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OPTIMIZATION OF MOLYBDENITE FLOTATION USING RESPONSE SURFACE METHOD

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ABSTRACT

Purpose. The paper is aimed to study the molybdenite flotation from a low-grade uranium ore containing 0.2% of Mo.

Methods. Three control parameters including frother (MIBC) dosage, collector (gasoline) dosage and pH, each in five levels, were investigated. Response surface methodology (RSM) was performed for statistical design and analysis of experiments and process modeling. Four quadratic mathematical models were derived for prediction of Mo recovery and Mo grade.

Findings. Analysis of variance showed that frother and collector dosage were the most significant factors affecting Mo recovery and grade. In process optimization, maximum values of Mo recovery and grade were achieved as 79.13% and 2.93%, respectively. Optimum frother concentration of 78.93 g/t, gasoline dosage of 507.70 g/t, and pH of 9.77, for Mo recovery were obtained. However, in optimization studies, a case proposed the model in which the same consumption of reagents is used.

Originality. There is a recognized need for type of uranium ore which contains Molybdenite, therefore working on molybdenite removing from this ore helps to recover uranium in the next steps. This research provides a novel approach to gain the optimum recovery and grade to extract uranium so easily.

Practical implications. This study showed that response surface methodology could be effectively used for flotation process modeling as well as finding an optimum condition to achieve maximum recovery and grade under minimum consumption of flotation reagents.

Keywords: molybdenite, flotation, response surface methodology, central composite design, design of experiments

1. INTRODUCTION

Response surface methodology (RSM) is a collection of statistical and mathematical techniques useful for the modelling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response (Grice & Montgomery, 2000).

In most RSM problems, the relationship between dependent and independent variables is unknown. Thus, the first step in RSM is to find a suitable approximation for a functional relationship between dependent and independent variables. If there is a curvature in the system, then a polynomial of higher degree is used. Second-order models are widely used in response surface methodology as they have several advantages. They are very flexible and can take on a wide variety of functional forms so they will work well as an approximation to the true response

surface. Moreover, it is easy to estimate the parameters in a second-order model using the method of least squares (Grice & Montgomery, 2000).

Central composite design (CCD) contains five levels of each factor: low axial, low factorial, center, high factorial, and high axial. With this many levels, it generates enough information to fit a second-order polynomial (Mason, Gunst, & Hess, 2003; Anderson & Whitcomb, 2007).

Sedimentary uranium rocks may contain molybdenite that its grade changes from a few ppm to more than 0.5%. The small amount of molybdenum interferes with the recovery of uranium so it must be separated (Gupta, 1992).

The major technologies involving molybdenum compounds viz catalysis, lubrication, refractories, paints and allied industries and agriculture are well established. Because of the molybdenite structure and bonds, its natural floatability, compared with other minerals is high (Garner, 1994).

As Fuerstenau, Jameson, & Yoon (2007) stated that the best method to separate molybdenite from other minerals is flotation and mentioned the industrial instances of molybdenite flotation. Shirley and Sutulov (1985) and Hernlund (1961) explained some effective factors on floatability of molybdenite such as: ore deposit mineralogy, slime coatings, grinding and liberation and flotation reagents. Zanin, Ametov, Grano, Zhou, & Skinner (2009) described the variation of molybdenite from operation to operation and conducted experiments within different ore bodies.

Some researchers by focusing on the chemical changes in reagents, suggested some methods to conduct the experiments for enhancing the best recovery. Triffett, Veloo, Adair, & Bradshaw (2008) proposed several changes to increase molybdenum recovery and then optimized frother to collector ration to earn adequate froth stability.

Biswas and Davenport (2013) explained that Kerosene and fuel oil are good collectors for molybdenite and used potassium amyl xanthate as scavenger flotation. In other researches (Schena & Casali, 1994; Bulatovic, Wyslouzil, & Kant, 1998; Rath & Subramanian, 1999; Rubio, Capponi, Rodrigues, & Matiolo, 2007) kerosene, vapor oil, fuel oil, transformer oil and other hydrocarbons were used in direct flotation of molybdenite. Additionally, Yin, Zhang, & Xie (2010) used kerosene as the collector in Xinhua Molybdenum Flotation Plant (Liao-

ning, China). Thus the floatability of molybdenite is enhanced if an insoluble, nonpolar hydrocarbon oil is added to flotation cell (Smit & Bhasin, 1985). Therefore, fuel oil used as collector in this study and methyl isobutyl Carbinol (MIBC) used as frother.

The main objective of this research was first to establish a functional relationship between three process variables (frother dosage, collector dosage, and pulp pH) and the flotation characteristics (Mo recovery, Mo grade of molybdenite concentrate), using a statistical technique. Central composite design approach was used to determine significant factors that affect the molybdenite flotation and to develop quadratic mathematical models for the optimization of the process. Consequently, the responses were maximized using the minimum consumption of molybdenite flotation reagents.

2. MATERIALS AND METHODS

2.1. Molybdenum ore characteristics

The low-grade ore sample containing 0.2% Mo was obtained from Narigan Uranium ore, Iran. For ore characterization, optical mineralogy, using the polished and thin sections, as well as x-ray diffraction techniques (XRD) were used. The XRD analysis of the original ore sample is also given in Table 1.

Table 1. XRD analysis of molybdenite sample

Compose	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	Cl	K ₂ O	CaO	TiO ₂
Concentration (%)	0.55	2.05	11.95	61.75	0.47	5.28	0.0003	2.83	6.86	0.82
intensity	1.9	33.7	604.8	5569.3	58.6	1114.6	8.9	987.6	3004.2	582.8
compose	Cr ₂ O ₃	MnO	Fe ₂ O ₃	CuO	Zn	As ₂ O ₃	ZrO ₂	UO ₂	MoO ₂	
Concentration (%)	0.055	0.19	6.24	0.05	0.21	0.14	0.04	0.06	0.20	
intensity	89.6	527.8	17355.3	56.2	401.4	462.6	351.4	255.1	659.8	

2.2. Experimental and modelling procedures

2.2.1. Flotation experiments

Flotation experiments were carried out in 2.5 l Denver laboratory flotation cell using 252 g ore sample. Gasoline and methyl isobutyl carbonyl (MIBC) were used as flotation collector and frother, respectively. The control factors and their levels were selected in the light of the feasibility flotation experiments and literature review.

Based on the previous studies three factors showed more influence on the process. The selected control factors, including frother dosage, collector dosage and pulp pH and their levels are shown in Table 2.

Table 2. The level of variables in the Central composite design

Variable	Low axial (-1.68)	Low factorial (-1)	Center (0)	High factorial (+1)	High axial (+1.68)
A: Frother dosage (MIBC) (g/t)	63	70	80	90	97
B: Collector dosage (Gasoline) (g/t)	432	500	600	700	768
C: pH	7.8	8.5	9.5	10.5	11.1

Each factor varied in five levels, whereas the other operational parameters of flotation were kept constant. Pulp density of 20% solid, conditioning time of 10 min

for collector, and conditioning time of 1 min for frother, the froth collection time of 7 min and rotor speed of 1200 rpm were used in all of the flotation experiments.

2.2.2. Central composite design

The most frequently used second-order designs are the 3^k factorial, central composite, and the Box-Behnken designs. Central composite design (CCD) is the most popular response surface method (Grice & Montgomery, 2000; Mason, Gunst, & Hess, 2003; Anderson & Whitcomb, 2007). This design consists of the following three portions:

- a complete (or fractional) factorial design, whose factors' levels are coded as -1, 1;
- an axial design, often a star design in which experimental points are at a distance of α , from its center;
- a central point.

The quadratic model, which is a second order polynomial, could be fitted using these levels. Fitting of the model can be computed by a standard statistical software. Using the statistical software packages, like "design of experiments" (DX7), based on RSM, the optimum flotation factors could be achieved with the minimum number of experiments. To define a central point, the polynomial function should be contained quadratic terms according to the following equation:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{<i<j}^k \beta_{ii} x_i^2 + \sum_{1<i<j}^k \beta_{ij} x_i x_j + \epsilon, \quad (1)$$

where:

k , β_0 , β_i , x_i , β_{ii} , β_{ij} and ε – number of variables, constant term, coefficients of the linear parameters, variables, coefficients of the quadratic parameters, coefficients of the interaction parameters and residual associated with the experiments, respectively (Bezerra, Santelli, Oliveira, Villar, & Escaleira, 2008).

3. RESULTS AND DISCUSSION

3.1. Construction of model equations

Twenty flotation experiments were designed using central composite design methodology. The experimental conditions and their responses are shown in Table 3.

Table 3. Experimental circumstances and obtained results

Run	A: Frother dosage		B: Collector dosage		C: pH		Recovery (%)	Grade (%)
	Coded	Actual	Coded	Actual	Coded	Actual		
1	-1	70	1	700	1	10.50	68.35	2.76
2	0	80	0	600	1.68	11.18	64.07	2.79
3	0	80	0	600	0	9.50	78.07	3.13
4	0	80	0	600	-1.68	7.82	64.45	2.81
5	0	80	0	600	0	9.50	73.7	2.97
6	-1.68	63.18	0	600	0	9.50	69.97	1.34
7	0	80	-1.68	431.82	0	9.50	59.74	2.85
8	0	80	1.68	768.18	0	9.50	78.62	2.41
9	1	90	-1	500	-1	8.50	57.25	3.2
10	0	80	0	600	0	9.50	77.8	2.7
11	0	80	0	600	0	9.50	74.76	3.05
12	-1	70	1	700	-1	8.50	61.98	2.1
13	1	90	-1	500	1	10.50	65.52	2.89
14	-1	70	-1	500	-1	8.50	65.1	1.51
15	0	80	0	600	0	9.50	79.11	3.07
16	1	90	1	700	1	10.50	74.89	1.75
17	-1	70	-1	500	1	10.50	65.66	1.97
18	0	80	0	600	0	9.50	75.15	3.1
19	1	90	1	700	-1	8.50	69.12	1.72
20	1.68	96.82	0	600	0	9.50	74.76	1.89

The quadratic models among several models were chosen and fitted to the results in “Design Expert (DX 7)” software. Two models were fitted to Mo recovery and Mo grade.

Quadratic models found to be adequate for the prediction of the response variables are given by the following equations (Mo recovery and Mo grade):

$$\begin{aligned} \text{Mo } R = & +76.42 + 0.64A + 3.77B + 1.53C + \\ & + 2.08AB + 1.53AC - 0.21BC - 1.49A^2 - \\ & - 3.35B^2 - 4.95C^2; \end{aligned} \quad (2)$$

$$\begin{aligned} \text{Mo } G = & +3.01 + 0.16A - 0.14B + 0.059C + \\ & + 0.50AB - 0.17AC + 0.068BC - 0.50A^2 - \\ & - 0.14B^2 - 0.078C^2. \end{aligned} \quad (3)$$

In these models, all variables are in coded values and A is frother dosage, B is collector dosage, C is pH and AB , AC and BC are interaction of main parameters. The results of analysis of variance (ANOVA) of the developed models are shown in Table 4.

Table 4. The results of ANOVA analysis of the developed models

		Sum of square	DOF	Mean square	F-value	P-value
Mo recovery	Model	753.60	9	83.73	8.09	0.0015
	Residual	103.44	10	10.34		
Mo grade	Model	6.79	9	0.75	40.37	<0.0001
	Residual	0.19	10	0.019		

It illustrates that all fitted models are significant in 95% confidence level (p -value < 0.05). Figure 1 and 2 represent predicted against actual values for Mo recovery and Mo grade, respectively.

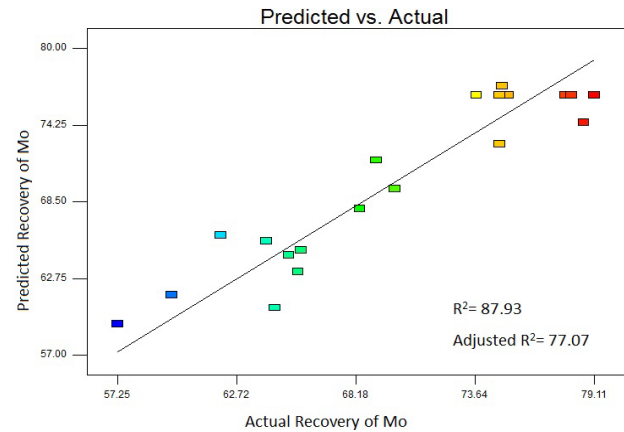


Figure 1. Predicted vs. actual values of Mo recovery, %

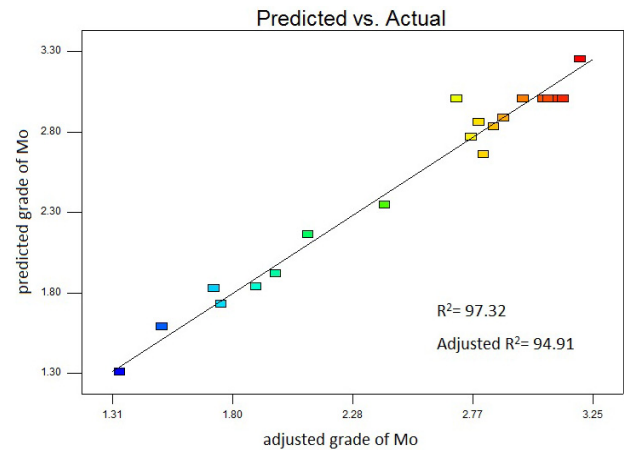


Figure 2. Predicted vs. actual values of Mo grade, %

Values of R-square for the models are shown in these figures. The high value of R-square indicates that the quadratic equation is capable of representing the system under the given experimental domain. Therefore, according to model interactions between variables have significant effects on responses, so results were presented and discussed in terms of interactions. Figure 3 shows the effect of frother (MIBC) dosage and collector (Gasoline) dosage on the Mo recovery.

It is indicated that Mo recovery depends more on the collector dosage rather than the frother dosage. Accordingly, collector dosage has shown a positive effect on Mo recovery.

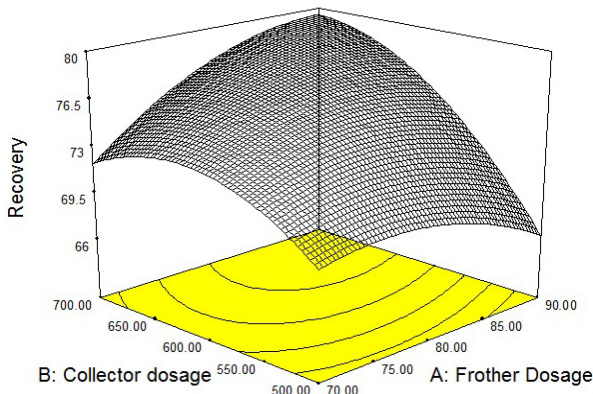


Figure 3. Effect of frother and collector dosages on Mo recovery in pH 9.77

Meanwhile, it has been the most significant factor affecting the Mo recovery in comparison with the other two factors. It can be deduced from Figure 4 that the effects of pH and collector dosage on the Mo recovery have been positive until the pulp pH reaches to 9.77.

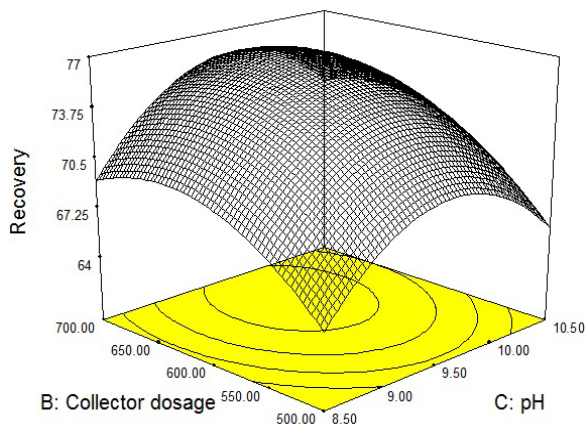


Figure 4. Effect of collector dosage and pH on Mo grade (%) in frother dosage of 78.93 g/t

Unlike Mo recovery, Mo grade increased with increasing frother and collector dosage until the specific point and then decreased (Fig. 5).

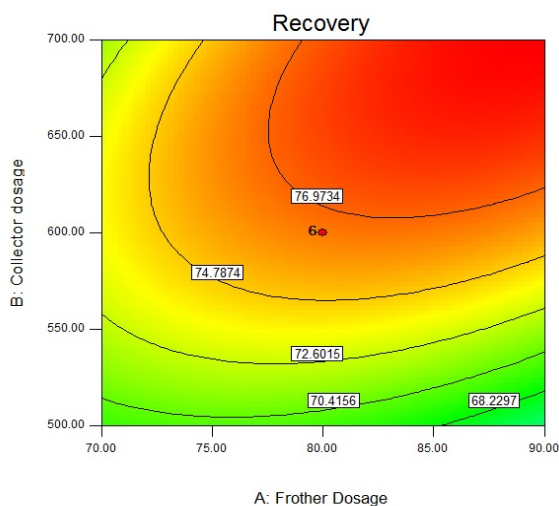


Figure 5. Effect of frother and collector dosages on Mo grade in pH of 9.77 (countor plot)

Figure 6 and 7 show the effect of frother and collector dosages on Mo grade. They are stated that both frother and collector concentration effect on the Mo grade in the constant pH = 9.77.

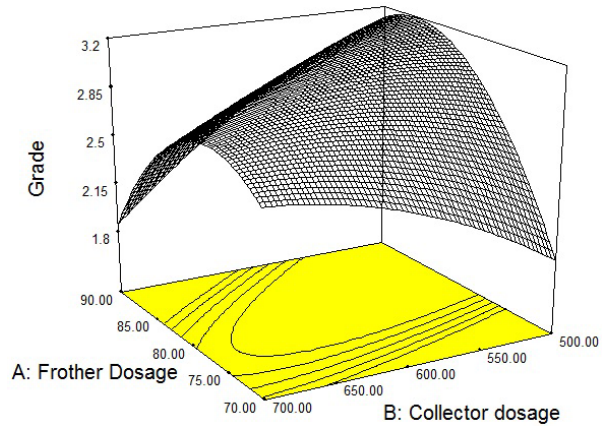


Figure 6. Effect of frother and collector dosages on Mo grade (%) in pH 9.77

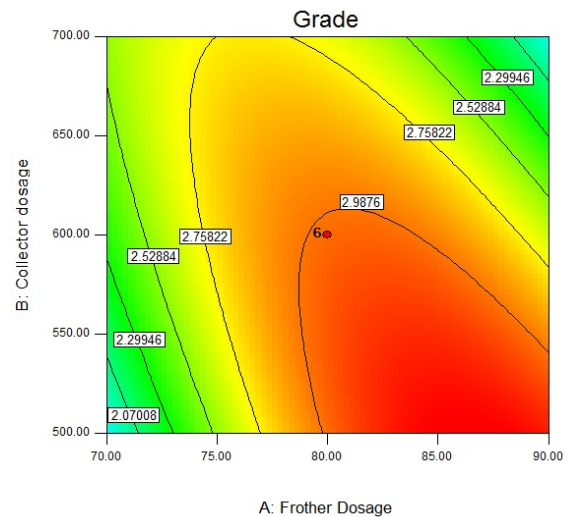


Figure 7. Effect of frother and collector dosages on Mo grade (countor plot)

As seen in Figure 8, Mo grade with increasing in collector and pH, increased until 2.93% and then decreased.

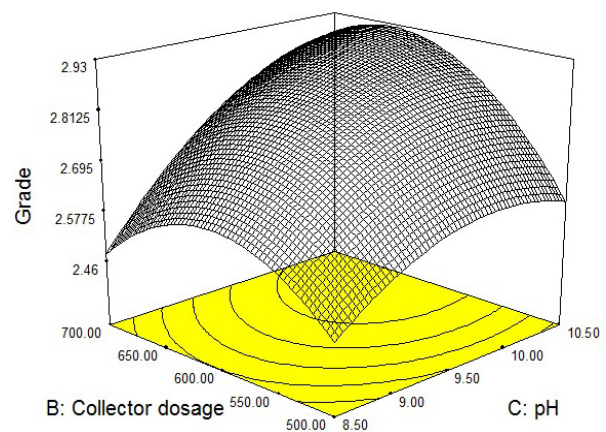


Figure 8. Effect of collector dosage and pH on Mo grade (%) in frother dosage of 78.93 g/t

3.2. Propagation of error

Propagation of error (POE) is a tool to find controllable factors settings that maximize quality, which we define as making a product to target with minimum variation. It requires construction of mathematical models via response surface methods (RSM) (Brown, Box, & Draper, 1990). Using the RSM and the POE techniques, we seek levels of the controllable factors that center response values on their respective targets while simultaneously reducing variation transmitted to the response from variation (lack-of-control) in the controllable factors (Whitcomb & Anderson, 1996). In designed experiments, variations of frother dosage, collector dosage and pH were calculated 1, 2 g/t and 0.05, respectively. Figures 9 and 10 illustrate the effect of frother and collector dosages on the POE values of the Mo recovery and grade, respectively.

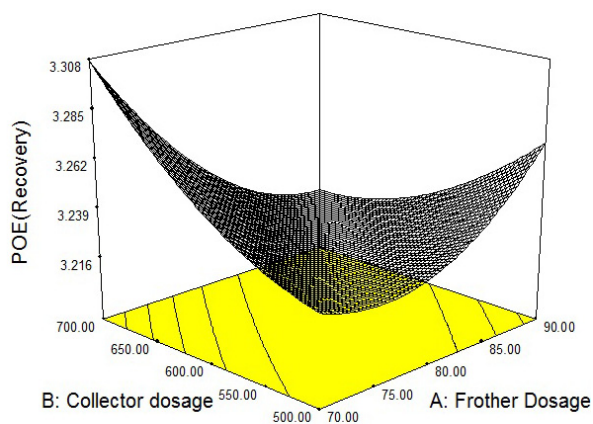


Figure 9. Effect of frother and collector dosages on POE values of Mo recovery

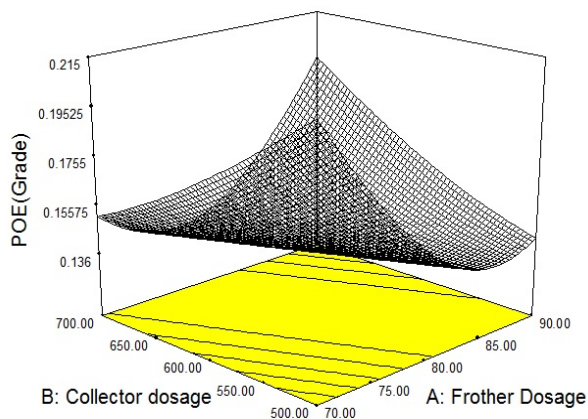


Figure 10. Effect of frother and collector dosages on POE values of Mo grade

In Figure 9 frother concentration increased POE in upper collector dosage, while its effect was conversed in lower collector dosages but in Figure10 in lower dosage of collector, frother decreased POE and then increased POE.

3.3. Optimization

Finding a desirable point in the design space is the goal of response surface optimization. The optimum point could be an extremum, or an area where the response is stable over a range of factors. A simultaneous

optimization method was used by DX7 software to optimize the responses. In this work, the POE minimized to obtain robust recovery and grade against factor variations, so POE values were minimized in all optimization cases.

In Table 5 optimum conditions and limit of them is shown. Table 6 shows the results for optimum conditions.

Table 5. Optimum process conditions

Name	Goal	Limit	Limit
Frother Dosage	minimize	70	90
Collector dosage	minimize	500	700
pH	is in range	8.5	10.5
POE (Recovery)	minimize	3.22	3.35
Grade	maximize	1.34	3.2
POE (Grade)	minimize	0.14	0.23

Table 6. Optimum process results

Solution							
Frother Dosage	Collector dosage	pH	Recovery	POE (Recovery)	Grade	POE (Grade)	Desirability
78.93	507.7	9.77	79.13	3.22	2.93	0.15	0.82

Maximum Mo recovery was reached to 79.13% using 78.93 g/t MIBC, 507.7 g/t gasoline and pH 9.77. Maximum grade also is 2.93% with minimum consumption of the flotation reagents were followed.

4. CONCLUSIONS

Modelling and optimization of the Mo flotation process from a low-grade Uranium ore containing 0.2% Mo was performed, using response surface methodology-central composite design (RSM-CCD). Frother (MIBC) dosage, collector (Gasoline) dosage and pulp pH were the control factors in this study. Four quadratic models for Mo recovery and Mo grade in the molybdenite concentrate were developed. Process optimization was carried out in Table 6. In recovery and grade, POE value was minimized. Some of the most important conclusions derived from optimization studies are as follows:

- maximum Mo recovery of 79.13% was obtained using 78.93 g/t MIBC, 507.7 g/t gasoline in pulp pH of 9.77;
- 2.93% for Mo grade of was obtained using 78.93 g/t MIBC 507.7 g/t gasoline in pH of 9.77.

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ОПТИМІЗАЦІЯ ПРОЦЕСУ ФЛОТАЦІЇ МОЛІБДЕНІТУ ЗА ДОПОМОГОЮ МЕТОДУ ПОВЕРХНІ ВІДГУКУ

Р. Бадрі, А.Р. Ханчі, А.Р. Зояжі, А.А. Рахмані

Мета. Дослідження особливостей процесу флотації молібденіту з низькозбагаченої уранової руди, що містить 0.2% молібдену.

Методика. Проаналізовано три контрольних параметра флотації – дозу спінювача (метил-ізобутил-карбінол), колекторне (газолінове) дозування і рН (кожен – у п'яти різницях рівнів). Для статистичного розрахунку, аналізу експериментів і моделювання процесу флотації застосовано метод поверхневого відклику (МПВ). Були розроблені чотири квадратичні математичні моделі для розрахункових даних отримання Мо і визначення його якості.

Результати. Встановлено, що спінювач і колекторне дозування є найбільш впливовими факторами при отриманні Мо та його якості. У процесі оптимізації максимальні рівні отримання Мо та якості були, відповідно, 79.13% і 2.93%. Виявлено наступну оптимальну концентрацію для Мо: спінювач – 78.93 г/т, газолінове дозування – 507.70 г/т, рН – 9.7, що підтверджено моделюванням.

Наукова новизна. Запропоновано інноваційний підхід для отримання легкого й оптимального способу відновлення і ступеня вилучення урану до високої якості.

Практична значимість. Метод поверхневого відклику може ефективно застосовуватися для моделювання процесу флотації з метою визначення оптимальних умов досягнення максимальної рекуперації та якості при мінімальних витратах флотаційних реагентів.

Ключові слова: молібденіт, флотація, метод поверхневого відклику, центральний композиційний план, схема проведення експериментів

ОПТИМИЗАЦИЯ ПРОЦЕССА ФЛОТАЦИИ МОЛИБДЕНИТА ПОСРЕДСТВОМ МЕТОДА ПОВЕРХНОСТНОГО ОТКЛИКА

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Цель. Исследование особенностей процесса флотации молибденита из низкообогатенной урановой руды, содержащей 0.2% молибдена.

Методика. Проанализированы три контрольных параметра флотации – доза вспенивателя (метил-изобутил-карбинол), коллекторная (газолиновая) дозировка и pH (каждый – в пяти разностях уровней). Для статистического расчета, анализа экспериментов и моделирования процесса флотации применен метод поверхности отклика (МПО). Были разработаны четыре квадратичные математические модели для расчетных данных получения Mo и определения его качества.

Результаты. Установлено, что вспениватель и коллекторная дозировка являются наиболее влияющими факторами при получении Mo и его качества. В процессе оптимизации максимальные уровни получения Mo и качества были, соответственно, 79.13% и 2.93%. Выявлена следующая оптимальная концентрация для Mo: вспениватель – 78.93 г/т, газолиновая дозировка – 507.70 г/т, pH – 9.7, что подтверждено моделированием.

Научная новизна. Предложен инновационный подход для получения легкого и оптимального способа восстановления и степени извлечения урана до высокого качества.

Практическая значимость. Метод поверхностного отклика может эффективно применяться для моделирования процесса флотации с целью определения оптимальных условий достижения максимальной рекуперации и качества при минимальном расходе флотационных реагентов.

Ключевые слова: молибденит, флотация, метод поверхностного отклика, центральный композиционный план, схема проведения экспериментов

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