery	249.85	3	83.28	49.90	< 0.0001

Main effects of factors on grade and recovery are indicated in Figures 6-8. These results were obtained at the midpoint of other factors. However, interaction between feed water flowrate and speed of drum factors had significant effects on recovery, therefore results were presented and discussed according to interaction. Figures 9 and 10 presents the effect of feed water flowrate and speed of drum on the Fe recovery.
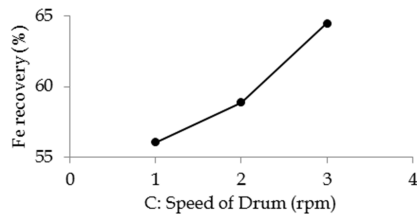
Figure 6. *Effect of drum speed factor on Fe recovery.*
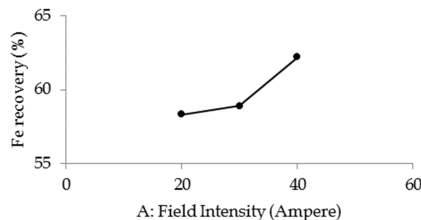
Figure 7. *Effect of field intensity factor on Fe recovery.*
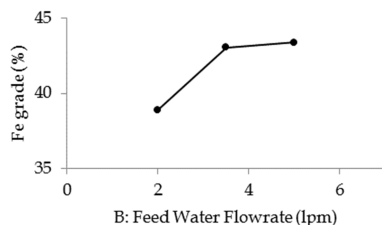
Figure 8. *Effect of feed water flowrate factor on Fe grade.*
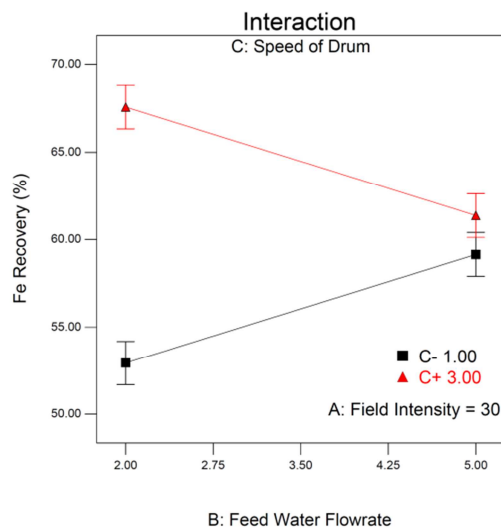
Figure 9. *Interaction effect of feed water flowrate and speed of drum on the Fe recovery in field intensity 30 ampere (A).*
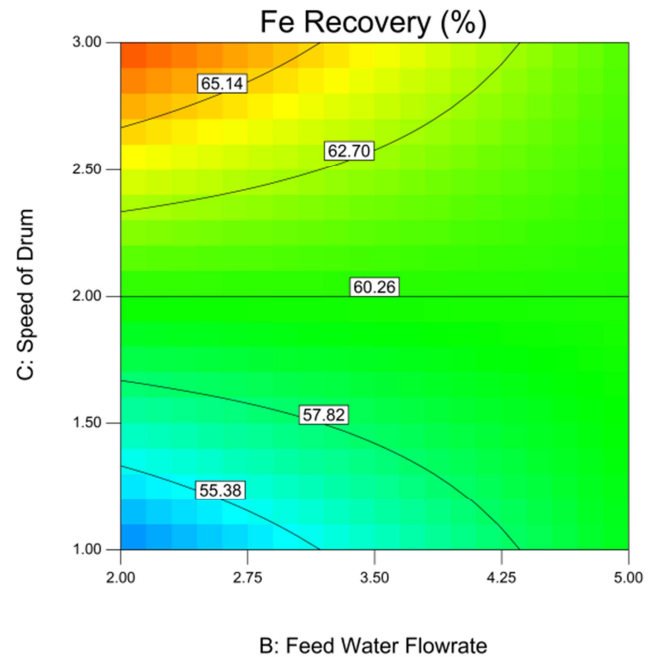
Figure 10. *Interaction effect of feed water flowrate and speed of drum on the Fe recovery in field intensity 30 ampere (A).*

As shown in Figure 6. Fe grade is related to the feed water flowrate and its value improves in terms of increasing feed water flowrate. Also field intensity and speed of drum factors have the main effects on Fe recovery and increasing of each factor causes to improve Fe recovery. Figure 7 represents the interaction effect of between feed water flowrate and speed of drum factors on Fe recovery. It is indicated that Fe recovery increases when the speed of drum factor is positioned on its low level, while Fe recovery decreases by selecting the high level of this factor.

2.5. Optimization of Experiments Results

The purpose of experiment design is to find a desirable situation in the design space which could be a maximum, a minimum, or an area where the response is stable over a range of factors [12]. In this research, a simultaneous optimization technique was used (by DX software) for optimization of multiple responses. Finding the conditions for achieving to maximum grade or minimum recovery was the target of these experiments. Several optimum conditions were shown in table 6.

As it has shown in table 6, achieved results of experiments confirm the models which presented by software. Maximum Fe recovery was reached to 69.31% using field intensity 40 ampere, feed water flowrate 2 lpm and speed of drum 3 rpm. Maximum Fe grade was also reached to 42.89% in the condition of field intensity 30 ampere, feed water flowrate 5 lpm and speed of drum 2.5 rpm and while Fe recovery was reached to 62.1%. An experiment was designed with target of Fe grade at least %40 and results show Fe grade and recovery was reached to 39.87% and 57.83%, respectively.

Table 6. Conditions and results of optimized experiments.

Condition	Levels of factors			Software results		Experiment results	
	A	B	C	Fe grade (%)	Fe recovery (%)	Fe grade (%)	Fe recovery (%)
Maximum grade	30	5	2.5	43.4	60.81	42.89	62.1
Maximum recovery	40	2	3	38.89	69.32	38.38	69.31
Fe grade: at least %40	40	3	1	40.4	57.37	39.87	57.83

2.6. Supplementary Experiments of High-Intensity Magnetic Separation
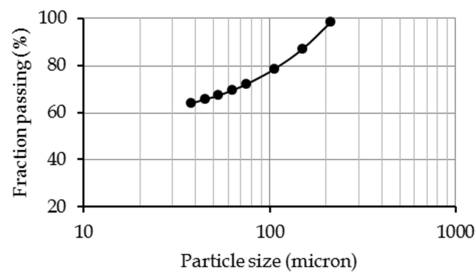

Figure 11. Size distribution plot of a representative sample from tailings of rougher experiments.

Several experiments were conducted on the tailings of experiments which done in rougher stage. At first, tailings of twelve rougher experiments were mixed together and the representative samples were selected after homogenizing. Average Fe grade of tailing sample was specified 21%. Some

magnetic experiments were implemented on these samples. Also the sieve analysis was done on a sample. The results of this sieve analysis were presented Figure 11.

Figure 11 is shown more than 64% of the rougher tailing sample is finer than 38 microns. So these samples were re-ground to a P80 of 35 microns and then three experiments with different conditions were implemented on them. Table 7 represents the results of magnetic experiments which were conducted on rougher tailings.

Tests 1 and 3 in table 7 were produced a concentrate with Fe grade lower than the Fe grade of rougher experiment feeds. Test no. 2 was produced a concentrate with a Fe grade of 32.75%, which is a little higher than rougher experiment feeds, although Fe recovery of this test was very low, 15.89%. Results of rougher tailing experiments were represented that there is a low potential for concentrating tailings of rougher experiments. So experiments were completed with target of maximum recovery in rougher stage and reprocessing of rougher concentrate.

Table 7. Obtained results of magnetic separation experiments on rougher tailings.

Test No.	Factor levels			Results		
	Field intensity (Tesla)	Feed water flowrate (lpm)	Speed of drum (rpm)	Concentrate Weight (%)	Fe grade (%)	Fe recovery (%)
1	1.7	2	3	32.96	24.92	39.11
2	1.45	3	1	10.19	32.75	15.89
3	1.2	3	2	18.43	28.47	24.98

2.7. Cleaner and Re-Cleaner Experiments on Rougher Concentrate

Several experiments with different operational conditions were implemented on rougher concentrate with the aim of increasing grade. Firstly, the maximum recovery was accounted as a target in experiments of rougher stage. Then experiments of

cleaner and re-cleaner stages were completed on rougher concentrate according to the best operating conditions on rougher stage. Cleaner experiment was completed on Concentrate of the rougher experiment no. 6 which produced a concentrate with maximum Fe recovery 69.31% and Fe grade 38.38%. Table 8 represents the levels of effective factors and the results of cleaner experiments on rougher concentrate.

Table 8. Achieved results of cleaner experiments on rougher concentrate.

Product	Weight (%)	Fe grade (%)	Relative Fe recovery (%)	Total Fe recovery (%)
Feed	100	38.38	100	69.31
Concentrate	63.90	48.16	70.18	48.64
Tailing	36.10	25.97	29.72	20.60

Cleaner experiment produced a concentrate with Fe grade and recovery of 48.16% and 48.64%, respectively, as shown in table 9. It is clear that the amount of recovery has considerably decreased, although Fe grade of cleaner

concentrate has risen to 48.16%. In order to investigate increasing of Fe grade, re-cleaner experiment was designed that implemented on concentrate of cleaner experiment. The results of this experiment are presented in table 9.

Table 9. Achieved results of re-cleaner experiment on cleaner concentrate.

Product	Weight (%)	Fe grade (%)	Relative Fe recovery (%)	Total Fe recovery (%)
Feed	100	48.16	100	48.64
Concentrate	68.04	56.12	79.28	38.56
Tailing	31.96	31.22	20.72	10.08

The results of re-cleaner experiment show that a concentrate was produced with Fe grade and recovery of 56.12% and 38.56%, respectively. Also, relative Fe recovery of cleaner and re-cleaner stages were 70.18% and 79.28%, while Fe recovery of rougher stage was 69.31%. Table 10 shows the final results of magnetic experiments with one rougher stage and two cleaner stages.

Table 10. Final results of magnetic experiments.

Product	Weight (%)	Fe grade (%)	Fe recovery (%)
Feed	100	31.48	100
Concentrate	25.06	56.12	38.56
Tailing	74.94	23.24	61.44

2.8. Dry High-intensity Magnetic Separation (DHIMS)

Dry high-intensity magnetic separators are proper for processing of coarse particles [13]. Dry high-intensity magnetic experiments were completed using dry induced roll separator which was available in mineral processing lab. Dividing blades between tailing, medium and concentrate parts are adjustable according to degree in this separator. Increasing angle of dividing medium-concentrate blade caused to increases Fe grade and decreases Fe recovery. Also increasing angle of dividing tailing-medium blade caused to pull more amount of feed to tailing.

Dry sieve analysis and Fe grade distribution on different size fractions were presented in Table 11. Regarding to Fe grade distribution on different size fractions, two samples were provided and tested from size fractions of -1000+150 micron and coarser than 1000 micron. All dry magnetic experiments were implemented in field intensity 1.7 tesla and speed of drum 80 rpm.

Table 11. Results of dry sieve analysis and Fe grade distribution on different size fractions.

Size fraction (micron)	Retained (%)	Fe grade (%)
+2000	6.6	30.6
-2000+1000	24.3	32.73
-1000+600	14.2	34.38
-600+300	16.4	34.54
-300+212	9.3	31.59
-212+150	3	28.08
-150	26.2	27.88

2.8.1. Dry Magnetic Experiments on Size Fraction Coarser than 1000 Micron

Several experiments were done on a sample that provided from size fraction coarser than 1000 micron. Tailing dividing blade was adjusted on 80 degrees and the particles that pulled into medium and concentrate parts, they were mixed and accounted as final concentrate. The final concentrate of first concentrating stage was reprocessed with conditions which were similar to first stage. Concentrate of cleaner stage was accounted as final concentrate. The results of dry magnetic experiments were presented in table 12.

Table 12. Obtained results of dry magnetic experiments on size fraction coarser than 1000 micron.

Product	Weight (%)	Fe grade (%)	Fe recovery (%)
First stage feed	100	31.39	100
First stage concentrate	77.62	39.24	97.003
First stage tailing	22.38	4.17	2.97
Second stage feed	77.62	39.24	97.03
Second stage concentrate	74.06	40.32	95.11
Second stage tailing	3.56	16.89	1.92
Final concentrate	74.06	40.32	95.11
Final tailing	3.56	16.89	1.92

The results of dry magnetic experiments showed that a concentrate was produced with Fe grade and recovery of 40.32% and 95.11%, respectively after two stages of concentrating. It is clear that one stage cleaning has very little effect on Fe grade and the majority of iron minerals locked within gangue materials.

2.8.2. Dry Magnetic Experiments on Size Fraction of -1000+150 Micron

In addition, several experiments were implemented on a sample that provided from size fraction of -1000+150 micron. Different angles of dividing blade were investigated in this part of experiments. Tailing dividing blade and concentrate dividing blade was finally adjusted on 80 and 90 degrees, respectively. First stage of magnetic separation had three products of concentrate, medium and tailing. Medium of first stage was reprocessed using separator. Concentrates of first and second stages were mixed together and they were defined as final concentrate. the results of these experiments were presented in table 13.

Table 13. Obtained results of dry magnetic experiments on size fraction of -1000+150 micron.

Product	Weight (%)	Fe grade (%)	Fe recovery (%)
First stage feed	100	31.33	100
First stage concentrate	29.32	46.07	43.11
First stage medium	56.84	30.86	55.94
First stage tailing	13.84	2.03	0.95
Second stage feed	56.84	30.86	55.94
Second stage concentrate	22.95	43.72	32.5
Second stage medium	28.70	24.15	22.49
Second stage tailing	5.19	5.66	0.95
Final concentrate	52.27	45.04	75.14
Final medium	28.70	24.15	22.49
Final tailing	19.03	3.02	2.37

Two stages of magnetic separation was produced a concentrate with Fe grade and recovery of 45.04% and 75.14%, respectively. The results of experiments which completed on this size fraction sample shows that liberation of iron minerals was slightly improved.

3. Results and Discussion

Depletion of high Fe grade ore obliges mineral companies to

concentrate low Fe grade ores in order to reach the quality demand of the steel mills [14]. Investigations and studies on processing low-grade ores showed that it is possible to increase the Fe grade from approximate 30% to more than 50%. In fact, the obtained concentrates when compared with the magnetic concentration feed, it is deemed satisfactory for rougher operation or even blast furnace [15-17]. In this paper, the iron

low-grade ore sample -with size distributions finer than 5 mm and concentration of more than 40% iron content at the particles finer than 75 microns- is processed. As shown at the table 14, there is the possibility of achieving to a concentrate with Fe grade more than 55% although Fe recovery is considerable lower than achieved values at the other papers.

Table 14. *The summary of experiments and achieved results.*

Processing Method	Experiment Conditions	The achieved concentrate		Remarks
		Fe grade (%)	Fe recovery (%)	
WHIMS	Using full factorial design for implementation of tests on the representative sample	42.89	62.1	Finding the optimised point by changing of parameters values Investigating of operational parameters effects on grade and recovery
	Performing additional tests on tailings of experiments which done in the first stage	32.75	15.89	Low processing potential of first experiments tailings due to low recovery of fine particles
	Implementing of Cleaner and re-cleaner tests on the first stage concentrate	56.12	38.56	Maximum achievable Fe grade during the experiments
DHIMS	Experimets on size fraction coarser than 1000 micron	40.32	95.11	Slight increasing of Fe grade due to majority of iron minerals locked within gangue materials at these size fractions
	Experiments on size fraction of -1000+150 micron	45.04	75.14	

Wet magnetic separation methods in comparison with dry processing methods are more efficient, because the most of iron minerals for this kind of ores are concentrated at fine particles and dry magnetic separation methods doesn't have appropriate efficiency for fine particles [18]. Degree of liberation is one of the most factors for selecting of processing method [13]. As mentioned above, because of low liberation degree of iron minerals at size fractions larger than 150 microns, DHIMS methods aren't able to increasing of iron grade considerably.

4. Conclusions

Mineralogical analyses, thin section and polish studies have specified that the deposit is a kind of low-grade iron pigment containing Fe grade 31% and earthy Hematite is main iron mineral. Wet and dry high intensity magnetic experiments were performed on this deposit. Rougher stage of WHIMS experiments was completed using full factorial design and field intensity, feed water flowrate and speed of drum were the effective factors on separator performance. Two quadratic models were developed for Fe grade and Fe recovery. Feed water flowrate was the most effective factor on Fe grade. Also speed of drum and field intensity and interaction between feed water flowrate and speed of drum were the most important factors which effect on Fe recovery. First stage of WHIMS was produced a concentrate with Fe grade and recovery of 38.38% and 69.31%, respectively while optimum operational conditions of separator were field intensity 1.7 tesla, feed water flowrate 5 lpm and speed of drum 2 rpm. Also two cleaning stages of rougher concentrate on WHIMS experiments was produced a concentrate with Fe grade and recovery of 56.12% and 38.56%, respectively. The results of WHIMS experiments showed that Fe recovery sharply decreases after two stage of cleaning because of being Hematite mineral in earhty form in this deposit. Dry

high intensity experiments produced a concentrate with Fe grade of %40.32 and relative Fe recovery of %95.11 on size fraction coarser than 1000 micron and -1000+150 micron with grade %45.04 Fe and relative recovery %75.14. This ore was a representative sample of low-grade iron pigment deposits in Iran and results of experiments shows that there is a low capability for improving of their qualities and grade. Concentrate which obtained from experiment could be used as an initial feed of producing pigment.

Acknowledgements

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