

A Study on the Beneficiation of Low Grade Ilmenite Ore for Industrial Applications

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Abstract

Ilmenite is a very important source for titanium. Egyptian ilmenite ore contains 26.20 % TiO_2 , 50.09 % Fe_2O_3 and 14.35 % SiO_2 . Ore contaminates of silica and iron oxides should be removed to achieve a suitable ilmenite grade. This study aims at upgrading Abu Ghouson ilmenite deposits at the Eastern Desert of Egypt. Beneficiation of fine low grade ilmenite ore was successfully performed using Falcon SB40 concentrator. Falcon SB40 was tested for different ilmenite size fractions. It is displayed that falcon separation efficiency increased with decreasing feed size as well as with narrow size fractions while falcon is limited to size fraction less than 25 micron. Results of ilmenite feed -80+25 micron showed that a concentrate of 40.60 % TiO_2 with 86.00 % recovery and 2.43 % SiO_2 was achieved. Applying magnetic separation on the falcon heavy fraction, a final concentrate with 46.31 % TiO_2 and 1.78 % SiO_2 was obtained. The produced ilmenite concentrate coincide the standard specifications required for different industrial applications.

Keywords: Ilmenite, Titanium dioxide, Falcon concentrator, Box-Behnken design

INTRODUCTION

Egypt possesses a massive capacity of mineral resources that would be utilized to cover a part of the industrial requirements. Naturally occurring titanium ores are the raw materials for many commercial products. Ilmenite, FeTiO_3 , is considered as the most widespread titanium bearing mineral and is composed of about 43-65% titanium dioxide (TiO_2) (Heikal et al. 2019). However, ilmenite ore contains unacceptable contents of gangue minerals such as silicates and iron oxides that make them undesirable for use in industrial processes without purification.

Titanium (Ti) metal has strong corrosion resistance and strength to weight ratio. Therefore it is alloyed with metals such as vanadium and aluminium for use in aircraft, spacecraft, jet engines and space applications (Abdou et al. 2011) Titanium is also used in chemical, marine, medical and ceramic industries. The major use of titanium in the industry is in its oxide form. Titanium dioxide, TiO_2 , is employed as a white pigment for rubber, plastics, paints and paper industries (Xu et al. 2014; Gázquez et al. 2014).

In Egypt, ilmenite resources are found in rock and beach deposits. The rock deposit occurs in Abu Ghalaga and Abu Ghouson in Eastern Desert while the beach deposit occurs in Rosetta, east Alexandria (Heikal et al. 2019). Abu Ghouson Ilmenite ore includes a high content of iron oxides and silicates. Therefore it is considered as low grade titanium ore with TiO_2 of lower than 28%.

Ore beneficiation to an industrial feedstock grade is favourable before chemical processes as it minimizes size and energy requirements as well as it significantly reduces the complexity of chemical processes and increasing their efficiency (Hassan et al. 2020). Beneficiation processes of titanium minerals include gravity, magnetic, electrostatic and flotation separation techniques (Abd El-Rahman et al. 2006; Fan and Rowson 2002).

Abd El-Rahman et al.(2006) studied the upgrading of Egyptian Ilmenite ore using shaking table concentrator followed by wet high-intensity magnetic separator and a final concentrate of 39% TiO₂ and 2.48% SiO₂ was obtained. Applying sequential operations, thermal heating, attrition scrubbing and desliming, on the magnetic concentrate increased the TiO₂ to 43% and decreased silica to 2%. Fan and Rowson (2002) studied the surface modification of ilmenite using microwave radiation. They observed an increase of ilmenite recovery by 20 %. This is probably due to the conversion of Fe⁺² on ilmenite surface into Fe⁺³ which enhanced ilmenite floatability.

Nowadays, falcon gravity concentrator is used to recover very fine minerals (Kroll-Rabotin and Sanders 2014). Falcon SB40 concentrator is a semi-batch centrifugal unit which is operating at higher gravitational forces, up to 300 G's. Falcon SB40 is successfully used for upgrading fine low grade cassiterite ore while shaking table failed to recover it (Abd El-Rahman et al. 2009). Falcon is also used in oil shale upgrading in order to recover kerogen as a source of alternative energy (Yehia et al. 2017). Marion et al. (2017) investigated the separation capacity for the dense medium of fine sized minerals using falcon separator. Results showed that both laboratory centrifuge and falcon separator resulted in a similar good performance for upgrading of rare earth minerals. Aydogan and Kademli (2019) studied the effect of size distribution on falcon concentrator efficiency. Results showed that the performance of falcon increased with narrow size fractions.

This work investigates the upgrading of Egyptian Ilmenite ore using different physical separation techniques. The beneficiation is performed using falcon SB40 concentrator and Eriez wet low intensity magnetic separator. A Box-Behnken design expert is applied to evaluate the best conditions for the beneficiation process to acquire high grade and recovery values.

2. MATERIALS AND METHODS

2.1. Ore Preparation

The Ore samples were collected from Abu Ghouson area, which is located in the South part of the Eastern Desert. Samples were crushed using Jaw crusher. Then the crushed sample was milled using rod mill to obtain the suitable size fractions for falcon SB40 concentrator.

2.2. Ilmenite Beneficiation Using Falcon SB40

Selection of gravity techniques depends on particle size as well as liberation behaviour of the material. So, Falcon technique was applied to recover the following size fractions; -200-0, -200+125, -125+80, -80 +45, -45+25 and -25 micron.

Falcon SB40 Experiments: Ilmenite beneficiation tests have been performed in a falcon SB40 centrifugal concentrator. Feed was once delivered as slurry via the central vertical feed pipe and accelerated using the impeller. Rapid stratification took place in accordance to specific gravity as the feed is driven up the segregation zone under the effect of a sizeable gravity field. In the separation zone, which is straight away above the migration zone, fluidization water was injected via the rotor wall to set up a fluidized bed. Dense particles became embedded in the separation zone and were retained until the falcon is stopped then rinsed down through the concentrate discharge ports. The produced concentrates were cleaned twice for obtaining high grade concentrates. The produced concentrates and tails were collected, dried, weighed and chemically analyzed (Abd El-Rahman et al. 2009; Yehia et al. 2017).

2.3. Magnetic Separation Experiments

Wet low intensity magnetic separation experiments have been performed using Eriez wet low intensity magnetic separator. The drum is moving clockwise in order to pack up the ferromagnetic particles only. The separating zone is spherical in shape with actual filling volume of 6 liters. The feed was conditioned in order to avoid particles agglomeration. The slurry was passed slowly through drum of wet magnetic separator. Magnetite was adhered to the roll and collected at the nonmagnetic zone while the ilmenite fraction was retained in the spherical separation zone and collected at the end. The parameters have been studied are separation time and feed pulp density (solid/ liquid ratio). Magnetite and ilmenite fractions were weighed and chemically analyzed.

2.4. Instrumentation

The chemical composition of the raw sample and the products were obtained using X-ray fluorescence spectrometry. The change in the mineral phases was obtained by X-ray powder diffraction (XRD, Philips APD-3720) with Cu K alpha radiation worked at 20 mA and 40 kV in the 2θ range of 5-80 at a scanning speed of 2°/min.

3. RESULTS AND DISCUSSION

3.1. Characterization of raw sample

The XRD pattern of the raw sample shows the presences of several mineral phases dominated by ilmenite, hematite, magnetite, albite and quartz, Figure 1.

The complete chemical analysis of the sample shows that it is composed of: 26.20% TiO₂, 50.09% Fe₂O₃, 14.35% SiO₂ and 4.33% Al₂O₃, Table 1.

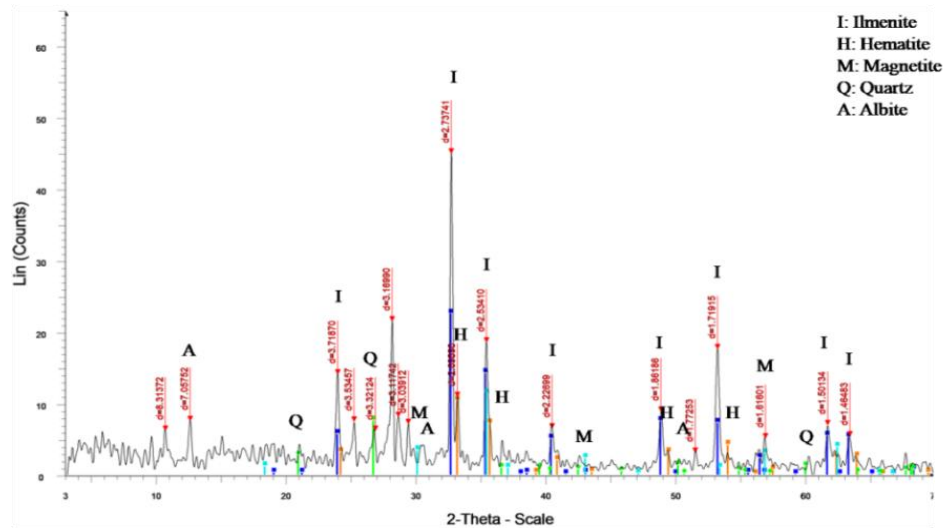


Fig. 1: XRD of the original ilmenite ore sample

Table 1: Chemical analysis of original ilmenite ore sample

Product	TiO ₂	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	CaO	L.O.I
Original ilmenite ore	26.20	50.09	14.35	4.33	1.75	0.99	0.33	1.94

3.2. Ilmenite Beneficiation using falcon SB40 Concentrator

The successful beneficiation of ilmenite mineral greatly depends on the ore physiochemical properties. Such as the difference in specific gravity between ilmenite and the gangue minerals, its response to a magnetic field, the ilmenite content and depends on the ore nature. The falcon gravity technique utilizes the earth’s natural gravitational (1G) force to recover the heavy ilmenite and iron oxide minerals from the lighter silicate minerals. This process applies enhanced gravity technique via using centrifugal forces, up to 300G, for efficient separation.

3.2.1. effect of size fraction on ilmenite beneficiation

Due to the considerable difference in specific gravity between valuable ilmenite mineral and gangue silicate; gravity technique is recommended (Hassan et al. 2017). In this study, falcon SB40 concentrator is applied as a gravity technique to recover the fine low-grade ilmenite ore sample of different feed size fractions; -200+0, -200+125, -125+80, -80 +45, -45+25 and - 25 micron, Table 2. Different ilmenite size fractions were tested using falcon SB40 separator at: fluidization water (4 psi) and centrifugal field (175 G’s), Table 3. It was shown that decreasing ilmenite size fraction increased titanium dioxide grade and recovery meanwhile decreased iron oxide and silicate contaminates.

Table 2: Chemical analysis of different ilmenite size fractions

Size fractions (micron)	wt%	TiO ₂	Fe ₂ O ₃	SiO ₂
Original (-200+0)	100.00	26.20	50.09	14.35
(-200+125)	16.02	26.09	50.33	14.48
(-125+80)	17.15	26.13	50.22	14.41
(-80+45)	25.40	26.23	50.09	14.36
(-45+25)	30.23	26.24	50.07	14.33
(-25)	11.20	26.29	49.77	14.12

Table 3: Size effect on ilmenite beneficiation using falcon SB40

Size fractions (μm)	Products	Wt, %	TiO ₂ , %	TiO ₂ recovery %	Fe ₂ O ₃ , %	Fe ₂ O ₃ recovery %	SiO ₂ , %	SiO ₂ recovery %
(-200+125)	Feed	100.00	26.09		50.33		14.48	
	conc.	39.25	33.78	50.82	51.99	40.54	5.59	15.15
	tail	60.75	21.12	49.18	49.21	59.40	20.23	84.87
(-125+80)	Feed	100.00	26.13		50.22		14.41	
	conc.	47.70	35.26	64.37	52.28	49.66	3.66	12.12
	tail	52.30	17.79	35.61	47.56	49.53	24.22	87.90
(-80+45)	Feed	100.00	26.23		50.09		14.36	
	conc.	53.25	38.35	77.86	53.42	56.79	3.12	11.57
	tail	46.75	12.42	22.14	42.14	39.33	27.19	88.52
(-45+25)	Feed	100.00	26.24		50.07		14.33	
	conc.	52.42	37.43	74.77	52.91	55.39	3.20	11.71
	tail	47.58	13.89	25.19	42.88	40.75	26.61	88.35
(-25)	Feed	100.00	26.29		49.77		14.12	
	conc.	45.82	33.32	58.07	51.08	47.03	4.02	13.05
	tail	54.18	20.35	41.94	47.23	51.41	22.68	87.03
Original (-200+0)	Feed	100.00	26.20		50.09		14.35	
	conc.	34.25	31.17	40.75	50.98	34.86	6.80	16.23
	tail	65.75	23.62	59.28	49.65	65.17	18.32	83.94

Figures 2 and 3 show the effect of size fraction on ilmenite beneficiation using falcon variables, fluidization water and centrifugal field. It was demonstrated that the separation efficiency is limited to size fraction less than 25 micron and this is in a good agreement with the literature as it was indicated that falcon SB40 model showed some limitations at size fractions lower than 25 microns (Yehia et al. 2017). It is also displayed that falcon separation efficiency increased with narrow size fractions, and there is a distinct recovery difference between the original sample of size fraction -200+0 micron and the rest of fractions which have narrow particle distributions. This is agreed with the work of Aydogan and Kademli, 2019.

Both size fractions, (-80 +45 micron) and (-45+25 micron), resulted in efficient ilmenite beneficiation as high TiO₂ grade and recovery is achieved at fluidization water 3-4 psi and centrifugal field 175-200 G's, Table 3 and Figures 2, 3. Therefore, falcon SB40 concentrator could be applied on size fraction range -80+25 μm .

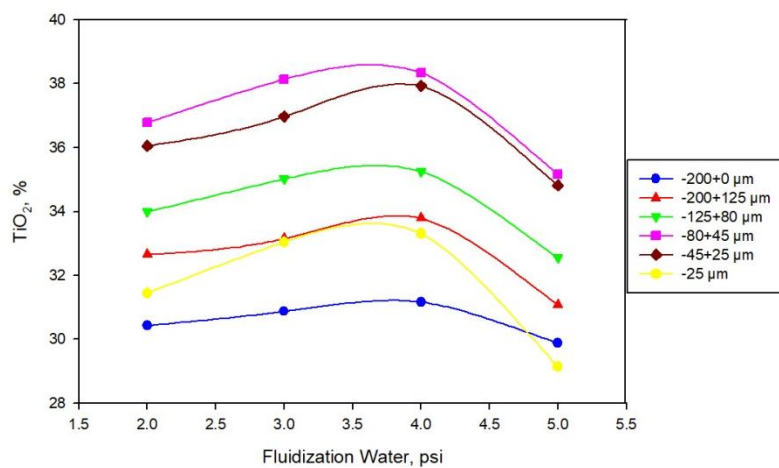


Fig. 2: Effect of different size fractions on ilmenite beneficiation using Falcon SB40 variable fluidization water at centrifugal field 175 G's and feed rate 100g/min

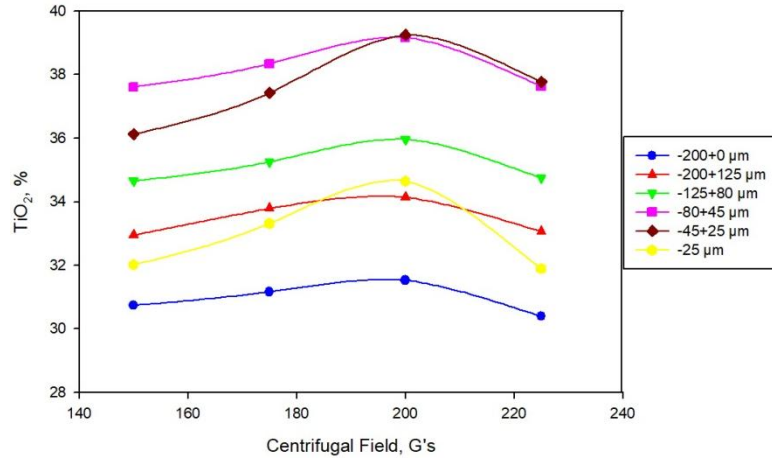


Fig. 3: Effect of different size fractions on ilmenite beneficiation using Falcon SB40 variable centrifugal force at fluidization water 4 psi and feed rate 100g/min

3.2.2. Applying Box-Behnken Design.

An experimental design technique, Box-Behnken Design, is used for beneficiation optimization, using falcon SB40 concentrator, for the size fraction -80+25 micron. This study permits the investigation of the effect of each factor, centrifugal field, fluidization water and feed rate, together with the interactions between factors. The optimum parameters were estimated according to the design using a second order polynomial function. Using this function, a correlation between studied parameters and response was created. The general form of this equation is (Hassan et al. 2017; Rostom et al. 2020; Abdel-Khalek et al. 2019a): (Eqn.1)

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2$$

Where Y is the predicted response; titanium oxide grade and recovery %, X₁, X₂ and X₃ are studied variables: centrifugal field, fluidization water and feed rate; β_{ij} are equation constants and coefficients.

The analysis of variance, ANOVA, was utilized in order to evaluate the statistical parameters. The determination coefficient, R², was used to demonstrate the degree of convenience of the experimental results to the polynomial model equation. The significance of all terms in the polynomial equation was estimated using F-test. The adequate precision measures the signal to noise ratio. An adequate signal is indicated when the ratio is greater than 4 (Rostom et al. 2020). The analysis of variance of the ilmenite beneficiation system ensures the well convenience of the experimental results to the polynomial model equation and therefore the accuracy of this model, Table 4. The model F-values of 51.33 and 120.61 indicates the model is significant. The adequate precision ratios of 18.31 and 38.84 imply an adequate signal. The grade and recovery of TiO₂ could be calculated by using equations (2) and (3), which were derived from the design:

$$TiO_2 \% = (Eqn. 2) + 40.5 - 1.07 \times A + 2.40 \times B - 2.70 \times C - 4.24 \times A^2 - 3.21 \times B^2 - 2.80 \times C^2 + 0.005 \times A \times B + 0.77 \times A \times C - 0.33 \times B \times C$$

$$TiO_2 \text{ Recovery } \% = (Eqn. 3) + 86 + 2.05 \times A - 1.37 \times B - 0.30 \times C - 0.81 \times A^2 - 0.81 \times B^2 - 0.46 \times C^2 - 0.13 \times A \times B - 0.17 \times A \times C - 0.38 \times B \times C$$

Where: A is the centrifugal field (G's), B is fluidization water (psi) and C is feed rate (g/min)

Table 4: Anova of response surface model for ilmenite beneficiation

The statistical parameters	Falcon SB40	
	TiO ₂ Grade %	TiO ₂ Recovery %
The standard deviation	0.790	0.230
R-Squared	0.985	0.994
Adequate Precision	18.31	38.84
The model F-values	51.33	120.61

A product of 40.5 % TiO₂ was obtained at fluidization water of 3.60 psi and centrifugal field of 194 G's. Further increasing of both fluidization water and centrifugal field leads to decreasing in ilmenite grade value, Figure 4 a, b.

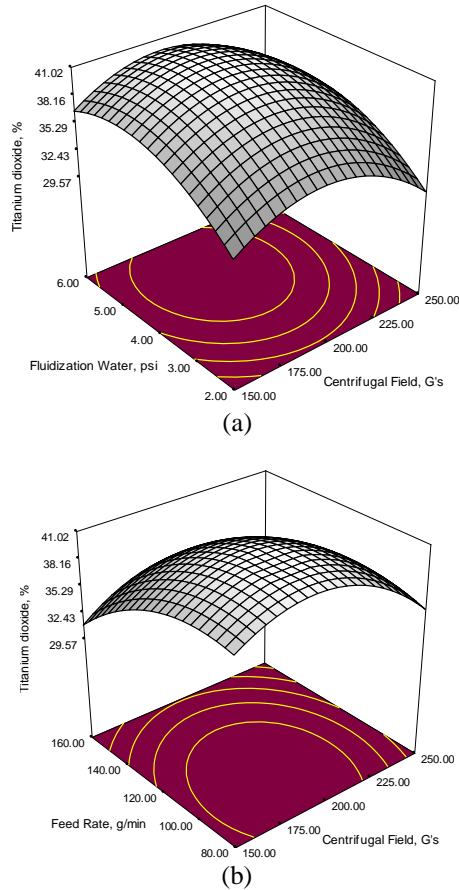


Fig. 4: The response surface plots a, b of titanium dioxide resulting from the main effects of Falcon SB40 variables, fluidization water, centrifugal field and feed rate

Falcon bowl has a surface of the limited fluidized groove at the top and a large conical non-fluidized section at the bottom. The lower fluidization water was not sufficient for escaping the lighter particles (quartz and albite) from heavier ones (ilmenite and iron oxides) which were held inside the falcon bowl; thus lower ilmenite grade was obtained. Accordingly, Falcon usually requires more fluidization water. In the retention zone, fluidization water creates a fluidized bed. Therefore, increasing the fluidization water increased grade of TiO_2 .

Increasing the gravitational force higher than 200 G's decreased the TiO_2 grade as the lighter quartz particles had the chance to hold inside the falcon bowl together with the ilmenite dense particles at very high G's.

At the high centrifugal field, high recoveries, up to 86% were obtained at low feed rate and fluidization water values. Increasing both feed rate and fluidization water decreased the recovery, Figure 5 a, b.

At moderate gravitational force values, 150 G's, low ilmenite recovery was obtained even with high water pressure. This is maybe due to the falcon rotation velocity was not sufficient to hold the dense ilmenite particles. Increasing the gravitational force to 250 G's increased the recovery levels. The higher G's was more suitable to hold a higher amount of dense ilmenite particles. Otherwise, the recovery decreased at fluidization water higher than 4 psi as some ilmenite particles escaped to the light particles zone.

High TiO_2 grade and recovery were obtained at a low feed rate. Increasing feed rate affected the segregation of particles and reduced the beneficiation efficiency of silicate particles from ilmenite ones.

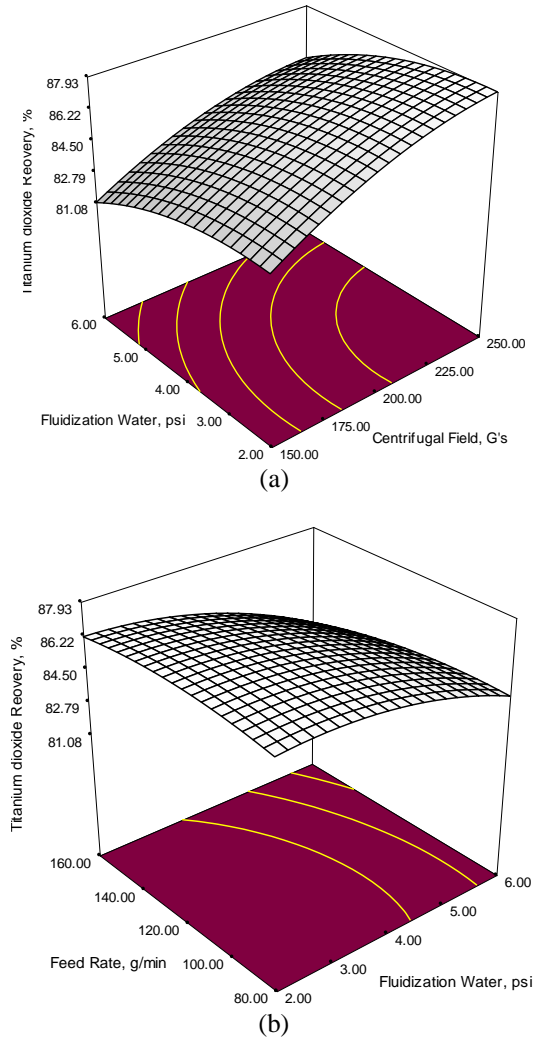


Fig. 5: The response surface plots a, b of titanium dioxide recovery resulting from main effects of Falcon SB40 variables, fluidization water, centrifugal field and feed rate

The optimal variables of the Box-Behnken design of ilmenite beneficiation using falcon SB40 separator are: feed rate (102 g/min), fluidization water (3.60 psi) and centrifugal field (194.4 G's). With these optimum parameters a concentrate containing 40.61% TiO₂ was obtained with operational recovery of 85.99%.

The XRD patterns of the falcon ilmenite concentrate and tail show the well separation of ilmenite from silicate minerals, Figure 6 a, 6. The XRF analysis of the falcon concentrate shows the increase of TiO₂ from 26.22 to 40.60% while SiO₂ is decreased from 14.34 to 2.40%., Table 5.

Table 5: Chemical analysis of the falcon SB40 products

Product	TiO ₂	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	CaO	L.O.I
Ilmenite feed (-80+25 micron)	26.23	50.08	14.34	4.31	1.76	0.98	0.34	1.94
Falcon concentrate	40.60	54.55	2.43	0.36	0.35	0.21	0.08	1.40
Falcon tail	9.32	37.78	29.65	10.14	4.95	3.26	1.89	2.98

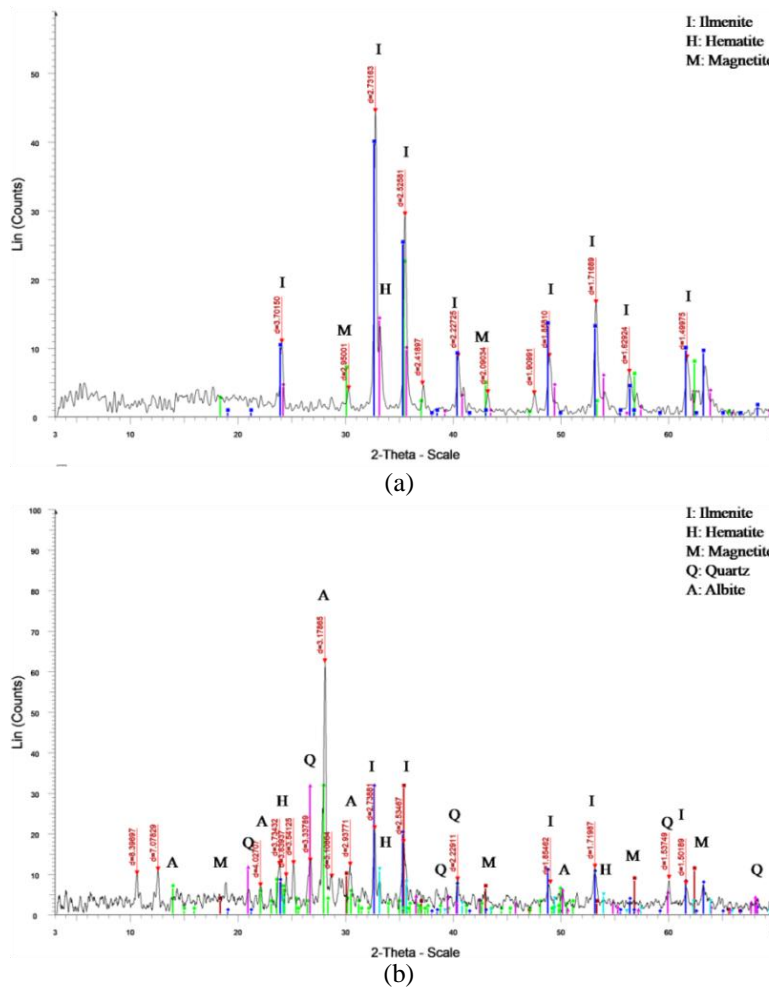


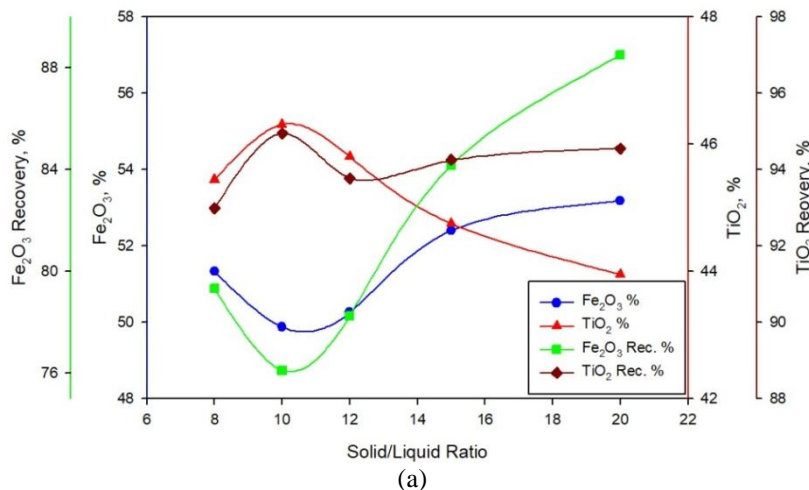
Fig. 6: XRD of a) Falcon ilmenite concentrate and b) Falcon tail (gangue minerals)

3.3. Upgrading of Falcon concentrate using magnetic separation:

Magnetic separation technique was applied, using Eriez WLIMS, on the falcon ilmenite concentrate in order to enhance the upgrading process. It was shown from XRD and XRF analyses that the falcon concentrate contains mainly ilmenite and iron oxides which are magnetite and hematite.

Low field intensity is necessary for efficient separation of the paramagnetic ilmenite mineral from the ferromagnetic magnetite (Abdel-Khalek et al. 2019b). Therefore, decreasing the field intensity to a minimum value (0.2 tesla) increased both grade and recovery of TiO₂. The parameters studied are; solid/liquid ratio and separation time.

High TiO₂ grade and recovery were obtained at average solid/liquid ratio. Increasing solid/liquid ratio more than 12% affected the distribution of the ferromagnetic and paramagnetic particles and resulted in crowding inside the spherical separation zone and hence reduced the separation efficiency, Figure 7a. Also, increasing the time of separation up to 20 min. They have increased the beneficiation efficiency of TiO₂. Separation time more than 20 minutes have no great effect on the process, Figure 7b.



(a)

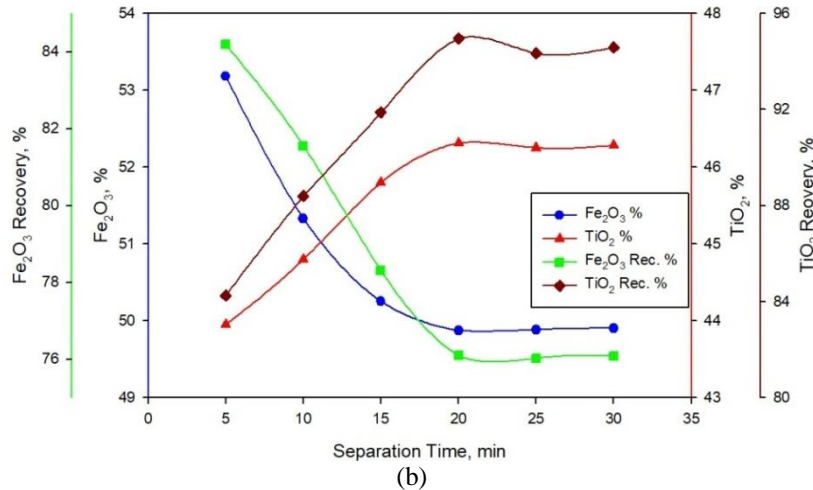


Fig. 7: Effect of a) solid/liquid ratio and b) separation time on the upgrading of falcon ilmenite concentrate using Eriez wet low-intensity magnetic separator

The best parameters of ilmenite upgrading using low intensity magnetic separator are 21% separation time, 12% solid/liquid ratio at minimum field intensity (0.2 teslas). With these optimum parameters, a concentrate containing 46.31% TiO₂ and 2.24% SiO₂ was obtained with a functional recovery of 94.9%, Table 6.

The XRD patterns of the weakly paramagnetic ilmenite concentrate show the efficient beneficiation of ilmenite from gangue minerals. Ilmenite represents approximately 86% with about 10% hematite mineral, Figure 8. Applying the optimum parameters of the Box-Behnken Design of ilmenite beneficiation using falcon SB40 concentrator followed by low-intensity magnetic separation resulted in a successful upgrading of ilmenite mineral, Table 5. Silicate minerals were separated from ilmenite using falcon SB40 concentrator. Magnetite, as well as some hematite, was efficiently separated from weakly paramagnetic ilmenite mineral using low intensity magnetic separator. A final product containing 46.31% TiO₂, which represents approximately 86% ilmenite mineral, was obtained, Table 6.

Table 6: Chemical analysis of the final produced products

Product	TiO ₂	Fe ₂ O ₃	SiO ₂	Al ₂ O ₃	Na ₂ O	K ₂ O	CaO	L.O.I
ilmenite feed (-80+25 micron)	26.23	50.08	14.34	4.31	1.76	0.98	0.34	1.94
Falcon concentrate	40.60	54.55	2.43	0.36	0.35	0.21	0.08	1.40
Ilmenite concentrate	46.31	49.87	1.78	0.33	0.19	0.14	--	1.36
Ferromagnetic fraction	14.90	79.80	2.80	0.45	0.23	0.21	--	1.35

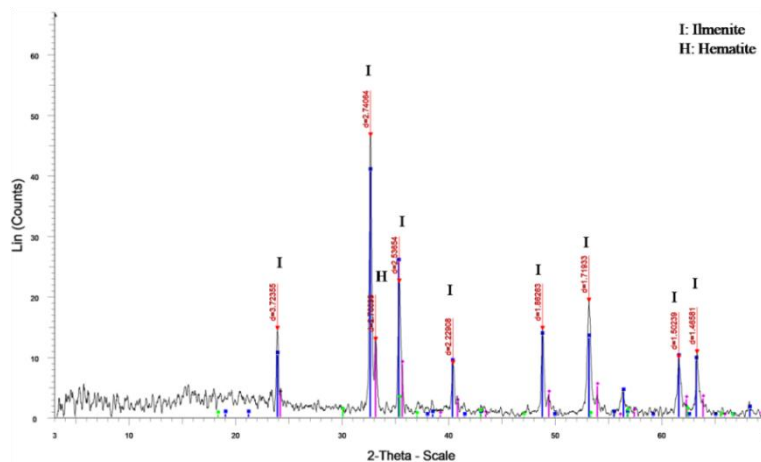


Fig. 8: XRD of paramagnetic ilmenite concentrate

CONCLUSION

The beneficiation of fine low grade ilmenite ore was successfully performed using Falcon SB40 concentrator and Eriez low-intensity magnetic separator. Different size fractions were tested on falcon SB40. Results showed that falcon separation efficiency increased with decreasing size as well as with narrow size fractions while it is limited to size fraction less than 25 microns. An efficient separation was achieved at size fractions -80+45 and -45+25 micron. A Box-Behnken design expert was successfully applied to evaluate the best optimum parameters of the beneficiation processes for ilmenite feed of -80+25 micron. Silicate

minerals were successfully separated from ilmenite as SiO_2 was decreased significantly from 14.34% to 2.43%. The optimal variables of the Box-Behnken design of ilmenite beneficiation were: feed rate (102 g/min), fluidization water (3.60 psi) and centrifugal field (194.4 G's). With these optimum parameters, a concentrate containing 40.61% TiO_2 was obtained with the operational recovery of 85.99%. Applying wet low-intensity magnetic separation on the heavy falcon fraction, magnetite, as well as some hematite, was efficiently separated from weakly paramagnetic ilmenite mineral. The final product, containing 46.31% TiO_2 , which represents approximately 86% ilmenite mineral, coincide the standard specifications required for different industrial applications.

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