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Optimization of operating parameters of graphite flotation circuit using box-behnken design

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Flotation is a continuous process, therefore conventional tree analysis tests do not fit to determine the quantity and quality of the products. Hence, it is necessary to incorporate flotation circuits. The present study presents optimization of three stage graphite flotation circuits. Response Surface Methodology (RSM) is used to determine the optimum flotation conditions. In addition, the relationship between process variables (diesel oil dosage, methyl isobutyl carbinol dosage and sodium silicate dosage) and the responses (weight, carbon content and recovery) has been investigated. Analysis of variance has been performed to check the suitability and significance of the quadratic models. The results are found to be compatible withproposed models (R^2 >0.97) which lead to obtain 13.48 weight % of clean graphite, 78.91% carbon content and 54.55% carbon recovery fromthree-stage flotation circuit.

Keywords: Box-Behnken design, Flotation circuit, Response Surface Methodology

The global graphite market consists of two main products; flake graphite and amorphous graphite[1,2]. Flake graphite is classified based on the size of the crystal flakes and graded according to their graphitic carbon content and particle size. Microcrystalline graphite is commercially called as amorphous graphite[3,4]. Amorphous graphite is found as fine particles in beds of metamorphic rocks such as coal, slate or shale deposits. Depending on the geological conditions, the graphite content ranges from 25% to 85%[5]. In case of lean and finely disseminated ores, fine grinding is essential to liberate values from gangue minerals[4,6].

Because of its natural hydrophobicity, graphite can be enriched easily by flotation[7-9]. Froth flotation process is widely used, as it results in producing a high-grade graphite concentrate[10,11]. Froth flotation involves knowledge of the three major components (chemistry, equipment and operation mode) of the flotation. Each component has a large number of factors that can be set. As a rough approximation, it is assumed that chemical component factors such as frother, collector, depressants etc. are independent to each other. Klimpel[12] concluded that chemistry component has much greater significance than it is often realized. The types and quantity of the reagents are the most important part of the flotation process. In a commercial plant, the control of reagent additions is the most important aspect of the flotation strategy[13,14]. In graphite flotation, hydrocarbons, e.g., kerosene, fuel oil, paraffin and diesel oil or ionic collectors, e.g., potassium amyl xanthate and dithiophosphate are generally used. Pine oil and methyl isobutyl carbinol (MIBC) are used as frother; sodium silicate, quebracho and starch are used to depress gangue minerals. The optimum *p*H in graphite flotation is between 8 and 9[8,15].

Although flotation experiments performed inlaboratory scaleare generallysingle-stageand called as a rougher flotation, flotation is carried out as a continuous operation in a series or bank of cells[16]. This increases the floating time allows ample opportunity for particle-bubble attachment to occur. The arrangement of a number of cells in series enables the collection of different products from the various cells[17]. Since the desired separation cannot be achieved in a single stage, various coupled stages are used. This is named as "flotation circuit"[18]. Quite often, the grade of concentrate recovered from a single stage of flotation is not high enough and requires re-floating in one or more stages of flotation referred to as cleaner or re-cleaner stages. The series of cells that produce the initial concentrate is called the rougher stage and any subsequent retreatment of the rougher tailings is referred to as scavenging. The scavenger section of the flotation circuit is given higher reagent dosages and long flotation time to float as much valuable mineral as possible to maximize recovery[17].

Results of experiments carried out in laboratory batch flotation are not responsive with those performed infullycontinuoussystems. So, it is necessary to use simulation methods. Closed-circuit flotation experiments are the experiments that simulate the industrial flotation in the laboratory conditions. As these tests take too much time, it has been reported that it could achieve considerable results using mathematical simulations instead of experimental simulation. To apply this, it is necessary to obtain first cycle data (some second cycle data)[16].

The statistical design of experiments has several advantages over the classical method of treating one variable at a time[7,13]. Response Surface Methodology (RSM) is a collection of statistical and mathematical techniques used for developing and optimizing processes which considers the probable interactions between operation parameters[19,20]. One of the common experimental designs used for engineering purposes is a Box-Behnken design that includes three variables and three factorial levels[21,22].

In the present study, three level Box-Behnken design was used for the optimization of some operating variables on the three stage flotation circuit. The aim of the experiments was to obtain high carbon content and carbon recovery of froth products with maximum concentrate weight.

The operating variables were collector dosage (X_1), frother dosage (X_2) and depressant dosage (X_3). The effect of these variables on the weight, the carbon content and the carbonrecovery of the concentrate were studied using Box-Behnken statistical design. In addition, empirical models were developed to correlate the responses with the operating variables. Determination of optimal dosages of variables was also studied. Additionally, effects of operating parameters on performance of graphite flotation were discussed using surface plots.

Experimental Section

Materials

Samples used in the tests were taken from the Kutahya- Altintas (Turkey) graphite ore deposit. The graphite ore exposed to X-ray diffraction studies for mineralogical phase analysis and to identify minerals X-ray diffraction pattern are shown in Fig 1. In the qualitative mineralogical analysis of raw graphite, it was found that the ore sample mainly consists of muscovite ($KAl_2(AlSi_3O_{10}(OH)_2)$), calcite ($CaCO_3$), quartz ($SiO_2$), dolomite ($CaMg(CO_3)_2$) and graphite. According to analysis results, the raw graphite contains 17.42% carbon. Raw graphite carbon was analyzed by ELTRA's CS-2000 carbon analyzer device according to ISO10694 procedure.

For the flotation tests, the sample with d_{80}=85 µm was prepared after a single stage crushing (jaw crusher) and a grinding (ball mill). The particle size distribution of the ground sample is shown in Fig. 2.

Method

Maximization of the carbon recovery of clean graphite concentrate with both maximum weight and carbon content was the major focus for the flotation process. For this purpose, flotation design was planned as three flotation stages namely rougher, scavenger and cleaner units as shown in Fig. 3.

The reagents used in the flotation experiments werediesel oil as collector, MIBC as frother and sodium silicate as depressant. Flotation experiments were performed in a one liter Denver laboratory type flotation machine. During the flotation tests, the solid content and impeller speed of the flotation machine were kept at 10% (by weight) and 1400 rpm at the

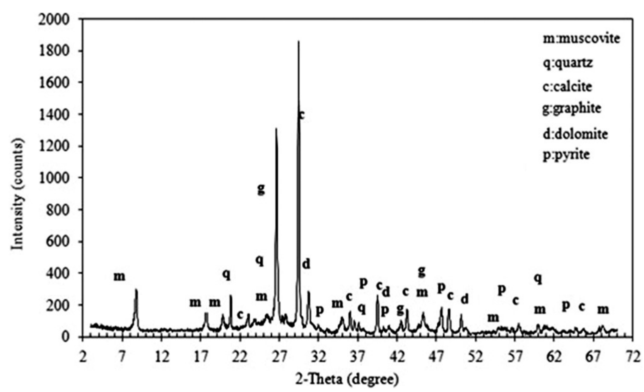


Fig. 1 — X-ray diffraction pattern of raw graphite ore

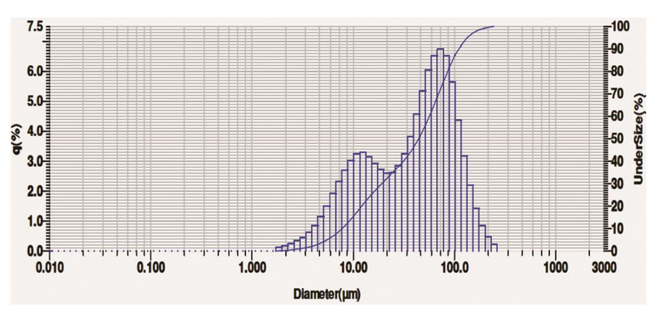


Fig. 2 — Particle size distribution of graphite sample after grinding (d_{80} =85 µm)

natural *p*H (8) respectively. The graphite slurry was conditioned with reagents for three minutes each. Lastly, the air was introduced into the cell and the froth products were collected until the froth formation ended.

Flotation tests were performed in two steps. In the first step, modeling and optimization of reagent dosages for rougher, cleaner and scavenger stages were performed. The main idea of the each flotation stage was to maximize the weight and carbon content in the concentrate with maximum carbon recovery. After the first stage flotation tests, the middling obtained from the cleaner and the scavenger units were mixed with raw graphite ore and fed material was formed for the latter tests. The second cycle flotation tests were performed using the same methodology. The results taken from each cycle flotation tests were compared.

Box-Behnken design

Box-Behnken design method has been employed to study the levels of operating parameters and the interaction between variables affecting the responses on graphite flotation. The variables considered as factors that heavily affect the response functions were collector dosage (X_1), frother dosage (X_2) and depressant dosage (X_3). Percentage of the weight of the concentrate (Y_{wn}), percentage of the carbon content of the concentrate (Y_{cn}) and percentage of the carbon recovery of the concentrate (Y_{rn}) were selected to be response parameters as dependent variables. Reagent dosages used in the flotation tests (the rougher, the cleaner and the scavenger stages) are given in Table 1 with the coded and actual values. The experimental design was consisted of 17 trials for each flotation stage and the independent variables are studied at three different levels as low (−1), medium (0) and high (+1).

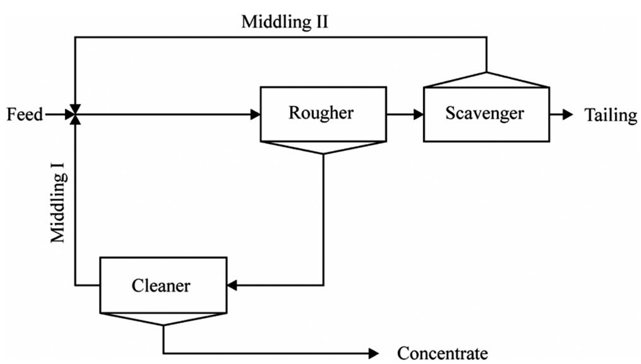


Fig. 3 — Schematic diagram of three-stage flotation circuit

The predicted responses values in each trial of the quadratic model were expressed as:

$$Y_{(w,c,r)n} = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_1^2 + \beta_5 X_2^2 + \beta_6 X_3^2 + \beta_7 X_1 X_2 + \beta_8 X_1 X_3 + \beta_9 X_2 X_3 \quad \ldots (1)$$

In this formula, $Y_{(i,\ j,\ k)\ n}$represents the measured response, β_0—intercept, β_1, β_2, β_3 are the linear coefficients, β_4, β_5, β_6—quadratic coefficients, β_7, β_8, β_9—interactive coefficients, X_1, X_2, X_3—independent variables. The relationship between the experimental results of each independent variable and the responses summarized by polynomial equation was given with the above formula. The results obtained from the experiments were subjected to multiple regression analysis by using Design Expert 8.0.7.1 software package program.Optimal reagent dosages were also determined for each stage with the same software program.

Table 1 — Reagents used for the flotation tests

Coded variables	Variable name	Units	Stage	First cycle tests Low actual	Center actual	High actual	Second cycle tests Low actual	Center actual	High actual
				-1	0	1	-1	0	1
X_1	Diesel oil dosage	(g/t)	Rougher	500	750	1000	400	600	800
			Cleaner	50	100	150	50	75	100
			Scavenger	150	200	250	100	150	200
X_2	MIBC dosage	(g/t)	Rougher	150	200	250	80	120	160
			Cleaner	20	40	60	15	30	45
			Scavenger	60	80	100	40	60	80
X_3	Sodium silicate dosage	(g/t)	Rougher	500	1000	1500	400	800	1200
			Cleaner	100	200	300	80	120	160
			Scavenger	300	400	500	100	200	300

Results and Discussion

Effects of reagents such as collector dosage (diesel oil), frother dosage (MIBC) and depressant dosage (sodium silicate) on weight, total carbon and recovery of the flotation products were studied by RSM. The first and second cycle flotation experiments were performed as rougher, cleaner and scavenger stages as described in Fig 3. Optimal reagent dosages and responses were determined for each stage then the experiments were performed and repeated continuously. Results of the first cycle flotation tests are given in Table 2.

After completing first cycle tests, the middling obtained from the cleaner and scavenger flotation units recycled to the rougher flotation unit. These products mixed with the raw graphite ore and used for the later flotation experiments as shown in Fig 3. Each flotation experiments were carried out under the conditions mentioned before and reagent dosages used in this period were given in Table 1. Results of the second cycle flotation tests are given in Table 3.

The results of the statistical analysis are given in Table 4. Joglekar and May[23] suggested that for a good fit model, R^2 should be at least 0.80. It was found that all values are above this fraction, so the models present high determination R^2 coefficients. The adjusted determination coefficients R^2 were also found to be satisfactory and had confirmed the significance of the models. Both coefficients were very close to 1. The "Pred. R-Squared" values were also in reasonable agreement with the "Adj. R-Squared" values. These values indicated that variability of responses was explained well by the models.

The analysis of variance (ANOVA) was performed to evaluatethe significance of effects for responses. The summarized statistics are given in Table 5. Values of Prop>F less than 0.05 indicate that models are significant. The probability (p-value) of the regression model isless than 0.0001, i.e., there is an important multiple regression relationship between the independent variables and the dependent variable. It is expected to get the huge F values in ANOVA. The P value corresponding to the F statistic value is used to decide whether a significant amount of variance. F-values of the responses imply that the models are significant. The Lack of fit f-values of the models show that the lack of fits are not significant relative to the pure error.

The aim of optimization was to maximize the all responses with minimum quantities of reagents. The details are given in Table 6. The results obtained from each step were used for the other steps. All reagents

Table 2 — Experimental matrix and results of the observed responses for the first step flotation tests

Test No	Coded variables			Rougher			Cleaner			Scavenger		
	Diesel oil dosage (X_1)	MIBC dosage (X_2)	Sodium silicate dosage(X_3)	Con. weight	Carbon content	Recovery	Con. weight	Carbon content	Recovery	Con. weight	Carbon content	Recovery
	(g/t)	(g/t)	(g/t)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
1	0	0	0	19.92	60.90	69.64	47.69	79.10	63.67	12.48	21.83	41.46
2	-1	0	-1	21.15	51.92	63.04	40.06	79.55	53.79	12.37	21.64	40.73
3	0	-1	1	18.78	61.02	65.78	36.31	81.22	49.78	9.78	24.96	37.15
4	1	0	1	23.67	49.69	67.52	44.38	79.49	59.55	13.78	20.91	43.75
5	0	0	0	19.88	60.93	69.53	47.13	79.60	63.32	12.48	21.83	41.46
6	-1	-1	0	16.75	63.03	60.61	35.14	81.57	48.38	9.11	25.52	35.38
7	1	-1	0	19.78	57.33	65.10	42.67	79.32	57.13	11.12	23.19	39.24
8	0	0	0	19.67	60.98	68.86	47.78	79.36	64.00	12.78	21.42	41.66
9	-1	0	1	21.52	52.88	65.33	38.21	80.65	52.02	10.92	24.11	39.97
10	0	1	1	21.82	52.86	66.21	54.18	74.02	67.69	13.42	19.89	40.62
11	0	0	0	19.87	60.78	69.33	47.85	79.10	63.89	12.92	21.67	42.60
12	0	1	-1	22.11	54.87	69.64	56.46	74.55	71.05	14.67	18.22	40.67
13	1	1	0	21.85	53.45	67.04	59.43	74.55	74.78	15.65	17.78	42.34
14	1	0	-1	22.72	51.96	67.77	48.34	80.22	65.46	13.97	19.58	41.62
15	-1	1	0	20.54	53.52	63.11	54.67	73.77	68.08	13.78	20.91	43.85
16	0	-1	-1	18.34	59.89	63.05	38.34	80.11	51.84	10.12	23.02	35.45
17	0	0	0	19.50	61.49	68.83	47.25	79.28	63.23	12.59	21.66	41.50

Table 3 — Experimental matrix and results of the observed responses for the second step flotation tests

Test No	Coded variables			Rougher			Cleaner			Scavenger		
	Diesel oil dosage (X_1)	MIBC dosage (X_2)	Sodium silicate dosage(X_3)	Con. weight	Carbon content	Recovery	Con. weight	Carbon content	Recovery	Con. weight	Carbon content	Recovery
	(g/t)	(g/t)	(g/t)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
1	0	0	0	26.98	50.22	69.48	45.76	79.78	60.08	14.23	21.28	52.68
2	-1	0	-1	21.70	63.45	70.61	37.45	82.11	50.32	9.82	26.12	44.62
3	0	-1	1	26.12	53.89	72.18	60.89	76.03	75.76	15.98	19.89	55.30
4	1	0	1	24.74	61.58	78.13	48.21	80.15	63.23	12.89	22.34	50.10
5	0	0	0	26.41	53.31	72.20	39.67	81.08	52.64	10.92	24.11	45.80
6	-1	-1	0	23.56	58.94	71.21	42.67	79.78	55.71	12.34	24.45	52.49
7	1	-1	0	24.27	54.31	67.60	55.90	74.56	68.21	14.67	22.39	57.14
8	0	0	0	24.62	61.96	78.23	49.12	80.44	64.58	13.34	21.98	51.01
9	-1	0	1	24.42	61.66	77.22	47.98	80.56	63.25	13.98	21.73	52.85
10	0	1	1	23.89	52.45	64.26	41.06	80.58	54.14	13.07	21.62	49.16
11	0	0	0	26.53	52.31	71.17	48.12	81.12	63.88	14.45	20.34	51.13
12	0	1	-1	23.14	61.54	73.03	38.12	81.34	50.74	10.34	23.34	41.99
13	1	1	0	22.12	55.12	62.53	57.89	75.94	71.94	15.10	18.83	49.47
14	1	0	-1	26.34	53.17	71.82	55.31	75.13	68.00	13.90	20.32	49.14
15	-1	1	0	24.79	61.83	78.60	48.45	80.26	63.63	13.18	22.24	51.00
16	0	-1	-1	24.36	60.98	77.24	48.76	80.14	63.95	13.45	22.15	51.83
17	0	0	0	22.06	61.09	69.11	38.34	81.42	51.08	10.14	24.56	43.33

Table 4 — Model Summary for the flotation tests

		Rougher			Cleaner			Scavenger		
		Con. Weight	Carbon content	Recovery	Con. weight	Carbon content	Recovery	Con. weight	Carbon content	Recovery
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
First Cycle Tests	Std. Deviation	0.260	0.41	0.61	0.43	0.21	0.64	0.21	0.13	0.62
	R Squared	0.990	0.996	0.979	0.998	0.997	0.997	0.994	0.998	0.973
	Adjusted R-Squared	0.978	0.991	0.952	0.996	0.994	0.993	0.985	0.996	0.939
	Predicted R-Squared	0.884	0.953	0.730	0.982	0.978	0.959	0.941	0.995	0.709
Second Cycle Tests	Std. Deviation	0.300	0.400	0.920	0.550	0.150	0.740	0.350	0.210	0.980
	R Squared	0.986	0.997	0.984	0.997	0.998	0.996	0.984	0.994	0.974
	Adjusted R-Squared	0.967	0.992	0.963	0.994	0.996	0.991	0.962	0.987	0.941
	Predicted R-Squared	0.818	0.971	0.801	0.973	0.994	0.953	0.916	0.968	0.823

dosages were reduced in second cycle flotation tests. While the weight of the concentrate was 20.57% in the first cycle flotation tests, that was increased to 24.98% in the second cycle flotation tests.

The total carbon content of the raw graphite ore was 17.42%. In the first cycle flotation tests, clean concentrate of 10.71 weight was obtained with 78.71% total carbon and 48.09% recovery. After first cycle flotation experiments, middling obtained from cleaner (9.86% by weight and 38.79% by total carbon) and scavenger stage (10.45% by weight and 21.26% by total carbon) were recycled to rougher flotation unit. In this case, total carbon content of the feed material in the rougher stage was increased to 19.50%. Finally, clean concentrate was obtained with 13.48% weight and 78.91% total carbon. The best performance on working conditions was obtained with 86.42 g/t of diesel oil, 36.49 g/t of MIBC and 109.26 g/t of depressant dosages. Comparing the results of both cycle tests, the weight of the clean concentrate could be obtained 25.81% higher than the first cycle tests. It was also observed an increment on the total carbon and the recovery. The summary of expected results for each stage is presented in Table 7.

Regression models for every response were used for simulation and optimization of the second cycle flotation tests. Rates of the weight, the carbon content and the carbon recovery of the concentrates were

Table 5 — The summary of analysis of variance

Source	First cycle tests						Second cycle tests					
	Rougher		Cleaner		Scavenger		Rougher		Cleaner		Scavenger	
	F Value	Prob > F	F Value	Prob > F	F Value	Prob > F	F Value	Prob > F	F Value	Prob > F	F Value	Prob > F
Weight of the concentrate												
Model	79.78	< 0.0001	493.60	< 0.0001	120.67	< 0.0001	53.06	< 0.0001	300.71	< 0.0001	46.40	< 0.0001
Lack of Fit	3.47	0.1302	2.73	0.1786	1.50	0.3433	4.34	0.0952	2.18	0.2330	0.43	0.7407
Carbon content of the concentrate												
Model	201.84	< 0.0001	272.60	< 0.0001	443.11	< 0.0001	225.65	< 0.0001	451.07	< 0.0001	134.83	< 0.0001
Lack of Fit	3.91	0.1105	1.00	0.4786	0.15	0.9275	1.25	0.4028	0.21	0.8828	0.53	0.6850
Recovery of the concentrate												
Model	36.33	< 0.0001	259.65	< 0.0001	28.21	< 0.0001	47.03	< 0.0001	189.52	< 0.0001	29.48	< 0.0001
Lack of Fit	4.84	0.0808	6.94	0.0460	2.39	0.2098	3.75	0.1172	2.76	0.1757	0.78	0.5630

Table 6 — Optimization results for each stage

	First cycle tests			Second cycle tests		
	Rougher	Cleaner	Scavenger	Rougher	Cleaner	Scavenger
Variables						
Diesel oil dosage (g/t)	833.69	125.51	226.37	644.96	86.42	178.09
MIBC dosage (g/t)	213.79	46.59	79.89	121.16	36.49	57.80
Sodiumsilicatedosage (g/t)	893.26	170.81	436.27	867.68	109.26	211.58
Responses						
Weight of the concentrate (%)	20.57	52.08	13.16	24.98	53.97	13.82
Carbon content of the concentrate (%)	59.32	78.18	21.27	60.79	78.91	22.00
Recovery of the concentrate (%)	69.84	68.67	42.55	77.96	69.59	52.91

Table 7 — Metallurgical balance for the flotation tests

	First cycle tests			Second cycle tests		
	Weight%	Total carbon %	Recovery%	Weight%	Total carbon %	Recovery%
Concentrate	10.71	78.18	48.08	13.48	78.91	54.55
Middling1	9.86	38.79	21.95	11.49	39.56	23.33
Middling2	10.45	21.26	12.76	10.37	22.00	11.70
Tailing	68.98	4.35	17.21	64.66	3.14	10.42
Total	100.00	17.42	99.99	100.00	19.50	100.00

taken as dependent variables; independent variables (factors) and their values and second-order polynomial regression models were estimated. It can be possible to find the levels of parameters to maximize the responses of the rougher, the cleaner and the scavenger stages. When error probability (α) is taken as 0.05, the results can be trusted with 95% confidence, i.e., these are statistically significant models. According to the results of regression analysis with Box-Behnken polynomial of second degree for the weight (Y_{w1}), the carbon content (Y_{c1}) and the carbon recovery of the rougher concentrate (Y_{r1}) are as follows:

$$Y_{w1}(\%)=24.59+ 0.87X_1 + 1.05X_2 + 0.76X_3-0.52 X_1X_3 +1.33 X_2X_3 + 0.93 X_1^2 -1.61X_2^2+0.43 X_3^2 \quad ...(2)$$

$$Y_{c1} (\%) = 61.60 - 1.02 X_1 - 3.57 X_2 -0.45 X3 + 1.02X_1 X_2 - 0.74 X_1 X_3 - 4.81 X_1^2 + 0.85 X_2^2 - 4.72 X_3^2 \quad ...(3)$$

$$Y_{r1} (\%) = 77.88 + 1.17 X_1 - 1.23 X_2 + 1.45 X_3 - 2.41X_1 X_3 + 3.30 X_2 X_3 - 3.66 X_1^2 - 3.82 X_2^2 - 4.94 X_3^2 \quad ...(4)$$

Positive sign in front of the terms describes that, with an increasing dependent variable, the response increases and vice versa. Taking into account the coefficients for each term, it can be seen that the greatest positive effect on the weight of the concentrate can be attributed to linear effect of multiplication of frother dosage and depressant dosage (X_2X_3). Next most significant effects are frother dosage (X_2) and collector dosage (X_3) respectively. Both variables also show linear effect. With the same manner, the most effects for the carbon content and the recovery of the rougher concentrate were linear interactive effect of X_1 X_2 and X_2 X_3 respectively.

The product fed to cleaner unit was calculated as 24.98% weight and 60.79% total carbon content. The quadratic models of the weight (Y_{w2}), the carbon content (Y_{c2}) and the carbon recovery (Y_{r2}) of the concentrate obtained from cleaner unit are presented in the following:

$$Y_{w2}\ (\%) = 48.50 + 2.92\ X_1 + 9.18\ X_2 - 0.76\ X_3 - 0.70\ X_2X_3 - 1.52\ X_1^2 + 2.24\ X_2^2 - 3.33\ X_3^2 \quad ...(5)$$

$$Y_{c2}(\%) = 80.31 - 0.20\ X_1 - 2.87\ X_2 - 0.20\ X_3 + 0.95\ X_1X_2 - 0.46\ X_1X_3 - 0.22X_2\ X_3 - 2.19\ X_2^2 + 0.33\ X_3^2 \quad ...(6)$$

$$Y_{r2}(\%) = 63.73 + 3.77X_1 + 9.51\ X_2 - 1.11\ X_3 - 1.07\ X_2X_3 - 1.99\ X_1^2 - 4.05\ X_3^2 \quad ...(7)$$

The frother dosage is the most effective parameter for the weight and the recovery. In the cleaner unit, the middling contained 11.19% weight and 39.56% total carbon. The feed to scavenger unit was 75.02% weight and 5.75% total carbon. The second degree polynomial orders for the weight (Y_{w3}), total carbon content (Y_{c3}) and the total carbon recovery (Y_{r3}) of the middling obtained from the scavenger unit are as follows:

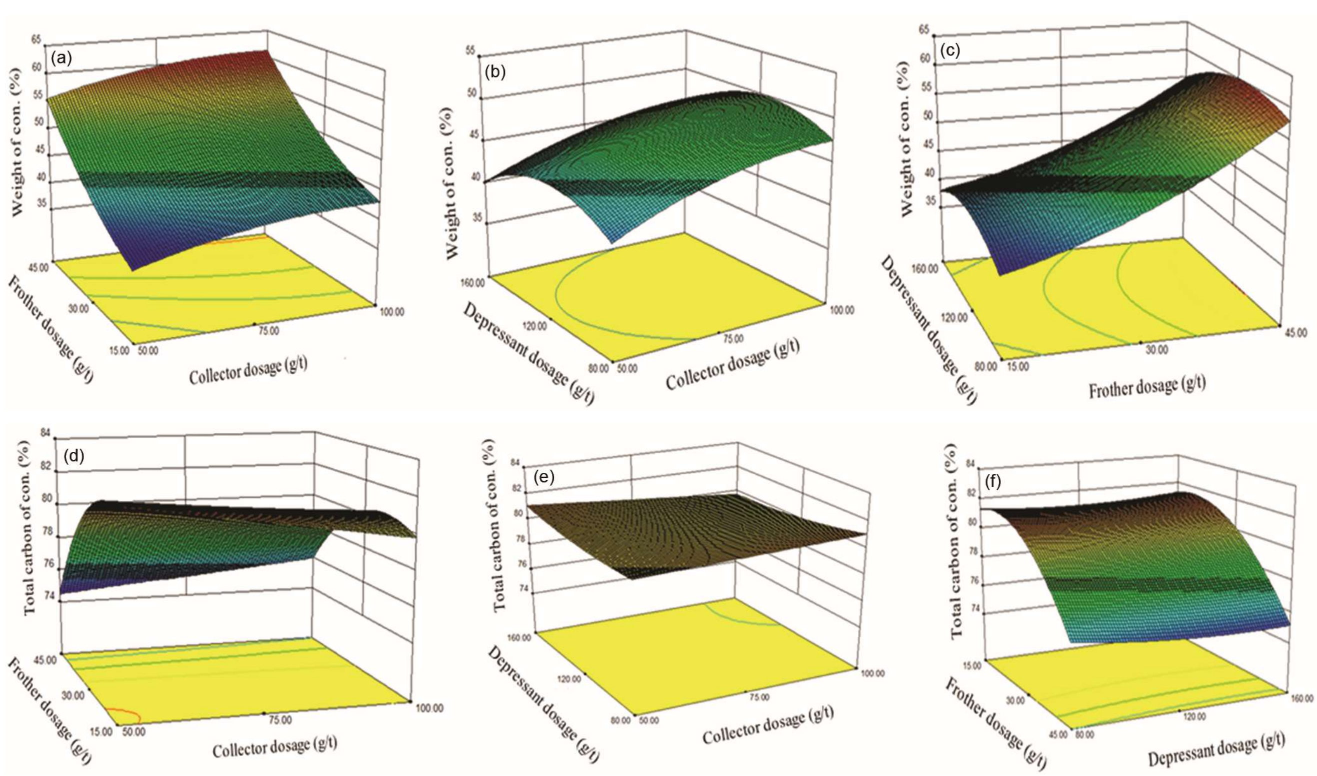
$$Y_{w3}\ (\%) = 13.37 + 1.07\ X_1 + 2.13\ X_2 - 0.47\ X_3 + 0.48\ X_1X_3 - 0.48\ X_2^2 - 0.52\ X_3^2 \quad ...(8)$$

$$Y_{c3}(\% = 22.09 - 1.04\ X_1 - 2.13\ X_2 + 0.77\ X_3 + 0.68\ X_1X_3 + 0.60\ X_1^2 + 0.52\ X_2^2 - 0.85\ X_3^2 \quad ...(9)$$

$$Y_{r3}\ (\%) = 51.36 + 1.86\ X_1 + 3.58\ X_2 - 2.43X_1X_2 + 1.23\ X_1X_3 + 2.37X_1 - 1.34\ X_2^2 - 4.04\ X_3^2 \quad ...(10)$$

It can be seen from the formulas (8)-(10) that the greatest effect on the weight of the middling can be attributed to linear effect of frother dosage. Collector dosage is the next most significant effect. The final tailing obtained from this unit contains 3.14% total carbon.

Using the quadratic models, three-dimensional (3D) plots were generated for weight, total carbon and recovery of clean concentrate (Fig. 4) to examine the



(*Contd.*)

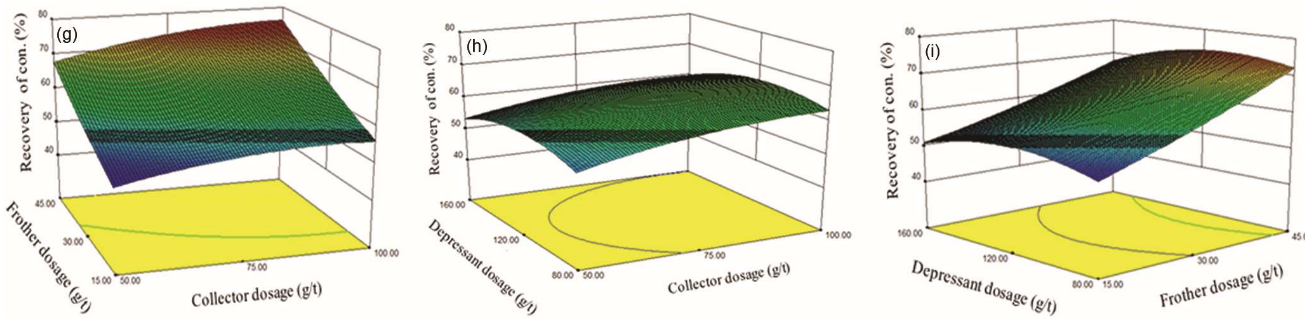


Fig. 4 —3D response surface plots for interactive effects of the independent variables

relative effect of the three factors and their interactions on the responses. Figure 4a shows that the weight of concentrate is obtained with the lowest level in the minimum frother and collector dosages. The weight of the concentrate reaches to a maximum value at around the midpoints of the both depressant and collector dosages and decreases beyond these points (Fig 4b.) Nearly the same trend can be seen in Fig.4c. The impact of the independent variables on total carbon shows different trends with respect to the region of maximum total carbon. Analyzing the trends from Fig. 4d, the highest total carbon comes to near the lowest frother and collector dosages. The total carbon linearly goes up from about 77% to about 81% as the sodium silicate dosage increases from 80 to 160 g/t. Similarly, the total carbon shows an increase with increasing collector dosage (Fig. 4e). Figure 4f shows the influence of depressant and frother dosages on the recovery. While depressant dosage slightly affects the total carbon, the higher frother dosage is dramatically better for total carbon. As it can be seen from Fig. 4g, maximum recovery is obtained at the highest f rother and collector dosages.However, the maximum recovery attains its highest level at the midpoints of depressant and collector dosages. Both high and low level of depressant dosage reduce recovery as expected (Fig. 4h and Fig 4i).

Conclusion

A Box- Behnken design is applied for modeling and optimization of flotation reagents of the graphite flotation. The regression models have been developed to quantify the effect of collector, frother and depressant to predict weight, total carbon and recovery of the products at different reagent dosages.

In the first step flotation tests, the rougher concentrate is obtained with 20.57% weight, 59.32% total carbon and 69.84% recovery at diesel oil 833.69 g/t, methyl isobutyl carbinol 213.79 g/t and sodium silicate 893.26 g/t. The clean graphite concentrate with 10.71% weight, 78.18% total carbon and 48.08 % recovery is calculated with diesel oil 125.51 g/t, methyl isobutyl carbinol 46.59 g/t and sodium silicate 170.81 g/t from the raw graphite ore having carbon content of 17.42%.

After three stage flotation circuit, middling obtained from cleaner (38.79% total carbon and 9.86% weight) and scavenger stages (21.26% total carbon content and 10.45% weight) are recycled to rougher flotation unit. In this case, total carbon content of the feed material in the rougher stage is increased to 19.50%.The rougher graphite concentrate of 24.98% weight, 60.79% total carbon with 77.96% recovery is calculated at diesel oil 644.96 g/t, MIBC 121.16 g/t and sodium silicate 867.68 g/t. It is possible to get clean graphite concentrate weight of 13.48%, total carbon of 78.91% with recovery of 54.55%. It could be possible to obtain clean graphite concentrates 25.58% higher than that of first cycle flotation tests.

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