Application of Response Surface Methodology on Beneficiation of Sudanese Chromite Ore via Pilot Plant Shaking Table Separator

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Abstract: High grade chromite ore has been decreased constantly due to the importance of chromium element in industrial uses such as metallurgical, chemical, and refractory industries. Therefore, beneficiation of low grade has been more significant .Response Surface Methodology (RSM) is combination of statistical and mathematical methods used for modeling and analyzing problems. In this study, the Central Composite Design (CCD) was applied by using Design-Expert (version 6.0.5) for modeling and optimizing the effect of operating variables on the performance of gravity separation via pilot plant shaking table for chromite ore. Three operating variables were studied, namely feed rate, tilt angle, and flow rate during the tests. The sample under study is low grade chromite ore, containing (30.21%, Cr₂O₃). The mathematical model equations of ANOVA model revealed that the grade of concentrate is more sensetive for feed rate (g/min) compered to water flow rate (l/min).whereas, recovery of concentrate is more sensetive for tilt angle compered to water flow rate (g/min). Optimized responses for the beneficiation process were found at concentrate with 48.52% Cr₂O₃ in with 83.09% recovery and it was achieved at water flow rate 15.33 l/min, tilt angle 2.16 \approx 2.00 degree, and feed rate of 195.38 g/min.

Keywords: Chromite, Mineralogical, Response Surface Methodology (RSM), Central Composite Design (CCD), Pilot Plant Shaking Table, Recovery, Grade, ANOVA Model.

1. Introduction:

Chromite (FeO, Cr_2O_3) is the strategic mineral source for chromium metal, chromium chemicals, refractories, and metallurgical uses[1]. The main use of chromium metal in refractories is the refractory bricks for lining of high temperature furnace, in metallurgy, it is used to produce stainless steels, tool and alloy steels for rod and ball mill media and liners. In chemical industries it is introduced in paint pigments and chemical compounds as an electrolyte in chromium plating baths [2, 3]. Chromite mineral has varied composition based on the chemical formula (Mg, Fe+2) (Cr, Al, Fe+3)₂O₄. Chromium element can be located as chromium spinel, a complex mineral containing magnesium, iron, aluminum and chromium in varying proportions depending upon the deposit. Iron is replaced by magnesium and similarly chromium by ferric iron and aluminum [4, 5]. Low grade chromite ore is usually treated via gravity separation depended on the differences in specific gravity between chromite mineral and the gangue minerals and rocks such as serpentine and olivine, Spiral concentrators and shaking tables have been used widely in low grade chromite ore processing[6]. Multi Gravity Separator (MGS) is centrifugal force separator used to separate the fine particles (500 -10µm) and two minerals closed in specific gravity [7-10]. Various attempts were conducted on development of gravity separation equipment such as shaking table separator[11]. Magnetic separation for chromite mineral is usually performed to improve the Cr/Fe ratio in the concentrate with a Cr: Fe ratio greater than 3:1 for metallurgical uses [12, 13]. In a lower magnetic field intensity (~0.1–0.7 T), chromite mineral can be separated from ferromagnetic minerals (iron-bearing gangue minerals) as the nonmagnetic product [14]. Physiochemical separation is used to treatment the ultrafine chromite ore which is generated from comminution stage , selective flocculation method is always used to separate chromite slimes from the gangue [15, 16].

(RSM) is defined as the combination of statistical and mathematical methods that are useful for modeling and analyzing problems[17]. Recently, the application of Response Surface Methodology (RSM) has been used in mineral processing application for obtaining suitable process variables with optimum results at mineral treatment, various experimental designs are used for different objectives such as central composite design was applied for coal cleaning by varying the process variables of multi

gravity separator[18].However, three-level with three-factor of full factorial experimental design was investigated in different operating parameters on the separation efficiency of Knelson separator[19]. As well as, Box Benhenken experimental design was applied to study the significance of operating parameters on beneficiation of ultrafine chromite particles by selective flocculation [20].

Sudanese chromite ore is considered an important and strategic resource for industrial mineral in Sudan, it occurs in Ingassana Hills in the Blue Nile region and Umm Saqata- Qala Elnahal in Southern Sudan [21]. Extensive work was conducted for processing of low grade Sudanese chromite using a laboratory shaking table separator and dense media separation [3, 22].

This investigated study aims for modeling and optimization of parameters process via Central Composite Design on beneficiation of massive low-grade Sudanese chromite by using pilot plant shaking table as the separator.

2. Materials and Methods:

2.1 Raw material of chromite sample:

A 100 Kg of low-grade chromite ore contained 30.21%, Cr_2O_3 was taken from the stockpiles of mines at Umm Saqata village in Gedarif, Sudan. The sample was mixed thoroughly to be homogenous, then the representative sample was characterized via Microscopic examinations and X-ray diffraction (XRD).

2.2 Analysis methods:

The instrument of X-Ray diffraction (XRD) manufactured by The Analytical X-ray company was used to define phases of minerals using Direct Optical Positioning system (DOPS). For quantitative analysis of minerals oxides, X-ray fluorescence (XRF) was utilized by applied mxios max 4.00kw model.

2.3 Sample preparation:

A representative sample of low-grade massive chromite ore was crushed, ground, then screened to -400 μ m. The ground product was deslimed via sieve size (80 μ m), the final ground product was of (-400+80) μ m particle size.

2.4 Beneficiation procedure:

The central composite design was applied to describe the behavior of relationship between three operating variables. A three factors (feed rate (g/min), tilt angle, and water flow rate (l/min)) and two-level coded (low coded -1 and high coded +1) were used to determine two responses (Grade and recovery) of the produced concentrate.

Feed rate (A), water flow rate (B), and tilt angle (C) were independent variables to predict the responses (grade and recovery) of the pilot plant shaking table separator. The independent variables (A, B, C) with their coded and actual levels are presented in Table 1.

Table 1.Variables and levels for the two-level and three-factor small factorial design

Variables	Symbol	Real Values of G	Real Values of Coded Levels			
			-1	+1		
Feed rate (g/min)	А	100	200			
Water flow rate (l/min)	В	15	20			
Tilt angle (degree)	С	2	4			

Shaking table of a pilot plant scale was used as the separator to beneficiate low grade chromite ore at Central Metallurgical Research and Development institute in Egypt as depicted in Fig.1. Five hundred grams of low-grade chromite sample (-400+80) μ m was used for each experiment. Prior to operation of shaking table, the table was set at required operating conditions for tilt angle and water flow rate l/min, and the then was started. Chromite sample was put into vibration feeder, then continuously fed into the feed box of the table at desired feed rate (g/min), while the vibration feeder was feeding the sample to the shaking table, the sample was separated for heavy minerals (concentrate) and light minerals (tailing). The concentrate and tailing were collected from their collecting pans, then dried and weighed. The concentrate was subjected to (XRF) analysis for chromium oxide quantity evaluation.

The above procedure was repeated for each experiment, then ANOVA for Response Surface Quadratic Model was applied on the results of beneficiation for modeling and optimization the parameters of concentration.

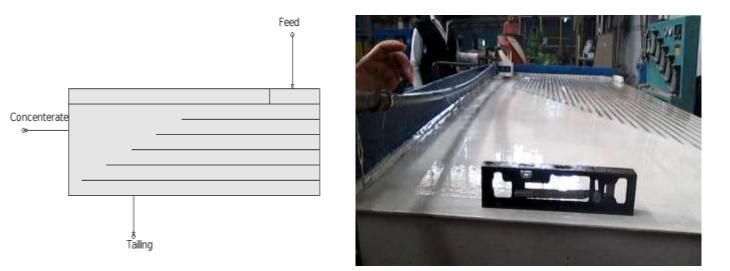


Figure 1.Pilot plant shaking table separator.

3. Results and discussion

3.1 Characterization studies:

The results of microscopic studies of the thin and polish sections made from the chromite ore sample are given in Plat.1. Plat.1 (a) reveals that the serpentine rock filling the cracks and it is surrounded by opaqueness minerals (chromite, magnetite, and hematite). Plat.1 (b) demonstrates that the chromite and magnetite particles appeared in euhedral shape and massive texture. The associated gangue minerals with chromite mineral are mainly silicates minerals.

Figure.1 shows the X-ray diffraction (XRD) pattern of chromite sample, it reveals that the main phases of mineral in the sample of investigated study are chromite (FeCr₂O₃), Talc (Mg₃ (Si₂O₅)₂(OH) ₂), and Magnetite (Fe₃O₄).

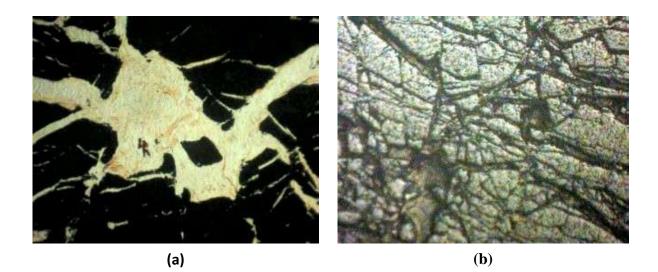


Plate. 1. Microscopic studies result for a) thin section sample and b) polish section sample

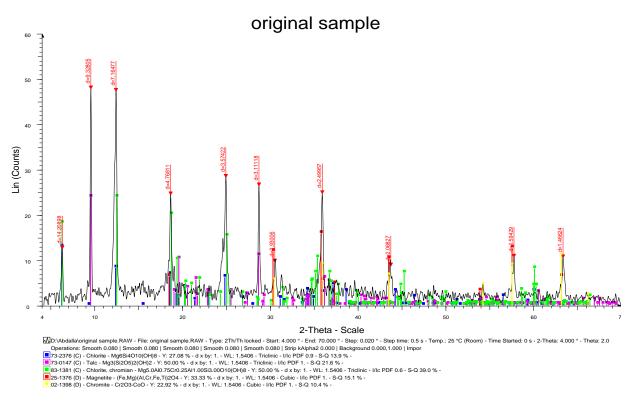


Figure 2.X-ray diffraction (XRD) pattern of chromite sample.

3.2 Statistical analysis:

Twelve experiments were calculated via Central Composite Design (CCD). Table.2 shows the actual factors values and respective values for two responses, it reveals that the recovery and grade values of chromite concentrate distributed in wide range. Table.3 shows the basic statistical analysis of the two responses, grade and recovery.

Table 2.Experimental runs for Central Composite Design with factor values in actual form and respective responses

Experimental Run Number	Factor 1 A: Feed rate	Factor 2 B: water flow rate	Factor 3 C: Title angle	Recovery %	Grade Cr ₂ O ₃ %	
	1	150.00	17.50	4.00	25.68	45.8
	2	150.00	17.50	3.00	67.34	43.94
	3	150.00	17.50	2.00	80.9	47.32
4	Ļ	100.00	20.00	4.00	36.2	47.75
4	5	200.00	17.50	3.00	74.45	40.76
	6	200.00	20.00	2.00	36.47	46.25
	7	150.00	17.50	3.00	73.6	45.48
8	3	100.00	15.00	2.00	50.48	39.81
9		100.00	7.50	3.00	80.72	41.079
1	0	200.00	15.00	4.00	24.23	47.96
1	1	150.00	20.00	3.00	48.46	46.051
1	2	150.00	15.00	3.00	45.61	47.1

Table 3.Statistical Analysis for Responses.

Name	Unit	Туре	Std.Dev.	Low High		
Feed rate	g/min	Factor	. 0	100	200	
Water Flow rate	l/min	Factor	: 0	15	20	
Tilt angle	Dgree	Factor	0	2	4	
Rrecovery						
of Concentrate	%	Respo	onse 3.130	33 24.23	80.9	
Grade (Cr ₂ O ₃)	%	Respo	onse 0.824	525 39.81	47.96	

Fisher's test (F test) with corresponding (P) values were used to study the effect of different parameters on two responses i.e. grade and recovery in the result analysis via ANOVA analysis model.

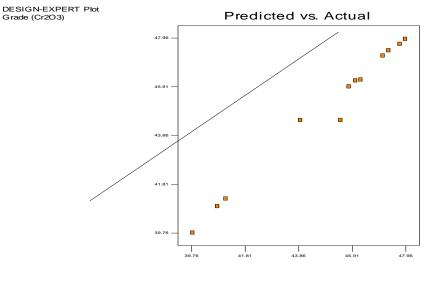
3.2.1 ANOVA analysis for concentrate grade.

Table 4. Shows the ANOVA analysis for grade of concentrate. It demonstrates the effect of factors with their interactions which are A , B,C,A^2,B^2,C^2,AB,AC , and BC where the A,B,and C are feed rate (g/min), water flow rate (l/min), and tilt angle respectively. Values of "Prob > F" less than 0.0500 indicate model terms are significant factors. As such, A^2 and AB are significant factors for this model , this indicates that, the grade of concentrate is more sensetive for feed rate (g/min) compered to water flow rate (l/min).

Table 4. ANOVA	analysis	for grade	of concentrate.
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Source	Sum of Squares	Mean DF	F Square	Value	Prob >	F
Model	89.67	9	9.96		14.65	0.0655
А	0.051 1	0.051	0.075		0.8101	
В	0.55	1	0.55		0.81	0.4633
С	1.16	1	1.16		1.70	0.3222
A ²	30.06	1	30.06		44.21	0.0219
в ²	11.97	1	11.97		17.61	0.0524
C ²	11.80	1	11.80		17.36	0.0530
AB	13.42	1	13.42		19.74	0.0471
AC	5.78	1	5.78		8.50	0.1002
BC	4.43	1	4.43		6.51	0.1254
Residual	1.36	2	0.68			
Lack of Fit	0.17	1	0.17		0.15	0.7672
Pure Error	1.19	1	1.19			
Cor Total	91.02	11				

Fig.3. shows the comparison between actual and predicted grade values. It explains that actual value of grade closed to predicted



X: Actual Y: Predicted

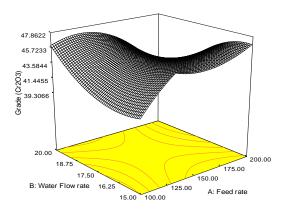
Figure 3. Comparison between actual and predicted grade values.

value of grade

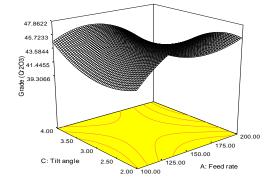
The final equation of this modele for grade of concentate is given in Equation(1) based on Coded Factors. Grade (Cr₂O₃) of concentrate = +44.49-0.16 * A-0.52 * B-0.76* C-3.47* A^2 +2.19* B^2 +2.17* C^2 -3.17* A * B-2.08 * A * C-1.82* B * C Equation(1) Where, A= Feed rate (g/min) B= Water flow rate (l/min) C= Tilt angle

Fig.4 shows the 3D response surface plots for effects of different interactions parameters (feed rate, water flow rate, and tilt angle) on grade of concentrate. Fig.4 (a)1 reveals that the increasing of feed rate with decreasing of water flow rate cases decreasing on

grade of concentrate. Whereas Fig.4 (a)2 explains that the decreasing of feed rate with increasing of tilt angle cases increasing on grade of concentrate. Fig.4 (a)3 demonstrates that the grade of concentrate increases when the tilt angle decreasing with increasing of water flow rate.









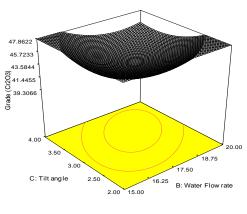




Figure 4. 3D response surface plots for effects of different interactions on grade of concentrate.

3.2.2 ANOVA analysis for chromite recovery in concentrate:

Table 5.shows the ANOVA analysis for the chromite recovery in concentrate, it observes the effect of factors with their interactions which are A, B,C,A²,B²,C²,AB,AC, and BC.where the A,B,and C are Feed rate (g/min), water flow rate (l/min), and tilt angle respectively. Values of "Prob > F" less than 0.0500 indicate model terms are significant factors. Therefor, in this case C, B², C², and AB are significant model terms, this indicates to, the recovery of concentrate is more sensetive for tilt angle compered to water flow rate (l/min) and feed rate (g/min). Fig.5 shows the actual value of recovery with predicted value of grade, it reveals that the actual value of recovery is quite well to predicted value of recovery.

	Sum of	Mean	F			
Source	Squares	DF	Square	Value	Prob > F	
Model	4822.94	9	535.88		54.69	0.0181
А	19.66	1	19.66		2.01	0.2924
В	4.06	1	4.06		0.41	0.5857
С	1524.62	1	1524.62		155.59	0.0064
A ²	124.79	1	124.79		12.73	0.0703
B ²	1378.86	1	1378.86		140.71	0.0070
C ²	742.18	1	742.18		75.74	0.0129
AB	586.88	1	586.88		59.89	0.0163
AC	4.99	1	4.99		0.51	0.5494
BC	15.05	1	15.05		1.54	0.3409
Residual	19.60	2	9.80	1		
Lack of Fit	4.167E-003	1	4.167E-003		2.127E-004	0.9907
Pure Error	19.59	1	19.59			
Cor Total	4842.54	11				

Table 5. ANOVA analysis for the chromite recovery in concentrate.

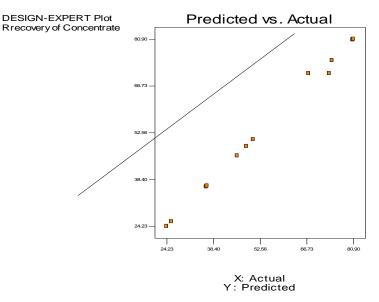


Figure 5.Comparison between actual and predicted recovery values.

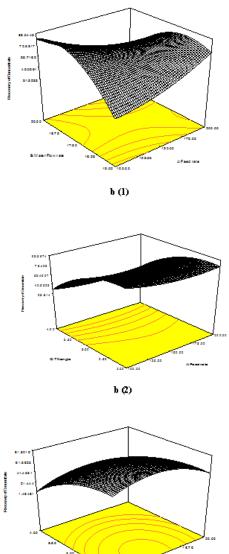
The final equation of this modele for recovery of concentate is given in Equation(2) based on Coded Factors. Recovery of Concentrate = $+70.50 - 3.13* \text{ A} + 1.43* \text{ B} - 27.61* \text{ C} + 7.06* \text{ A}^2 - 23.49* \text{ B}^2 - 17.23* \text{ C}^2$

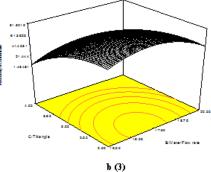
-20.98*A* B +1.93*A* C +3.36*B *C Equation(2)

Where, A= Feed rate (g/min) B= Water flow rate (l/min)

C= Tilt angle

Fig.6 shows the 3D response surface plots for effects of different interactions parameters (feed rate, water flow rate, and tilt angle) on recovery of concentrate.Fig.6 b(1) reveals that the recovery of concentrate increases with decreasing of water flow rate and increasing of feed rate .whereas, the decreasing of tilt angle with increasing of feed rate cases increasing on recovery of concentrate Fig.6 b(2).Fig.6 b(3) explains that the decrasing of water flow rate with decreasing of tilt angle cases increasing on recovery of concentrate.





3.3 Parameters Optimization:

Table 6 and Table 7 show the constraints of parameters and optimized Solutions for optimization the separation process parameters which is considered multi objective by obtaining maximum grade and recovery of concentrate. It reveals that the maximum grade (48.52 Cr₂O₃, (%)) and recovery (83.09%) with high production for chromite concentrate can be obtained at water flow rate 15.33 l/min, tilt angle $2.16 \approx 2.00$ degree, and feed rate195.38 g/min.

Table 6. Constraints for optimization of gravity separation parameters via pilot plant shaking table

Name	Goal	Lower Limit	Upper Limit	
Feed rate	is in range	100	200	
Water Flow rate	is in range	15	20	
Tilt angle	is in range	2	4	
Rrecovery of Concentrate Gade (Cr ₂ O ₃)	maximize maximize	24.23 39.81	80.9 47.96	

Table 7. Optimized Solutions of beneficiation.

Number	Feed rate g/min	Water Flow rate l/min	Tilt angle,Degree	Rrecovery of Concentrate (%)	Grade Cr ₂ O ₃ , (%)
1	114.56	19.49	2.06	83.8335	48.247
2	131.07	18.75	2.00	80.9899	47.9808
3	107.74	19.85	2.07	84.8459	48.6378
4	195.38	15.33	2.16	83.0903	48.5212
5	116.34	19.56	2.07	82.1133	48.5238
6	126.95	19.04	2.07	81.0477	47.9961
7	121.43	19.10	2.01	82.9558	48.0487
8	188.29	15.83	2.01	82.715	48.5688
9	125.78	19.06	2.05	81.3513	48.062
10	163.19	17.22	2.00	80.579	47.6694

4. Conclusion:

High grade chromite ore has been decreased constantly due to the importance of chromium element in industrial uses such as metallurgical, chemical, and refractory industries. Therefore, beneficiation of low grade has been more significant (Put the above statements at abstract). The investigated chromite ore is low grade containing $30.21 \ \% \ Cr_2O_3$. XRD results revealed that the minerals phases of sample were Magnetite and Chromite. Central Composite Design (CCD) of Response Surface Methodology (RSM) was applied for modeling and optimizing the beneficiation process of low-grade chromite ore via pilot plant shaking table. Feed rate, water flow rate, and tilt angle were considered as the operating variables. ANOVA analysis model for recovery and grade reveals that the grade of concentrate is more sensetive for feed rate (g/min) compered to water flow rate (l/min),whereas , the recovery of chromite in concentrate is more sensetive for tilt angle compered to water flow rate (l/min) and feed rate (g/min). Optimized responses for beneficiation process was found to be: 48.52% grade of Cr₂O₃ in concentrate with 83.09% recovery, achieved at water flow rate 15.33 l/min, tilt angle 2.16 \approx 2.00 degree, and feed rate 195.38 g/min.

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