Evaluation of hydrodynamic factors on column bioleaching of uranium ore

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Abstract

Hydrodynamic factors have been proved to effectively influence the high-performance heap leaching, hence this study evaluates them on column bioleaching of low grade uranium ore. Response surface methodology (RSM) was applied to predict the behavior of effective parameters particle size, irrigation rate, aeration rate and their interactions in the bioleaching process. Obtained results showed that the best model for the recovery of each metal was the quadratic model. The maximum values of uranium recovery at the optimum condition, (d80 5, mm particle size, 0.34, l/m2/min irrigation rate, and 210, l/m3/min aeration rate), were 63.85%. The results from the model and the experimental data show good agreement.

key words

Bioleaching

Uranium ore

hydrodynamic

Response surface methodology

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1. Introduction

The heap leaching process remains limited by low recoveries and long extraction times, Even though it is by now a well-established technology choice in the mining industry [1]. As heap-leach-only operations are becoming more frequent, more attention is required on improving heap performance in terms of rate of extraction as well as total extraction. For this purpose, it is necessary to understand what limits heaps under current operating conditions. Successful heap leach operation requires a thorough understanding of the underlying principles for optimal operation[2].

Uranium is the main contributor for the nuclear fuel industry. Uranium is the most representative actinide element that is of fundamental importance in the nuclear fuel cycle. The nuclear fuel cycle involves several major steps consisting of the leaching of uranium ore in sulfuric acid [3]. Utilizing low-grade uranium ores is a great challenge with the depletion of high-grade uranium ores and the increasing demand for nuclear energy produced from uranium material. Conventional acid heap leaching, which has been widely used in uranium mines, requires large amounts of H_2SO_4 acid and also often brings environmental problems. The microbial leaching in uranium industry has many advantages, such as adaptation to low-grade ores, short leaching cycle, relatively low cost and low contamination [4].

The effective parameters on uranium biological dissolution depends upon the properties of the micro-organisms, ore specifics including surface area of the minerals, particle size, water availability, temperature, pH, redox potential, oxygen and carbon dioxide supply, supply of other nutrients (nitrogen compounds and phosphate) and toxic substances, and formation of secondary minerals [5]. Column leaching operates on the principle of percolator leaching and is used as a model for heap or dump leaching processes, which gives information about what has to be expected in heap or dump leaching and how the leaching conditions can be optimized [6].

Understanding of hydrodynamics and transport phenomena in a porous packed bed is essential for the heap bioleaching. The liquid flow is considered to be governed by both the gravitation and capillary forces. A part of the liquid is holdup between the adjacent void cells. The transport of microbial cells is mainly connected with the leaching solution migration through the porous media [7].

The flowing-liquid holdup corresponds roughly to the liquid collected during drainage and depends primarily and, to a lesser extent, on (1) the fluid physical properties, (2) the liquid flow rate, and (3) the gas flow rate. A substantial change in the flowing liquid holdup is observed when the flow rate is large enough such that air-filled voids begin to pinch off and flooding occurs (Sylvie C. [8].

Liquid flow in heaps is controlled variably by gravitational and capillary forces as a result of the particle size distribution in heaps ranging from sub-millimetre to multiple centimetres [9].

Heap bioleaching performance is dependent on the contacting of the leach solution with the ore bed, hence on the system hydrodynamics[10]. Many authors have reported different hydrodynamic condition in bioleaching of metals. The column characterstics of bioleaching process from different ores are summarized in Table 1.

Conventional methods for optimization involve changing one independent variable at a time while the other variables remain fixed. Statistical optimization reflects the role of each component and interactions among the parameters of the process. Several other advantages of the statistical method are rapidity and the saving of time and manpower. Response surface methodology (RSM) is an efficient strategic experimental tool in which the optimal conditions of a multivariable system are determined.

Because of the lack of the studies on interactions of hydrodynamic factors in column bioleaching, the aim of this study is to evaluate particle size, irrigation rate and aeration rate on uranium recovery from ore, as well as their interactions on the column bioleaching process were also studied using RSM.

2. Material and Methods

2.1. The ore sample and its characterization

A bulk low-grade uranium ore was obtained from the Saghand uranium mine in the center of Iran. The bulk sample was initially crushed by a jaw crusher from the maximum size of 150 mm down to 20 mm. The sample was prepared in 3 particle sizes: d80=5, 10, 15mm. The ore particle size distribution is shown in Table 2. The chemical composition of the uranium ore is given in Table 3. The X-ray diffractometer (XRD, D8-Advance, Bruker AXS) was used to qualitatively analyze the mineral phases at room temperature. The analysis

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Table 1. List of various column characteristics of bioleaching process

Height (cm)	Diameter (cm)	Ore size (mm)	Irrigation rate	Aeration rate	Reference
33	5.6	15	0.03-1 h^{-1}	15 ml/sec	[11]
50	6.5	12.5	0.66 l/h	35 l/min.m^2	[12]
58	13	5.5	50 ml/min	1.75 $Nm^3/m^2/h$	[13]
600	32	12.5>	6 $L/m^2/h$	0.69 NL/min	[14]
58	13	12.5	175 l/h	50 mL/min	[15]
170	25.4	8=d80	5 $l/m^2/h$	1.5 l/min (CO_2 v/v 2.5%)	[16]
100	10	8=d80	10 $l/m^2/h$	9.5 $m^3/m^2/h$	[17]
200	30	15>	10 $l/m^2/h$	0.2-0.3 l/h	[18]
600	15	12.7>	l/m2/h 9.1	1.5 $m^3/m^2/h$	[19]
36	10	8=d80	2,6,18 $l/m^2/h$	2.75 Nm^3/h.ton	[20]
36	10	1>		200 mL/min	[21]
50	10	1>	5 $l/m^2/h$	2 $m^3/m^2/h$ (1% CO_2)	[22]
53	10	12.5>	5 $l/m^2/h$	l/min 1.5 (1% CO_2)	[23]
75	6.5	5>	-	4 l/h	[24]
70	7	6>	-	0.1-0.3 l/h	[25]

results show that major minerals are talc, magnetite, hematite and pyrite. The pyrite content was 5.4%. The mineralogy of ore showed that uraninite (average size 100μm) was the main uranium mineral in the ore (Fig. 1).

2.2. Microorganism and media

Previously isolated bacterium Acidithiobacillus ferrooxidans strain ZT-94 from uranium mine was used for this work. This isolate was grown in modified medium (pH 2) with 20 g/L $FeSO_4.7H_2O$, 2.0 g/l $(NH_4)2SO_4$, 0.5 g/l $MgSO_4.4H_2O$, 0.5 K_2HPO_4, 0.1 g/l KCl and 0.01 g/l $Ca(NO_3)_2.4H_2O$ were incubated in Erlenmeyer flasks of corresponding medium on a rotary shaker at 150 rpm and 30 °C. The cell concentration was about 2.8×107 cell/mL.

2.3. Analysis methods

Samples of leaching solution were regularly withdrawn for measurement of pH, redox potential and concentrations of uranium and iron. Uranium concentration was determined by ICP-OES (Perkin Elmer Optima 2000 DV). The pH value was measured with a pH meter and the redox potential was measured with a platinum electrode with an Ag/AgCl reference electrode (Metrohm 827).

2.4. Column bioleaching experiments

A number of 6 Columns were fabricated from 5 mm thick glass, which was 50 cm high with an internal diameter of 7.5 cm. A plexiglass support plate with multiple holes (ø 1.5 mm) was fixed at the bottom of the column, allowing air to be injected and dispersed uniformly over the particle bed in the column. The leaching solution was passed through the ore sample by gravity and collected in PVC container. The leaching solution wasn't re-circulated.

The leaching experiments were carried out at ambient temperature. Agglomeration of ore was done by solution content 1.3M H_2SO_4 with humidity percentage of 6, 5.5 and 5 for particle size 5, 10 and 15 mm respectively. 3Kg of The agglomerated ore was loaded in the columns.

2.5. Design of experiments and optimization by response surface method

A standard response surface methodology (RSM) based on Box-Behnken design was utilized for the statistical optimization of experimental conditions. A Box-Behnken design with three variables was used to determine the response pattern and then to establish a model. This design led to study the effects of three factors in a single block of 17 sets of test conditions that were generated with 5 replicates of the central point. As presented in Table 3, three independent variables used in this work were particle size, irrigation rate and aeration rate, which were prescribed into three coded levels(−1, 0, +1) for each set of experiments. Regarding the applied design, seventeen combinations were exe-

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Table 2 Particle size distributions of ore.

Screen size (mm)	Cumulative passing (%)		
	d80=15 mm	d80=10 mm	d80=5 mm
19.000	97.17	-	-
16.000	81.76	-	-
12.700	68.88	95.69	-
9.510	59.48	77.05	-
8.000	48.16	65.97	95.01
4.760	37.75	53.90	78.48
2.380	29.72	40.12	58.84
1.000	26.67	29.78	42.77
0.707	22.77	25.97	37.23
0.500	18.78	21.66	30.98
0.297	13.12	16.49	23.48
0.210	7.80	10.34	14.55
0.105	3.23	3.69	4.91
-0.105	0	0	0

cuted and the mathematical relationship between the three independent variables were approximated by the second order polynomial model (eq 1):

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3}\sum_{j=1}^{3} \beta_{ij} X_i X_j + \sum_{i=1}^{3} \beta_{ii} X_i^2 \quad (1)$$

where Y is the predicted response (uranium recovery); X_i's are the independent variables that are known for each experimental run. The parameter β_0 is the model constant; β_i is the linear coefficient; β_{ii} are the quadratic coefficients; and β_{ij} are the cross-product coefficients. The experimental design and the statistical analyses of the data were performed using the Design-expert 7.0 (State-Ease, Inc., Minneapolis, MN, USA). Analysis of variance (ANOVA) was used to estimate the statistical parameters. The extent of fitting the experimental results to the polynomial model equation was expressed by the determination coefficient R^2. F-test was used to estimate the statistical significance of all terms in the polynomial equation with-

Table 3 Range and coded levels of independent variables used in the Box-Behnken design

Composition	U	Fe_2O_3	SiO_2	MgO	CaO	Al_2O_3	K_2O	Na_2O	P_2O_5
Content (%)	0.023	42.05	26.39	22.22	2.35	2.21	0.63	0.11	0.71

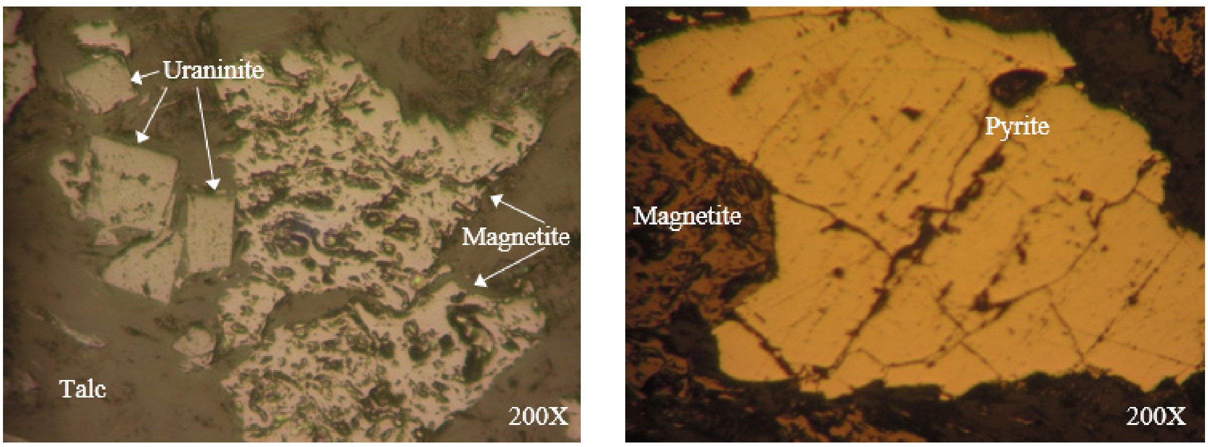

Fig. 1. Pictures of the mineral constituents of Saghand uranium ore under optical microscope

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Table 4 The Box-Behnken experimental design with three independent variables

No.	Variables	Units	Symbol code	Coded levels		
				-1	0	1
1	Particle size	d80, mm	A	5	10	15
2	Irrigation rate	$l/m^2/min$	B	0.15	0.40	0.65
3	Aeration rate	$l/m^3/min$	C	200	500	800

in 95% confidence interval.

2.6. Validation experiments

Three independent bioleaching experiments were conducted applying the optimal conditions obtained from the Box-Behnken design to realize whether the results from recovery models are in accordance with the experimental ones or not.

3. Results and discussion

The data obtained from the column bioleaching experiments were statistically analyzed to identify the significance of main effects as well as the interaction effects. Each factor was varied at three different levels while the other parameters were kept constant. The relative importance of particle size, irrigation rate and aeration rate for recovery of uranium were investigated using Box-Behnken design. Table 4 represents the design matrix of the variables together with the experimental results. Analysis of variance (ANOVA) of the experimental results was carried out to determine the significant and insignificant effects and thus obtain the best possible statistical model.

3.1. Statistical analysis

Eq. (1) was obtained from the 17-Columns runs. It was found that the models could best fit the experimental data. By applying multiple regression analysis to the experimental data, the experimental results of the Box-Behnken were fitted with a qua-

Table 5 Statistical results of the ANOVA for response surface quadratic model.

Run	Factors			Response (%)
	A: Particle size (d80, mm)	B: Irrigation rate ($l/m^2/min$)	C: Aeration rate ($l/m^3/min$)	
1	5	0.15	500	56.26
2	10	0.40	500	56.06
3	10	0.15	200	59.48
4	10	0.40	500	54.05
5	5	0.40	200	62.44
6	5	0.40	800	49.95
7	5	0.65	500	58.45
8	15	0.40	800	45.85
9	10	0.40	500	58.01
10	15	0.65	500	46.20
11	15	0.15	500	53.75
12	10	0.40	500	58.90
13	10	0.65	800	50.55
14	15	0.40	200	50.95
15	10	0.65	200	54.15
16	10	0.40	500	55.45
17	10	0.15	800	47.85

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dratic polynomial model. The empirical relationship between uranium recovery and the three test variables in coded terms obtained by the application of RSM are given by the following equation (eq 2):

$$\text{Recovery}(\%): U=56.49-3.79A-B-4.10C-W.44AB+1.85AC+2.01BC-1.77A^2-1.06B^2-2.43C^2 \quad (2)$$

The statistical significance of the model equations (Eq. (2)) and the model terms were assessed by the F-value for analysis of variance (ANOVA) (Table 5). The 'Prob > F' value for the model was <0.0007 (p-value <0.05), which indicates that the model was statistically significant with a confidence interval of 95.00%. Besides, the coefficient of determination (R^2) that shows the quality of fit of the second-polynomial equations was 0.9546 for U recovery which implies that the model was suitable for sufficient representation of the real relationship among these variables.

Eq. (2) express that among three factors (A, B, C) in giving ranges all have the negative linear effect on column bioleaching of uranium ore. The most effective factor is aeration rate and after that particle size and irrigation rate respectively. The model also shows the negative effect interaction between variables A (particle size) and B (irrigation rate) and positive effect interaction between variables B and C (aeration rate) and also A and C.

Based on Prob>F, significant model terms are: A, C, AB, AC, BC and C^2. Adequate precision measures the signal to noise ratio. The desired value is greater than 4; this value was found to be suitable to support the fitness of the model. Moreover, a low value for the coefficient of variation (CV) (less than 10) indicates that variation in the mean value is quiet and accuracy is good (Table 5).

It should be noted that the polynomial model are reasonable approximations of the true functional relationship over relatively small regions of the entire space of independent variables. Fig. 3 represents the predicted vs. actual metal recovery percentage. Actual values are the measured response data for a particular run and the predicted values are evaluated using the polynomial equations generated as model (Eq. (1)). The accumulation of the points around the 45˚ line indicates a satisfactory correlation between the experimental data and the predicted values which means the models is appropriate for predicting the responses (Fig. 3).

3.2. Contour plots and 3D response surfaces

The complete understanding of simultaneous effects of two factors on column bioleaching by generation of three dimensional response surfaces and contour plots is accessible.

Figs. 4–6 show the three-dimensional response surfaces of uranium recovery (%) as well as contour plots of the relationship between different parameters at the optimized values. Due to the meaningful effects of the interactions on metals recovery, the axes in these plots were selected as the interaction statements with p-values <0.05 and the largest absolute coefficients in the fitted mod-

Table 5 Statistical results of the ANOVA for response surface quadratic model.

Statistical result	Recovery U
Model F-value	16.34
Model Prob>F	0.0007
Lack of fit F-value	0.15
Lack of fit P-value	0.9251
R-Squared	0.9546
Adj R-Squared	0.8962
Pred R-Squared	0.8631
Std. Dev.	1.56
Mean	54.02
C.V %	2.89
Adeq Precision	13.193

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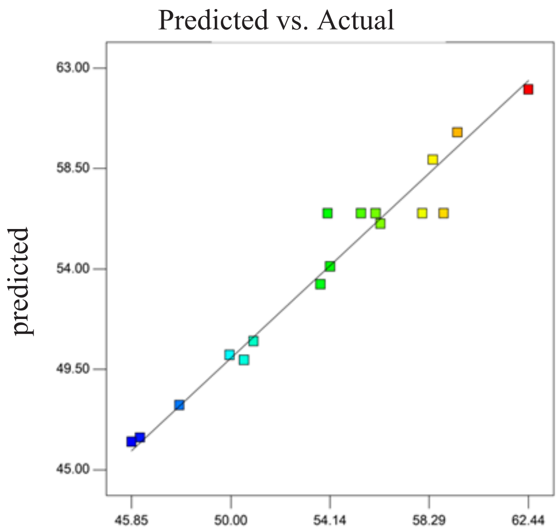


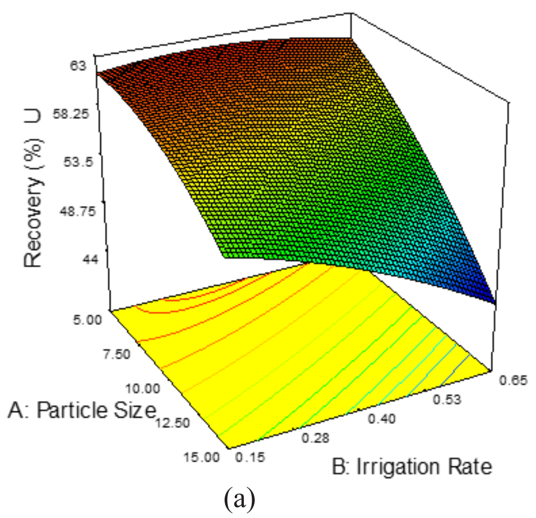
Fig. 2. Scatter graph of the predicted response values versus the actual response values for uranium recover (%).

el. According to the models, interactions between variables have significant effects on the responses; therefore, results were presented and discussed in terms of interactions. Irrigation rate and particle size pose major interaction with coefficient more than others and have negative effect on uranium recovery (Eq. (1)).

The movement of solution through the ore has important impact on efficiency of bioleaching operations, because the solution transport lixiviates into and metal ions out of the column. Maximizing solution contact and minimizing preferential flow leading to significant bypassing of ore by the leach solution are crucial for enhancing leaching efficiency and ore recovery. But the flow behavior in the column ore is very complex due to a wide range of particle size, complex configuration of the heap structure, and interaction between fluid and particles[26].

From Fig. 4, it is evident that, in the irrigation rate range from 0.3 to 0.4 l/m^2/min, a decrease in particle size causes an increasing trend for the recovery of uranium from ore. There was a nonlinear relationship between uranium recovery, irrigation rate and particle size. Increasing irrigation rate decreases selectivity in bioleaching, Incremination of bioleaching rate and decomposition of agglomerate particles, and decreasing irrigation influences lead to bioleaching selectivity upsurge, slower bioleach rate, slower pH breakthrough, acid limitations, and excessive ferric precipitation [27]. As could be seen from Fig.4, increasing of irrigation rate to optimum point increases uranium recovery; but, after that point decrease recovery.

Interaction plots between the aeration rate and irrigation rate on uranium recovery are illustratesd in Fig. 5, where the particle size was constant. The aeration rate decreases and irrigation rate increas-



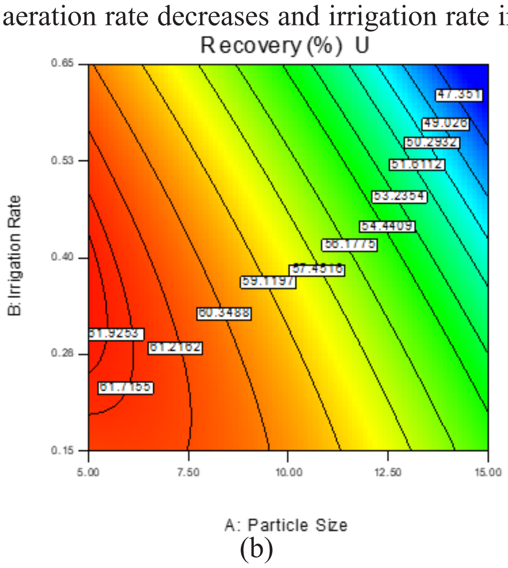


Fig. 3. (a) Contour plots and (b) response surface for interactive effect of particle size and irrigation rate at constant aeration rate for U-recovery.

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es from 0.15 l/m^2/min to optimum point, uranium recovery increase to maximum value. The relation between the irrigation rate, aeration rate and uranium recovery is nonlinear. When liquid holdup in column decrease, metal concentration increase at leach solution [8]. Maximum liquid holdup occurs at minimum irrigation and maximum aeration rate, as depicted in Fig. 5. At this point uranium recovery is minimum. It means that uranium extraction reactions were done at optimum liquid holdup.

The reaction (1) showed that oxygen is important for bacterial oxidation of sulfide minerals. Fe^{3+} Supply, produced by bacterial oxidation of Fe^{2+}, causes leaching of uranium from ore (reactions (2) and (3)) [28,29]. As the diffusion rate of oxygen in water is several orders of magnitude less than in air, keeping a heap under unsaturated conditions could benefit the transportation of oxygen[30].

Increased irrigation causes the flow velocity and water content in the column to increase, and reduces the air-filled porosity. An optimum irrigation rate exists to provide sufficient both reagents and oxygen for the leaching reaction. Other factors that can influence flow conditions include decrepitation of the substrate, compaction, precipitation of salts from the liquid phase and transport of fine particle[26].

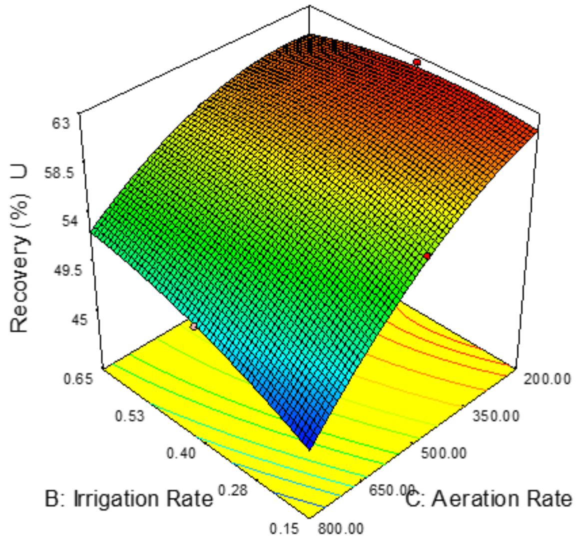
Fig. 6a and b shows the second-order contour and three-dimensional surface plot for the U recovery percentage depending on the aeration rate and particle size when the irrigation rate was fixed. A nonlinear relationship is observable between uranium recovery and these two parameters. The maximum recovery can be obtained at the lowest particle size and aeration rate.

3.3. Determination of optimum conditions

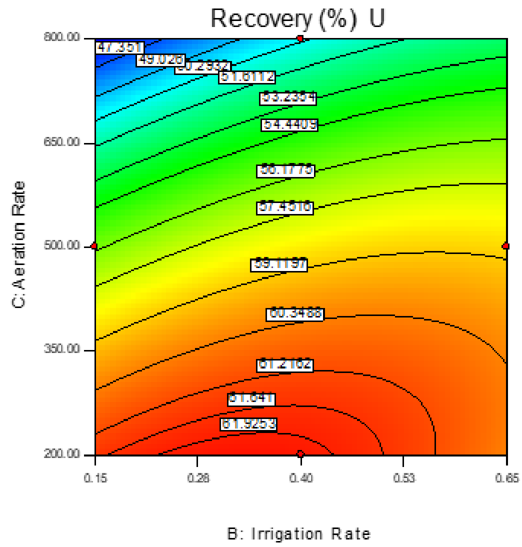
In the numerical optimization, a minimum and a maximum level must be provided for each parameter. The goals are combined into an overall desirability function. Desirability is an objective function that ranges from zero outside of the limits to one at the goal. The program seeks to maximize this function. By starting from several points in the design space, chances of finding the best local maximum are improced. Level of all parameters within the range of investigation was set for maximum desirability. The best local maximum was found to be at the obtained value of desirability, showing that the estimated function may present the experimental model and desired conditions. It should be noted that the goal of optimization is to find a good set of conditions that will meet all of the goals. The optimum conditions proposed by the model were particle size d80=5mm, irrigation rate 0.34 l/m^2/min and aeration rate 210 l/m3/min, with which the maximum uranium recovery of 62.10% was achieved. These values are all in agreement with the results obtained from the three-dimensional surface plots.

3.4. Confirmatory experiment

To test the validity of the optimized conditions given by the model, an experiment was carried out with the parameters suggested by the model. Table 6 presents the results of the experiment conducted at the optimal conditions, highlighting the verification experiment. The predicted values from fitted correlations were in close agreement with confirmation experiment results at a 95% confi-



(b)



(a)

Fig. 4. (a) Contour plots and (b) response surface for interactive effect of irrigation rate and aeration rate at constant particle size for U-recovery.

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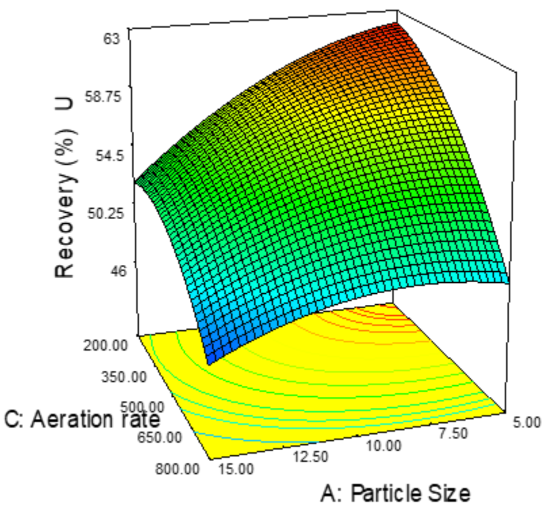
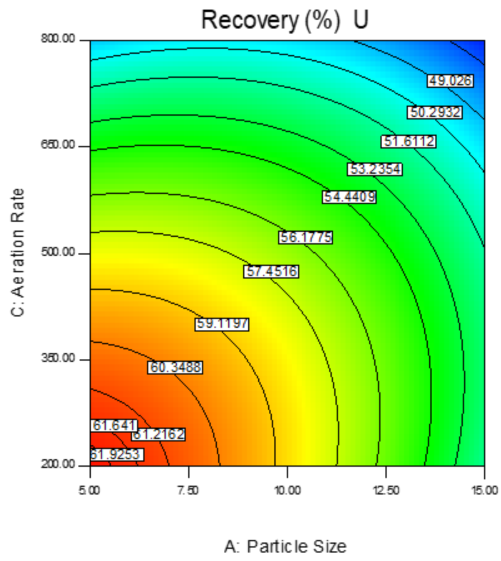




(b) (a)

Fig. 5. (a) Contour plots and (b) response surface interactive effect of aeration rate and particle size at constant irrigation rate for U-recovery.

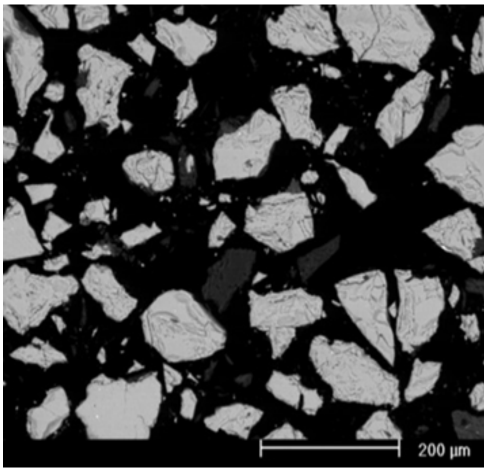
dence interval. The analysis results indicated that the experimental values were in good agreement with the predicted values, and hence, the model is successful in predicting responses. Under these conditions, the experimental value for uranium recovery was found to be 63.85% at 19 days. These results confirmed the validity of the model, and the experimental values were determined to be quite close to the predicted values. The SEM photomicrographs of the uranium ore before and after bioleaching (Fig. 7a and b), reveal that layer performed on particles. Microprobe analysis showed that produced layer is K-Jarosite.

4. Conclusion

Column bioleaching of uranium ore by focusing on hydrodynamic effects using indigenous strain of Acidithibacillus ferrooxidans was studied. Hydrodynamic factors such as irrigation rate, aeration rate and particle size were examined to evaluate on uranium recovery from low grade ore using RSM. The findings in this study identified the development of mathematical model for process simula-

Table 6 Point prediction and verification of the responses at the optimal conditions.

Response	Prediction	Confirmation experiment (%)	95% CI low	95% CI high
U	62.10	63.85	58.91	65.22



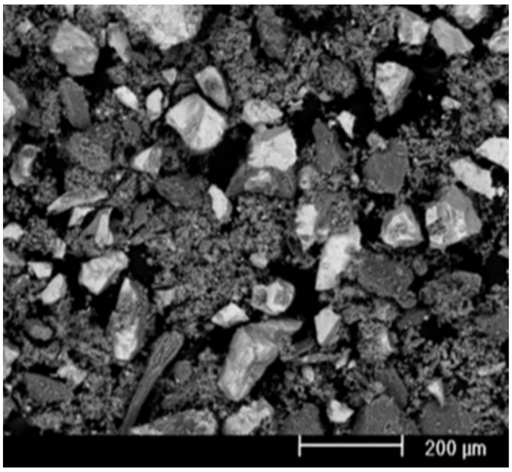


Fig. 6. SEM image (100X magnification) of (a) uranium ore before bioleaching and (b) bioleached ore in column after 19 days at optimal condition.

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tion based on statistics can be useful in predicting and understanding the effects of experimental factors. Results showed that the best model for the recovery of metals was the quadratic model. It was found that at the optimum condition of particle size d80=5mm, irrigation rate 0.34 l/m2/min, and aeration rate 210 l/m3/min, the maximum bioleaching of uranium was 63.85%.

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