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Simultaneous Ash and Sulphur Removal from Bitumen Using Column Flotation Technique: Experiments, RSM Modeling and Optimization

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An efficient method was employed for the ash and pyrite sulphur removal from bitumen using column flotation process. The bitumen samples containing 9.6% sulphur (6.81% in the pyrite sulphur form) and 26.4% ash were successfully showed up to 75.20% of pyrite sulphur (*e.g.*; 51.66% of total sulphur) and 70.81% of ash removal. The Box-Behnken Design (BBD) was employed to design the experiments, to optimize and evaluate the individual and interactive effects of six effective independent factors: the amounts of collector and frother agents, solid weight percentage in the pulp, particle size, wash water rate and feed rate. In order to investigate system a laboratory scale rig was built. In all experiments, pine oil and kerosene were considered as frother and collector agents, respectively. The best result for column flotation process were amount of collector (1.5 $kg/t_{bitumen}$), amount of frother (0.3 ppm), pulp content (10% of solid), particle size (100 mesh), wash water rate (0.4 $l\ min^{-1}$), feed rate (1.5 $l\ min^{-1}$) and flotation time (10 min). The coefficient of determination, R^2, showed that the RSM model can specify the variations with the accuracy of 0.924 and 0.938 for the percentage of ash and pyrite sulphur removal from bitumen, respectively.

Keywords: Ash removal, Pyrite sulphur removal, Bitumen, Column flotation process, Response surface methodology

INTRODUCTION

The high price of oil and environmental pollution of fossil fuels has led many countries around the world to look for alternative energy resources. Non-exploited energy sources such as biomass, oil shale, tar sand and bitumen are regarded as potential alternatives for fossil fuels. Although these alternative sources exist in abundant quantities, they are always associated with high risk factors related to technical and economical feasibilities [1]. There are rich mines of bitumen in Canada, Venezuela, Russia, Australia, and Iran [2]. Iran possesses some of the richest bitumen mines and is a major exporter of bitumen to India, China and many other countries in the Middle East. Iranian bitumen called Gilsonite is found in the Kermanshah, Khuzestan, Ilam provinces and other west and southwest regions. Bitumen extracted in these regions is used in many industries, including road construction, polish, and the printing inks and paint [3]. Iran bitumen has some drawback; the most important of which are high contents of sulphur and mineral impurities. Among all the elements in bitumen, sulphur has the most effect to limit the bitumen utilization as a clean fuel [4]. The sulphur content may be found as the organic, pyrite and/or inorganic sulfate. Generally, more than half of the sulphur in bitumen is in the pyrite form [5]. Bitumen also contains inorganic minerals, which are commonly called ash. The main minerals in coal are silicates or shales (kaolinite type), quartz and sandstone, pyrite and siderite carbonates and anchorite [6].

There are many methods for reduction of the ash and sulphur content of bitumen. Among them, the flotation techniques are widely used [7-9]. Flotation is a separation process that depends on the difference in the surface properties of substances. Column floatation is a process

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utilized to selectively separate hydrophobic minerals suspended in a solution by attaching them to air bubbles and transferring them into froth layers. This is attained by using surfactants and wetting agents. It is considered the cheapest and most widely used method for separation of valuable minerals [1].

While Iran has an outstanding volume of bitumen mines, particularly in Kermanshah province, is lacking the processing technology of this valuable mineral. Consequently, decreasing ash and sulphur with a novel and improved method is inevitable in this area. In this study, we aimed to suggest an efficient process to remove ash and pyrite sulphur from bitumen using column flotation process. To do so, we used the box-behnken design (BBD) of response surface methodology (RSM) for analysis and optimization of operational conditions. It should be noted that using RSM to optimize and evaluate interactive effects between variables for ash and sulphur removal from bitumen is a novel method.

MATERIALS AND METHODS

Materials and Analytical Tests

The bitumen samples were supplied by mines, in Kermanshah/(Iran). Pine oil as frother was supplied by Boyakhsaz Company/(Iran) in liquid form. Kerosene as collector was purchased from the National Iranian Oil Products Distribution Company (NIOPDC)/(Iran). Nitric acids with purity of 65% and hydrochloric acid (HCl) with 37% volumetric purity were provided from the Merck Company/(Germany). Desiccators (WG Dry model Box-503, Merck), and atomic absorption (GBC-932, GBC Australia) were used to prepare and analyze the sample and final product.

Experimental Apparatus

The novel apparatus was designed and built to study ash and pyrite sulphur removal from bitumen. As shown schematically in Fig. 1, a 10 cm-diameter flotation column of 2.5 m height made from Plexiglas material was employed in this study. A steel framework was used to keep the stability of the column. A polyethylene vessel with volume of 20 l equipped with an electric mixer was designed and used to provide initial load. Pulp feeder point was placed at 65 cm below the top of column. Pulp supplied was pumped into the column through a peristaltic pump (IP 55, WATSON-MARLOW, UK) with maximum power of 2.5 l min^{-1}. Wash water was inserted through a shower for washing foams in the column and separation of undesirable material from bubble-particle which was applied. Air was supplied through an internal sparger with 20 cm in diameter and 25 cm in height, located at the bottom of the column. Tailings tube of the column was passed from the bottom of sparger.

Experimental Procedures

The particle sizes of less than 250 μm were prepared by crushing the bitumen samples using filter. The pulp 5% was prepared using 50 g of a prepared bitumen sample (with specified characteristics). Then, it was washed using 500 ml water at 30 °C and decanted into a vessel. The paste mixture was rested for 1 h. The volume was reached to 1 l by adding distilled water to the vessel. This mixture was stirred for 3 min. The collector was added to the vessel and the mixture was further stirred for 3 min. The foaming agent was added and mixed for 3 min. The mixture was transported to the column through a peristaltic pump and the process was started. The flotation time was fixed at 10 min when the foam was collected on the top of the column (froth zone). The concentrate was washed with wash water with a flow rate of 0.3 l min^{-1} for 5 min to wash the hydrophilic impurities along with air bubbles. Consequently, the concentrate was dried in the oven for 1 h at 110 °C and pyrite sulphur, total sulphur and ash contents of the dried samples were determined by the method published in our previous work [10].

Design of Experiments

The software Design Expert 7 was applied for the experimental design, statistical analysis of data, development of regression models and optimization of process conditions. The response surface methodology (RSM) was used for fitting a quadratic surface and to analyze the interactions among the parameters. The ash and pyrite sulphur removal were selected as the studied responses and the amount of collector and frother agents, solid weight percentage in the pulp, particle size, wash water rate and feed rate were chosen as the studied factors.

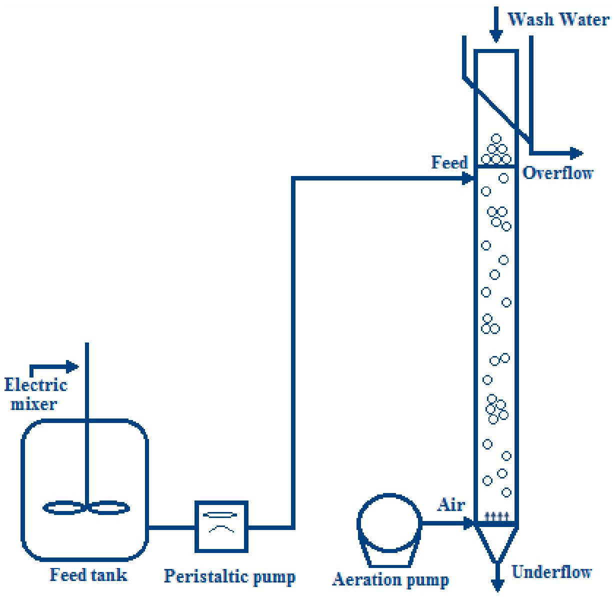

Fig. 1. A schematic illustration of the column flotation apparatus.

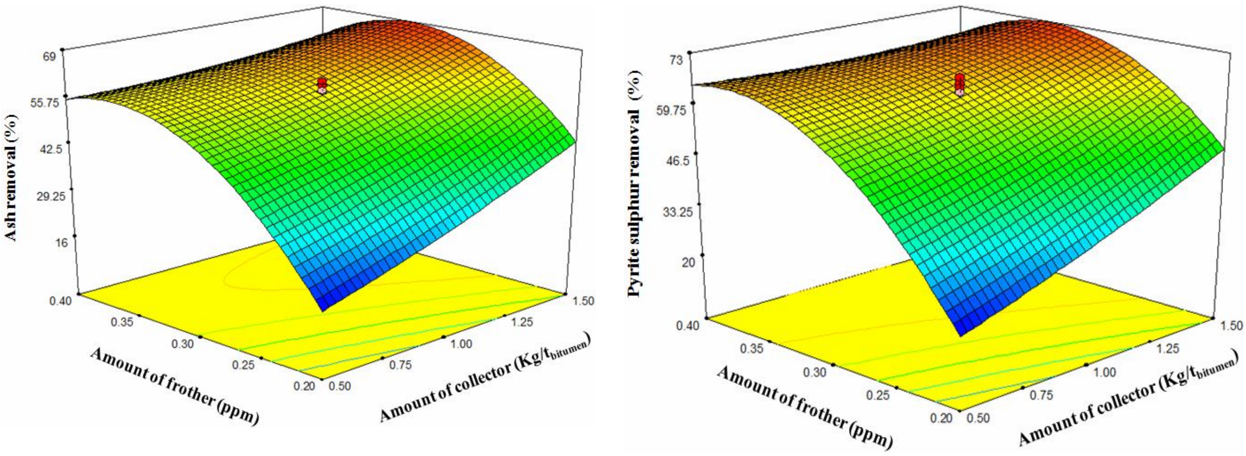

Fig. 2. Ash and pyrite sulphur removal as a function of amount of collector and frother agents ($X_3 = 10\%$, $X_4 = 200$ mesh, $X_5 = 0.3\ \mathrm{l\ min^{-1}}$ and $X_6 = 1.5\ \mathrm{l\ min^{-1}}$).

All six factors were scrutinized each at three levels. The most appropriate design to conduct such a 6-factor-3- level set of experiments was the 54-trial set of box-Behnken design (BBD) combined with RSM (Table 1) [11]. The detailed processing conditions are summarized in Table 2. The fitted model was evaluated for each response function and significance of response variable (*i.e.* percentage of ash and pyrite sulphur removal) was analyzed statistically applying analysis of variance (ANOVA).

The following second order polynomial equation [Eq. (1)] was utilized to predict the chosen responses as a function of independent variables and their interactions [11]:

Table 1. Independent Variables and their Levels for the Box-behnken Design Used in the Present Study

Independent variables	Unit	Symbols	Level of factors		
			-1	0	1
Amount of collector	$Kg/t_{bitumen}$	X_1	0.5	1	1.5
Amount of frother	ppm	X_2	0.2	0.3	0.4
Solid weight percentage in pulp	%	X_3	5	10	15
Particle size	mesh	X_4	100	200	300
Wash water rate	$l\,min^{-1}$	X_5	0.2	0.3	0.4
Feed rate	$l\,min^{-1}$	X_6	1	1.5	2

$$y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X_j^2 + \sum_{i<j}^{k} \sum_{j=2}^{k} \beta_{ij} X_i X_j \qquad (1)$$

In this equation y is the predicted response variable, β_0, β_j, β_{jj}, and β_{ij} are regression coefficients for intercept, linear, quadratic and interaction terms, respectively, and X_i, and X_j are the independent variables.

RESULTS AND DISCUSSION

Effects of Process Parameters on the Responses

Effects of amount of collector and frother agents. Figure 2 implies that the amount of collector has a positive effect on both the ash and pyrite sulphur removal from bitumen. These results confirm very well the findings reported by (Naik *et. al.,* 2005 and Jena *et. al.,* 2008) [12, 13]. This is because the absorption of collector on bitumen increases the hydrophobicity and consequently the floating property of particles. In the other words, the amount of pulp that goes to the froth zone increases because the contact angle of bitumen particles with water improves [14].

The results show that frother agent had the most significant effect on both the ash and pyrite sulphur removal from bitumen (β_2 = 13.82 for ash removal and β_2 = 15.09 for pyrite sulphur removal). This result confirms the findings reported by (Naik *et. al.,* 2005) [12]. Figure 2 shows that the removal yields increase significantly with increasing the frother to 0.3 ppm, whereas, further increase above 0.3 ppm decrease the removal results. This result is consistent with the findings reported by (Angadi *et. al.,* 2011) [14]. Increasing the frother to 0.3 ppm increases the air bubbles, which stabilize the foam on the froth zone. Further increasing the frother rise of the ash and sulphur in the froth zone because mineral matter have very little time for hydrophilic and enter the tailings part and stick to the bubbles and come with them to the froth zone [15,16]. On the other hand, increasing in frother can cause an increase in the hydrophobicity of ash particles because of the interaction between hydrophilic part of pine oil (frother) and the hydrated mineral matter, which later improves the recovery process. Adding pine oil diminishes the surface tension at the liquid-vapor interface, consequently generating a finer bubble size distribution, which leads to increase the flotation rates and recovery values. The role of frothers in flotation is to produce smaller air bubbles.

Effects of solid weight percentage in pulp and particle size. As observed from Fig. 3, percentage of solid weight has negative effect on the ash and pyrite sulphur removal from bitumen. A similar conclusion was reported by (Tao *et. al.,* 2000) [17]. Increasing solid weight percentages in pulp causes the decline of the contact angle of the bitumen particles and the air bubbles and also reduces sticking time. Therefore, the possibility of connection of the particles and the air bubbles decreases. Any decrease in the connection between the particles and the air bubbles can reduce percentage of the ash and pyrite sulphur removal from bitumen. Besides, flotation rate decreases with an increase in solid weight percentage in the pulp resulting in

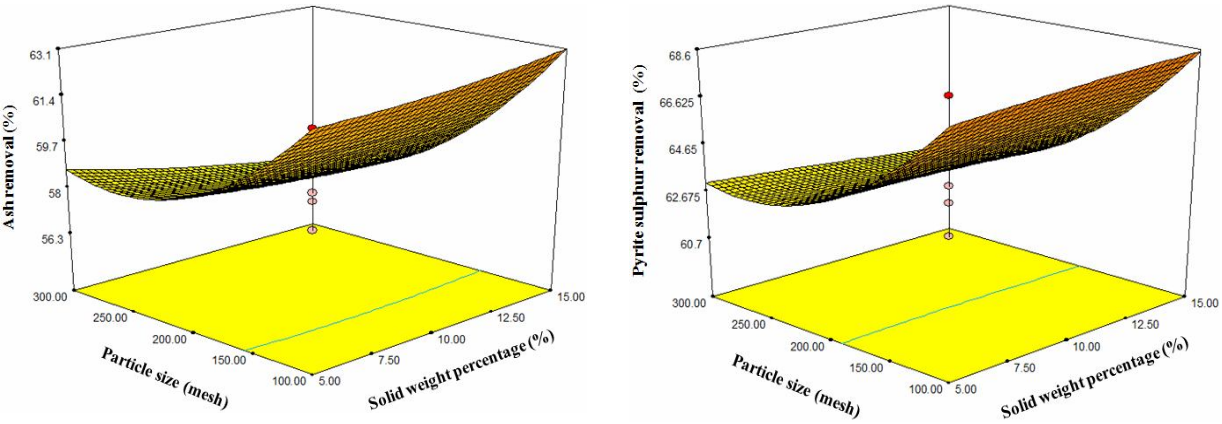

Fig. 3. Ash and pyrite sulphur removal as a function of solid weight percentage in pulp and particle size ($X_1 = 1.5$ kg/t$_{\text{bitumen}}$, $X_2 = 0.3$ ppm, $X_5 = 0.3$ l min^{-1} and $X_6 = 1.5$ l min^{-1}).

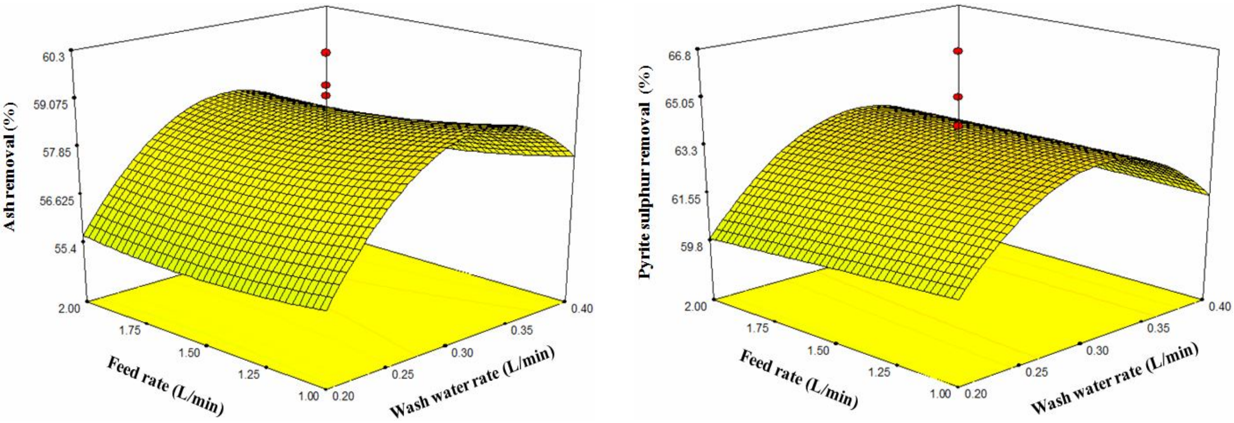

Fig. 4. Ash and pyrite sulphur removal as a function of solid weight percentage in pulp and particle size ($X_1 = 1.5$ kg/t$_{\text{bitumen}}$, $X_2 = 0.3$ ppm, $X_3 = 10\%$, $X_4 = 200$ mesh).

an increase in frother and collector amount. Furthermore, an increase in operation costs is observed due to the more collector and frother consumption [17].

The ash and pyrite sulphur removal from bitumen declined with the rise of particle size (See Fig. 3) [18], most possibly due to the lowered particle contact with air bubbles and detracted particles ability to raise bubbles to the froth zone resulting in a decrease in removal yields.

The efficacious area for heat and mass transfer increases when the particle size parameter is reduced. This last causes a growth in conversion factor of pyrite to sulfate which consequently improves the sulphur removal. Small particles have some benefit compared to large particles. For example, flotation and recovery of small particles are much better than large particles. Also, susceptibility of small and large particles is different for each reagent. For instance, the large particles need more collectors to have the same value of hydrophobicity as those of small particles; this increases the operating costs owing to collector and frother consumption increase. In general, the flotation of large particles is feasible only in the presence of oil collectors and higher aeration rates and longer time compared to smaller particles.

Table 2. BBD with Experimental and Predicted Values

Run	Independent variables						Experimental (%)		RSM (%)	
	X_1	X_2	X_3	X_4	X_5	X_6	Ash removal	Pyrite sulphur removal	Ash removal	Pyrite sulphur removal
1	-1	-1	0	-1	0	0	19.49	25.51	15.43	20.35
2	-1	-1	0	1	0	0	16.33	20.42	18.67	24.33
3	-1	0	-1	0	0	-1	52.84	58.21	52.44	57.02
4	-1	0	-1	0	0	1	63.61	68.09	52.44	57.02
5	-1	0	0	-1	-1	0	55.01	59.57	51.40	58.53
6	-1	0	0	-1	1	0	55.96	60.23	53.05	59.91
7	-1	0	0	1	-1	0	50.19	55.61	46.52	52.92
8	-1	0	0	1	1	0	51.29	57.12	48.17	54.30
9	-1	0	1	0	0	-1	54.15	59.26	52.44	57.02
10	-1	0	1	0	0	1	52.14	57.27	52.44	57.02
11	-1	1	0	-1	0	0	52.74	67.32	62.05	73.88
12	-1	1	0	1	0	0	41.57	48.05	49.05	58.69
13	0	-1	-1	0	-1	0	23.40	27.28	26.89	30.42
14	0	-1	-1	0	1	0	25.22	29.70	28.54	31.80
15	0	-1	0	0	-1	-1	24.61	28.44	26.89	30.42
16	0	-1	0	0	-1	1	23.33	27.36	26.89	30.42
17	0	-1	0	0	1	-1	26.49	29.91	28.54	31.80
18	0	-1	0	0	1	1	24.41	28.63	28.54	31.80
19	0	-1	1	0	-1	0	24.15	28.51	26.89	30.42
20	0	-1	1	0	1	0	25.51	29.03	28.54	31.80
21	0	0	-1	-1	0	-1	63.17	68.25	62.77	68.95
22	0	0	-1	-1	0	1	62.44	67.71	62.77	68.95
23	0	0	-1	1	0	-1	61.51	64.77	57.89	63.34
24	0	0	-1	1	0	1	55.49	60.71	57.89	63.34
25	0	0	0	0	0	0	57.49	62.19	60.33	63.94
26	0	0	0	0	0	0	59.17	64.06	60.33	63.94

Table 2. Continued

27	0	0	0	0	0	0	56.39	60.74	60.33	63.94
28	0	0	0	0	0	0	59.44	65.11	60.33	63.94
29	0	0	0	0	0	0	57.83	62.93	60.33	63.94
30	0	0	0	0	0	0	60.23	66.71	60.33	63.94
31	0	0	1	-1	0	-1	64.29	69.39	62.77	68.95
32	0	0	1	-1	0	1	63.14	68.20	62.77	68.95
33	0	0	1	1	0	-1	57.15	62.21	57.89	63.34
34	0	0	1	1	0	1	56.62	61.60	57.89	63.34
35	0	1	-1	0	-1	0	55.70	62.40	54.54	60.61
36	0	1	-1	0	1	0	56.82	62.14	56.19	61.99
37	0	1	0	0	-1	-1	56.37	63.91	54.54	60.61
38	0	1	0	0	1	-1	57.77	63.91	56.19	61.99
39	0	1	0	0	-1	1	56.20	62.19	54.54	60.61
40	0	1	0	0	1	1	57.19	63.88	56.19	61.99
41	0	1	1	0	-1	0	56.33	63.82	54.54	60.61
42	0	1	1	0	1	0	58.92	65.01	56.19	61.99
43	1	-1	0	-1	0	0	48.84	52.73	42.07	47.94
44	1	-1	0	1	0	0	61.43	65.90	45.31	51.91
45	1	0	-1	0	0	-1	64.29	67.80	68.22	70.85
46	1	0	-1	0	0	1	63.75	67.61	68.22	70.85
47	1	0	0	-1	-1	0	66.37	70.31	67.18	72.36
48	1	0	0	-1	1	0	70.81	75.20	68.84	73.73
49	1	0	0	1	-1	0	61.44	65.29	62.31	66.75
50	1	0	0	1	1	0	62.57	66.44	63.96	68.12
51	1	0	1	0	0	-1	65.37	69.49	68.22	70.85
52	1	0	1	0	0	1	64.55	68.77	68.22	70.85
53	1	1	0	-1	0	0	68.61	72.00	66.98	73.94
54	1	1	0	1	0	0	56.74	61.02	53.98	58.75

Table 3. Response Surface Quadratic Evaluation of Data

Response	Optimized coded equation	F value	Degree of freedom	R^2	Adjusted R^2	Predicted R^2	Adeq precision
Ash removal (%)	$+60.33 +7.89X_1 +13.82X_2 - 2.44X_4 +0.83X_5 -5.43X_1X_2 - 4.06X_2X_4 -16.14X_2^2 -2.65X_5^2$	68.38	8	0.924	0.910	0.815	29.677
Pyrite sulphur removal (%)	$+63.94 +6.91X_1 +15.09X_2 - 2.80X_4 +0.69X_5 -6.88X_1X_2 - 4.79X_2X_4 -14.92X_2^2 +2.21X_4^2 - 2.81X_5^2$	73.45	9	0.938	0.925	0.832	30.296

Table 4. The Predictability of the Optimized Models Using Five Independent Experimental Runs

Run	Independent variables						Experimental (%)		RSM Prediction (%)	
	X_1	X_2	X_3	X_4	X_5	X_6	Ash removal	Pyrite sulphur removal	Ash removal	Pyrite sulphur removal
1	-1	-1	0	-1	0	1	18.94	25.06	15.43	20.35
2	-1	1	0	-1	0	1	52.22	66.89	62.04	73.88
3	0	0	0	-1	0	1	62.11	67.24	62.77	68.94
4	1	-1	0	-1	0	1	48.59	52.30	42.07	47.94
5	1	1	0	-1	0	1	68.19	71.75	66.98	73.94

Effects of wash water rate and feed rate. Wash water rate positively affects ash and pyrite sulphur removal, though its effect is very small. (Fig. 4). This can be explained by the increase in water ratio to air ratio and the large bubbles explode in the froth zone at higher wash water rate.

Figure 4 presents ash and pyrite sulphur removal percentage *vs*. feed rate. It is obvious that feed rate has a negligible impact on ash and sulphur reduction. When feed rate is increased, removal yields reduces; most likely due to an increase in suspended solids in the froth zone and turbulence in the pulp.

The Results of Experimental Designs

In the present study, to evaluate the competency of models (*e.g*. linear, 2FI, quadratic and cubic) used to present ash and pyrite sulphur removal from bitumen, two different tests including sequential model sum of squares and model summary statistics were performed on the experimental data [19]. Evaluation of data by design matrix evaluation showed no aliases when response surface quadratic model was applied. Using this model, degree of freedom for lack of fit and pure error were obtained 40 and 5 for ash removal and, 39 and 5 for pyrite sulphur removal, respectively, which are higher than the minimum amount recommended (3 and 4, respectively [11]). The variance of inflation values (VIF) were very close to desirable value (*i.e.* 1.00) [11] which means that all coefficients are productively estimated. The reduced model results indicated that the model is significant (p-value less than 0.05). The other adequacy measures, *i.e.* R^2, adjusted R^2 and predicted R^2, showed a reasonable agreement and were close to 1, supporting the adequacy of the model [11]. Using backward elimination regression process ($\alpha = 0.10$) the final revised models were obtained and summarized in Table 3. The large F-value of all studied responses indicated that the selected models are significant. The "adequate precision" compares the signal-to-noise ratio; a ratio greater than 4 is desirable [11]. According to Table 3, the desirable value of this parameter indicated that this model could be used to navigate the design space.

Process Optimization

The conditions were optimized based on the best combination of factor levels that obtain maximum amounts for the studied response. The chosen criteria for optimization goal were 'maximize' for responses (percentage of ash and pyrite sulphur removal) and 'in range' for input factors. Among 30 proposed solutions, the top 26 solutions were expressed with higher desirability. The distinguished optimal conditions were the amount of collector (1.35-1.50 kg/$t_{bitumen}$), amount of frother (0.32-0.36 ppm), solid pulp content (5.00-14.74%), particle size (100.04-135.25 mesh), wash water rate (0.24-0.39 l min^{-1}) and feed rate (1.00-2.00 l min^{-1}) with the peak desirability value 100%. The maximum percentage of ash and pyrite sulphur removal from bitumen was 72.28% and 77.46%, respectively.

The predictability of the optimized models was investigated using five independent experimental runs. The summarized results in Table 4 demonstrate excellent agreement between the predicted and measured values.

CONCLUSIONS

In the present study, RSM modeling was applied to design the experiments and optimize the ash and pyrite sulphur removal from bitumen using column flotation process. Based on the present research the following conclusions can be made:

1. Increasing the amount of collector and wash water rate increases ash and pyrite sulphur removal from bitumen; whereas, increasing solid weight percentage, particle size and feed rate decreases both responses.
2. The ash and pyrite sulphur removal from bitumen increases with the amount of frother until it reaches to 0.3 ppm, then decreases as the amount of frother is increased.
3. The best result was achieved by setting the experiment with amount of collector at 1.5 kg/$t_{bitumen}$, amount of frother at 0.3 ppm, solid weight percentage at 10%, particle size at 100 mesh, and wash water rate at 0.4 l min^{-1} and feed rate at 1.5 l min^{-1}. In these conditions, 75.20% of pyrite sulphur (*e.g.;* 51.66% of total sulphur) and 70.81% of ash were removed.
4. The amounts of collector and frother agents play a significant role in the ash and pyrite sulphur removal from bitumen, while the feed rate has negligible impact on the removal of yields.

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