

*J. Min. Metall. Sect. B-Metall.* 48 (1) B (2012) 89 - 99

Journal of  
Mining and  
Metallurgy

## DETERMINATION OF OPTIMAL CONDITIONS FOR PRESSURE OXIDATIVE LEACHING OF SARCHESHMEH MOLYBDENITE CONCENTRATE USING TAGUCHI METHOD

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*(Received 08 March 2011; accepted 26 August 2011)*

### Abstract

The present research work is based on finding the optimum conditions for pressure oxidative leaching of the molybdenite concentrate to produce technical-grade molybdic oxide ( $\text{MoO}_3$ ) with high recovery through further treatment of the filtrate solution. The Taguchi method was used to design and minimize the number of experiments. By using Taguchi orthogonal ( $L_{25}$ ) array, five parameters (time, temperature, oxygen pressure, pulp density and acid concentration) at five levels were selected for 25 experiments. The experiments were designed and carried out in a high-pressure reactor in the presence of nitric acid as solvent and oxidizing agent for the molybdenite concentrate and its  $\text{ReS}_2$  content. The optimum conditions for pressure leaching of molybdenite were obtained through using Signal to Noise analysis and modified by using Minitab software prediction tool. Furthermore, the optimum condition for an economical pressure leaching of rhenium sulfide ( $\text{ReS}_2$ ) was achieved with the same process. Analysis of variance (ANOVA) showed that the pulp density is of paramount importance in this process.

*Keywords:* Molybdenite concentrate, Pressure leaching, Rhenium sulfide, Taguchi method.

### 1. Introduction

Molybdenite ( $\text{MoS}_2$ ) is the main source of molybdenum element which occurs in porphyry copper deposits. Roasting of

molybdenite around  $650^\circ\text{C}$  is the most common pyrometallurgical process to extract molybdenum and rhenium [1].

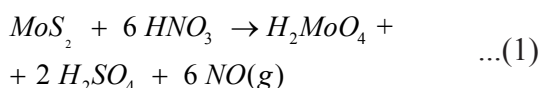
Environmental pollution caused by the released sulfur dioxide and considerable

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amount of rhenium waste in the roaster flue gas are the major disadvantages of the molybdenum pyrometallurgical processes [1].

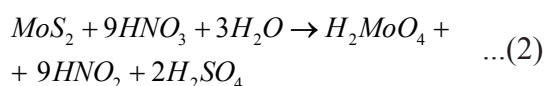
Potential hydrometallurgical processes for molybdenum and rhenium extraction are: a) leaching in the presence of oxidizing agents such as oxygen, ferric ions, nitric acid, concentrated sulfuric acid, chlorine water and sodium hypochlorite; b) pressure leaching at elevated pressures and temperatures; c) electro-oxidation leaching; and d) bacterial leaching [2-13].

The reaction of molybdenite and nitric acid under atmospheric pressure can be derived by the following equation [14]:

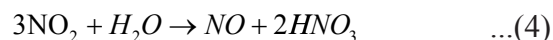
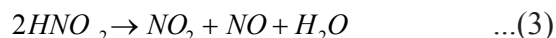


Low solubility of molybdic acid ( $\text{H}_2\text{MoO}_4$ ) in water (1.82g/l) is an advantage of this process because a supersaturated solution of molybdic acid decomposes to hydrated molybdic oxide precipitates. As a result of sulfuric acid production and the increase in its concentration, the solubility of molybdic acid increases (maximum 8g/l for molybdenum). Decomposition of supersaturated molybdic acid solution to hydrated molybdic oxide precipitates occurs at the temperatures above 80°C. The kinetic of this process is low which can be a drawback for an industrial process (reaction completion may take 8 days) [15].

Pressure leaching of  $\text{MoS}_2$  in dilute  $\text{HNO}_3$  at high temperatures (near 100°C) and under oxygen pressure can be readily derived from the following reaction [16, 17]:



Produced nitrous acid rapidly decomposes to NO and  $\text{NO}_2$  by the following reaction [18]:



$\text{NO}_2$  reacts with water to form nitric acid. Also in presence of oxygen, NO is oxidized to  $\text{NO}_2$  (Reaction 4) and then, to nitric acid (Reaction 4) [19].



Regeneration of the nitric acid is a useful process and causes the required initial amount of nitric acid to be less than theoretical and stoichiometric.

It is well established for the saturation limit of molybdic acid, that approximately 85% of entire molybdenum in molybdenite concentrate precipitates as hydrated molybdic oxide. Also, rhenium sulfide in the molybdenite concentrate reacts with dilute nitric acid and produces perrhenic acid ( $\text{HReO}_4$ ) solution. The solution, containing rhenium and the remaining 15% of the molybdenum, is subjected to solvent extraction for further purification.

Since the leaching experiments are time-consuming and expensive, Taguchi method has been established to optimize and predict the effects of various leaching parameters in many hydrometallurgical researches [21-26]. Analysis of variance on the collected data from Taguchi design of experiments can be used to select new parameter values to optimize the performance characteristics [20] of investigated process. The experimental design proposed by Taguchi involves using orthogonal arrays to organize

the parameters affecting the process and the levels at which they would be varied; it allows for the collection of the required data to determine which factors most affect product quality with a minimum amount of experimentation, thus saving time and resources.

In the current study, experiments were carried out to optimize conditions for the pressure oxidative leaching of the molybdenite concentrate in nitric acid using Taguchi method for designing and analyzing the experiments.

## 2. Experimental procedures

### 2.1. Materials

The molybdenite concentrate was supplied by Sarcheshmeh Copper Complex in Iran. Chemical composition of the molybdenite concentrate is shown in Table 1. All the chemical reagents used in this research work were of analytical grade and used as received without further purification.

Remained flotation oil in molybdenite

concentrate was removed by triple washing of the concentrate in acetone, then in water, and it was finally dried at 100°C. Particle size analysis of the obtained molybdenite concentrate showed that 80% of particles were less than 40µm in diameter.

### 2.2. Experimental Plan

On the basis of the previous researches, time, temperature, acid concentration, pulp density, and oxygen pressure were chosen as five factors to be investigated. The size of the powder particles of the molybdenite concentrate was in the range of 80% <40µm and it was impossible to study the effect of particle size on pressure leaching. According to Taguchi design methodology, five levels were designed for each factor as illustrated in Table 2. Considering five factors and relevant five levels, Taguchi orthogonal array (L<sub>25</sub>) was established to execute the experiments. One to five levels appearing in Table 3 are relative levels of the parameters indicated in Table 2. Twenty five experiments were conducted as shown in

Table 1. Chemical analysis of Sarcheshmeh Molybdenite concentrate

Elements	Mo (wt%)	S(wt%)	Fe(wt%)	Cu(wt%)	Si(wt%)	Zn(ppm)	Re(ppm)	Se(ppm)
Content	55.55	38.9	1.5	0.5	1.01	510	600	560

Table 2. Experimental parameters and their relative levels

Parameters	Levels				
	1	2	3	4	5
Time (min)	60	120	180	210	240
Temperature (°C)	120	130	140	150	160
Oxygen overpressure (kPa)	827.38	965.27	1103.16	1241.06	1378.95
Pulp density (g/l)	100	125	150	175	200
Acid concentration (g/l)	25	30	35	40	50

Table 3. The interactions between parameters were not taken into consideration.

### 2.3. Procedure

All experiments were conducted in Stainless Steel pressure vessel equipped with a mechanical stirrer in accordance with test conditions specified in Table 3. After each experiment the precipitates and filtrate were

separated by sintered glass. The precipitates were washed, dried and weighed. To analyze total molybdenum, iron and copper content of the filtrate, the filtrate solution samples were subjected to Atomic Absorption Spectroscopy (by a GBC Avanta PM spectrometer). Rhenium content of the samples was determined by a bausch and lomb spectronic 20 model Visible Spectrophotometer, and sulfur content of

Table 3. Taguchi orthogonal array ( $L_{25}$ ) for influencing parameters

Exp. No	Parameters and their relative levels				
	Time	Temperature	Oxygen overpressure	Pulp density	Acid concentration
1	1	1	1	1	1
2	1	2	2	2	2
3	1	3	3	3	3
4	1	4	4	4	4
5	1	5	5	5	5
6	2	1	2	3	4
7	2	2	3	4	5
8	2	3	4	5	1
9	2	4	5	1	2
10	2	5	1	2	3
11	3	1	3	5	2
12	3	2	4	1	3
13	3	3	5	2	4
14	3	4	1	3	5
15	3	5	2	4	1
16	4	1	5	4	3
17	4	2	1	5	4
18	4	3	2	1	5
19	4	4	3	2	1
20	4	5	4	3	2
21	5	1	4	2	5
22	5	2	5	3	1
23	5	3	1	4	2
24	5	4	2	5	3
25	5	5	3	1	4

precipitates via Schtroline furnace.

Taguchi method recommends using loss function for measuring the performance characteristics which deviate from the desired values. The value of the loss function is further transformed into a signal-to-noise ratio. The signal to noise ratio (SN) expresses the scatter around a target value. The larger the value, the smaller the scatter. Whenever an experiment involves repeated observations at each of the trial conditions, the SN ratio has been found to provide a practical way to measure and control the combined influence of deviation of the population mean. Usually, there are three categories of performance characteristics in the analysis of the SN ratio which are: the lower the better; the higher the better; and the nominal the better. The SN ratio for each level of the process parameters is computed based on the SN analysis. Regardless of the category of the performance characteristics, larger SN ratio corresponds to better performance characteristic. Therefore, the optimal level of the process parameters is the level with the highest SN ratio [27].

With respect to pressure leaching of the molybdenite, higher number of SN ratio is better.

$$SN_L = -10 \log \left( \frac{1}{r} \sum_{i=1}^r \frac{1}{Y_i^2} \right) \quad \dots(6)$$

where,  $r$  is the number of repetition for each experiment and  $Y_i$  is the response of each experiment.

The amount of leaching efficiency for the molybdenum is calculated as following:

$$Y = \frac{m + m'}{m_0} \times 100 \quad \dots(7)$$

where,  $m$  is the weight of molybdenum in

liquor,  $m'$  is the weight of molybdenum in precipitated molybdc oxide,  $m_0$  is the weight of molybdenum in molybdenite concentrate and  $Y$  is the leaching efficiency.

## 2.4. Data Analysis

Analysis of Variance (ANOVA) is a statistical technique to provide a measure of confidence. The technique does not directly analyze the data, rather determines the variability (variance) of data. Confidence is measured from the variance. Analysis provides the variance of controllable and noise factors. By understanding the source and magnitude of variance, robust operating conditions can be predicted [20].

All rhenium and molybdenum leaching and oxidation data were subjected to Analysis of Variance (ANOVA) by using the Equations 8-16 presented in appendix A [20].

## 3. Results and Discussion

The results, shown in Table 4 and obtained by Taguchi orthogonal array (L<sub>25</sub>), are in accordance with Table 3. Using Minitab 15 statistical software [30], the results were analyzed to derive SN graphs as shown in Fig. 1. Each experiment was repeated twice and thus, two responses for both molybdenum and rhenium were achieved.

Since higher SN ratio is better in molybdenite pressure oxidative leaching, the leaching is optimized when responses are as large as possible. Considering Fig. 1, the level related to the maximum SN value in each graph is considered as the optimum condition. It must be mentioned that these

graphs are used only to show the trend of factors and not to predict other conditions.

Due to maximizing the amount of SN ratio and accordingly the leaching efficiency for molybdenum and rhenium, optimized conditions were selected as shown in Table 5.

It was verified that oxygen pressure had no significant effect on leaching efficiency of both molybdenum and rhenium as shown in Fig. 1c.

Table 4. Responses and Signal to Noise ratios for leaching of molybdenum and rhenium

Exp No	Responses for Molybdenum			Responses for Rhenium		
	1	2	SN <sub>L</sub>	1	2	SN <sub>L</sub>
1	52.63	50.46	34.24	52.5	54.22	34.54
2	51.3	57.67	34.68	73.87	67.6	36.97
3	33.94	38.59	31.14	42.73	44.67	32.8
4	56.26	57.1	35.07	53.2	59.6	34.98
5	57.45	56.15	35.09	52.67	62.63	35.12
6	42.08	43.82	32.65	47.56	57.78	34.31
7	51.34	48.94	34	72.3	68.5	36.94
8	30.17	36.28	30.32	38.7	33.6	31.1
9	98.72	98.71	39.89	99.99	99.99	40
10	56.69	54.71	34.91	46.53	56.49	34.12
11	39.54	42.1	32.2	44	42.9	32.76
12	97.97	98.04	39.82	72.83	81.88	37.73
13	83.92	79.65	38.24	98	99.99	39.91
14	75.2	76.23	37.58	58.67	43.1	33.83
15	34.15	36.4	30.94	42.59	35.67	31.75
16	43.56	39.19	32.3	34.67	47.24	31.94
17	60.3	58.34	35.46	61.5	65.8	36.06
18	98.96	99	39.91	99.99	98.5	39.93
19	73	74.33	37.34	98.67	88.13	39.37
20	45.32	48.96	33.45	43.73	40.9	32.52
21	90.11	91.28	39.15	99.99	93.4	39.69
22	29.46	32.63	29.81	37.6	33.9	31.03
23	38.93	43.65	32.27	33.68	42.84	31.47
24	48.89	48.31	33.73	39.23	38.74	31.82
25	98.95	98.95	39.91	96	99.99	39.82

This shows that only nitric acid acted as oxidizing agent. Main role of oxygen was to keep the pressure of the vessel high enough to prevent liquid from boiling and oxidation of produced nitrous acid to nitric acid.

The Pulp density was the most important influencing parameter on the leaching efficiency of molybdenum, which is conversely proportional to the SN ratio (Fig. 1d). This could be resulted from the lack of acid due to leaching and oxidizing the excessive amount of the molybdenite concentrate. Mass balance calculations for all conducted experiments showed that the utmost 50g/l of leached molybdenum precipitated as molybdic oxide. This fact shows that about 100 g/l of molybdenite concentrate reacted with nitric acid in acid concentration rang of 25-50 g/l to produce precipitated molybdic oxide and soluble molybdic acid (H<sub>2</sub>MoO<sub>4</sub>).

In the absence of the nitric acid, oxygen acted as an oxidizing agent only at higher pressures and temperatures. Previous studies on pressure leaching of molybdenite showed that molybdenite leaching occurred at temperatures higher than 200°C and oxygen pressures higher than 1723.69 kPa [6]. Thus, leaching of molybdenite at temperatures lower than 200°C and by water was time-consuming. The optimized level of nitric acid concentration was only enough for 100g/l of molybdenite concentrate.

The next step of data evaluation consisted of Analysis of Variance (ANOVA) using Equations 8-16 (Appendix A). Results of the mentioned analysis are illustrated in Table 6 and 7 for molybdenum and rhenium, respectively. Again, according to Table 6, the pulp density had the highest contribution percentage in pressure leaching of molybdenum. Therefore, it

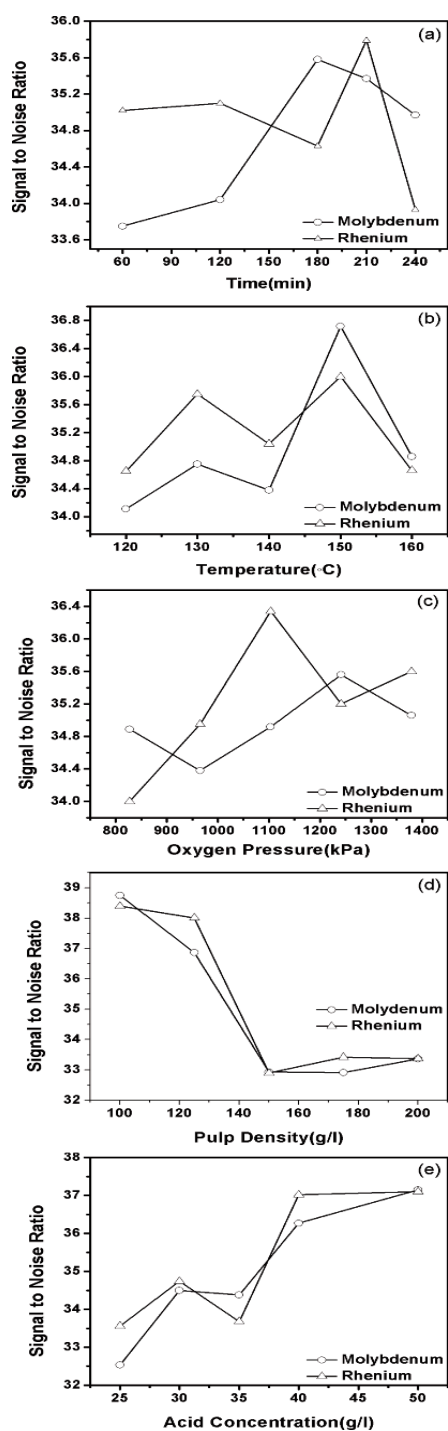


Fig. 1. Effect of controllable parameters (a: Time, b: Temperature, c: Oxygen pressure, d: Pulp density, e: Acid concentration) on Signal to Noise ratio for pressure leaching of molybdenum and rhenium.

was proven that the leaching conditions were very sensitive to pulp density.

As presented in Tables 6 and 7, the variance of ratio at a level of reliability (F) showed the statistical confidence of each factor on the molybdenum and rhenium leaching efficiencies [20]. The computed F ratios shown in Tables 6 and 7, confirmed that only the pulp density and acid concentration had statistically significant effect on the leaching efficiencies of the molybdenum and rhenium.

Minitab 15 software provides the ability of responses prediction for various combinations of factors. This feature is based on Response Surface Methodology (RSM) which was modified by professor Taguchi. Response surface methodology is already established as an important tool in experimental design in terms of developing new processes and optimizing their performance [28, 29]. If there are minimal interactions among the factors or if the interactions have been correctly accounted for by the predictions, the observed results would have been close to the prediction. On the other hand, if there is substantial disagreement between the prediction and the observed results, then there may be unaccounted for interactions or unforeseen noise effects.

To examine the ability of prediction for the current study, the plots of predicted versus measured values were derived (Figs. 2a and b).  $R^2$  value for Figs. 2a and 2b showed good fittings for both molybdenum

Table 5. Optimal working conditions for leaching of Molybdenum and Rhenium

Parameters	Molybdenum	Rhenium
Time(min)	180	210
Temperature(°C)	150	150
Oxygen over pressure (kPa)	1241.06	1103.16
Pulp density(g/l)	100	150
Acid concentration(g/l)	50	40

and rhenium. As mentioned before, optimum conditions for the time, temperature and oxygen pressure were ineligible.

Considering this fact and by using Minitab prediction tool, it was predicted that at  $t=60$  min,  $P_{O_2}=965.27$  kPa and nitric acid concentration of 40g/l, the reaction of the molybdenite and nitric acid can be successfully completed.

To examine the validity of achieved results for modified optimum conditions (predicted by Minitab software) of molybdenum and rhenium pressure leaching, an experiment was performed at  $t=60$  min,  $T=150$  °C,  $P_{O_2}=965.276$  kPa, Pulp density of 100g/l and nitric acid concentration of 40g/l. The results of this experiment (illustrated in Table 8) revealed good fitting between

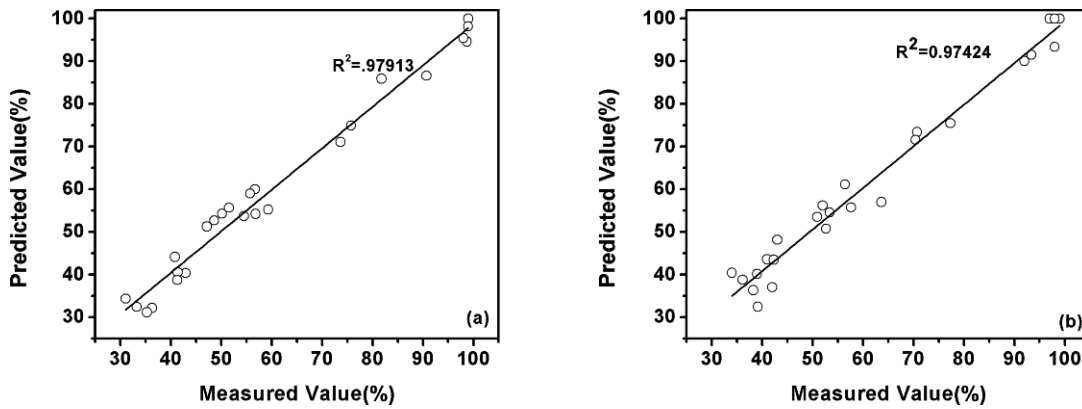


Fig. 2. Predicted versus measured value plots for: a) molybdenum and b) rhenium.

Table. 6. Statistical Analysis of Variance (ANOVA) for the molybdenum( $C.F=80133.24$ )

Parameters	f	S	F ratio	V	P (%)
Time	4	1544.46	2.48	386.11	6.17
Temperature	4	1629.87	2.62	407.47	6.52
Oxygen over pressure	4	568.01	0.91	142	2.27
Pulp density	4	15374.02	24.7	3843.51	61.47
Acid concentration	4	5273.63	8.47	1318.41	21.08
Error	4	622.41	1.00	155.6	2.49
Total	24	25012.4			100

Table. 7. Statistical Analysis of Variance (ANOVA) for the rhenium ( $C.F=72256.25$ )

Parameters	f	S	F ratio	V	P (%)
Time	4	669.07	0.47	167.27	2.51
Temperature	4	785.3	0.56	196.33	2.95
Oxygen over pressure	4	1954.74	1.38	488.69	7.34
Pulp density	4	16189.11	11.45	4047.28	60.8
Acid concentration	4	5615.26	3.97	1403.81	21.09
Error	29	1414.47	1.00	353.62	5.31
Total	24	26628.4			100



predicted and measured values.

For observing the changes, Molybdenite concentrate and precipitated molybdc oxide were subjected to X-ray Diffraction analysis (Figs. 3a and 3b) and SEM imaging (Figs. 4a and 4b), using PHILIPS PW1800 diffractometer and Tescan VegaII scanning electron microscope, respectively.

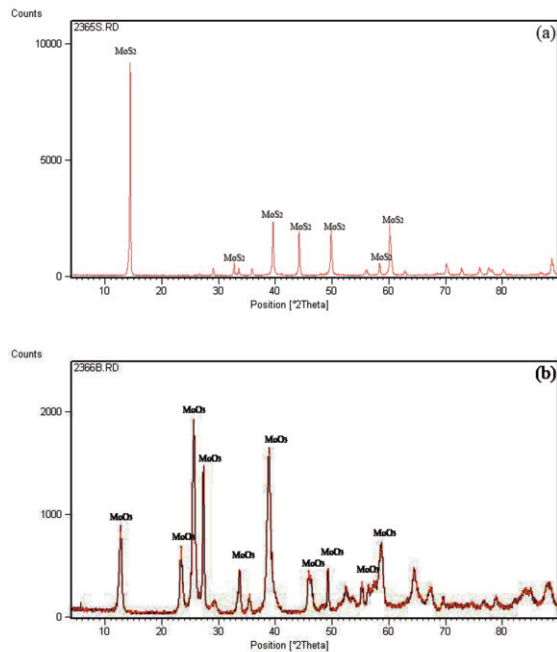


Fig. 3. X-ray diffraction patterns of: a) molybdenite concentrate, b) synthetic molybdc oxide resulted from the experiments.

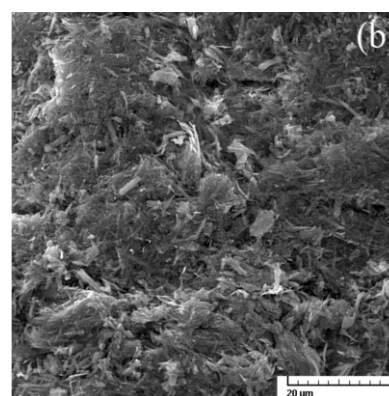
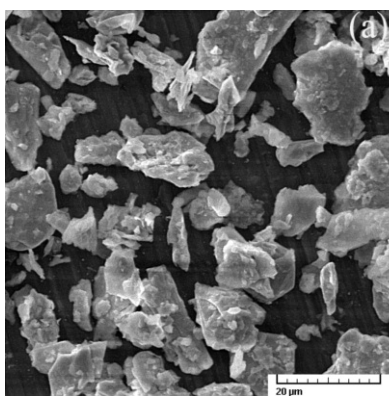


Fig. 4. SEM microstructure of: a) molybdenite concentrate, b) synthetic molybdc oxide resulted from the experiments.

It was found that X-ray diffraction spectrum of the precipitate was approximately similar to synthetic molybdc oxide. Existence of noise in the precipitate pattern can be a result of changes in crystalline structure of synthetic molybdc oxide.

SEM image of precipitated molybdc oxide showed that precipitated molybdc oxide had a fibrous structure. This means that molybdc oxide particles precipitated on previous stable nucleus and grew as same as fiber-like dendritic solidification in casting (Fig. 4b).

Table. 8. Result of pressure leaching at modified optimum conditions

Leaching Conditions	
Time (min)	60
Temperature (°C)	130
Oxygen overpressure (kPa)	965.27
Pulp density (g/l)	100
Acid concentration (g/l)	40
Results	
Rhenium leached (%)	99.99
Molybdenum leached and oxidized (%)	99.36
Sulfur content of remained precipitates (%)	0.2
Concentration of molybdenum in liquor(g/l)	7.6

#### 4. Conclusion

In the current study, optimized conditions for pressure oxidative leaching of molybdenite concentrate in acid nitric media were determined. Using the Taguchi orthogonal array(L<sub>25</sub>) and considering the time, temperature, oxygen pressure, pulp density and acid concentration as controllable parameters, the optimized conditions for pressure leaching of molybdenite were calculated as t=180 min, T=150 °C, P<sub>O<sub>2</sub></sub>=1241.06 kPa, pulp density of 100 g/l and acid concentration of 50 g/l. Moreover, using analysis of variance (ANOVA), it was verified that the pulp density played the most significant role in the process. Nitric acid was the oxidizing agent and oxidation of the molybdenite proceeded only when nitric acid was available in the system. Minitab prediction tool was used to modify the optimum conditions for pressure leaching of the molybdenum and rhenium. When conducting the experiment under modified optimum conditions, the maximum efficiency for molybdenite conversion to soluble molybdic acid and precipitated molybic oxide (99.36%) was obtained. Same performance was observed for the rhenium (99.99%). Thus, the optimum condition for pressure leaching of molybdenum sulfide covered that of rhenium sulfide.

#### Appendix A

##### Equations expressing the ANOVA calculations:

Mean square values

$$V_i = \frac{S_i}{f_i} \quad \text{for factor } i \quad \dots(8)$$

$$V_e = \frac{S_e}{f_e} \quad \text{for error} \quad \dots(9)$$

Percent contribution

$$P_i = S_i \times \frac{100}{S_T} \quad \text{for factor } i \quad \dots(10)$$

$$P_e = S_e \times \frac{100}{S_T} \quad \text{for error} \quad \dots(11)$$

Correction Factor

$$C.F = \frac{T^2}{N} \quad \text{and } T = ((Y_1 - Y_0) + \dots \dots + (Y_n - Y_0)) \quad \dots(12)$$

$$S_A = \sum_{k=1}^L \frac{1}{n_k} \left[ \sum_{i=1}^n (A_{ik} - Y_0)^2 \right] \quad \dots(13)$$

Some of the squares for each factor were calculated as following:

$$S_A = \sum_{k=1}^L \frac{1}{n_k} \left[ \sum_{i=1}^n (A_{ik} - Y_0)^2 \right] \quad \dots(14)$$

$$S_e = S_T - (S_A + S_B + S_C + S_D + S_E) \quad \dots(15)$$

$$f = n - 1 \quad \dots(16)$$

where:

L: number of levels

n<sub>i</sub>, n<sub>k</sub> : number of test samples at level A<sub>i</sub> and A<sub>k</sub> respectively

##### Definitions:

C.F: Correction factor n: Number of experiments

e: Error (experimental) r: Number of repetitions

F: Variance ratio P: Percent contribution

F: Degree of freedom T: Total (of results)

S: Sum of squares f<sub>e</sub>: Degree of freedom of error

f<sub>T</sub>: Total degrees of freedom

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