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Optimization of microwave-assisted manganese leaching from electrolyte manganese residue

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Abstract: The process optimization of microwave assisted leaching of manganese from electrolytic manganese residue (EMR) was conducted. The Box-Behnken design (BBD) was utilized to determine the number of experiments as well as to assess the effect of the main leaching parameters, including the reaction temperature, reaction time, concentration of sulfuric acid and dosage of citric acid. A quadratic model was found to best fit the experimental data and was utilized to optimize the process parameters to maximize the percentage manganese recovery. 3-D response surface plots and contour plots were generated utilizing mathematical models to understand the effect of variables as well as to identify the optimal conditions. The optimum conditions of microwave assisted leaching were: temperature of 76°C, time of 55 min, H_2SO_4 concentration of $0.76 \text{ mol}\cdot\text{L}^{-1}$, dosage of citric acid of 3.51 mg/g. Under these conditions, the percentage manganese recovery higher than 90% could be achieved.

Keywords: electrolytic manganese residue; microwave assisted leaching; response surface methodology; manganese

1 Introduction

Electrolytic manganese residue (EMR) is a one of the industrial solid waste generated in manganese hydrometallurgical processing, usually contains manganese and ammonia nitrogen [1]. Since 2000, China has become the largest electrolytic manganese metal producing country, representing 98% of the world's manganese metal output in 2017. Generally, producing 1 ton

of manganese would generate 6-10 tons of EMR depending on the grade of manganese ore [2]. In China, currently more than 10 million tons per year of EMR are being discarded as solid waste pile up which result in massive land resources occupied and serious environmental risks. There is an increasing attention on recycling valuable resources from electrolytic manganese residue to overcome environmental concerns in recent years. As high as 4-7% w/w of manganese element remained in EMR [3], therefore the development of efficient treatment technologies for the recovery of manganese from EMR is essential [4].

The EMR contains gangue minerals which engulf a proportion of the manganese compounds [5], thereby reducing the extraction efficiency and therefore these require removal. Recent efforts to improve this include hot sulfuric acid leaching [6], bioleaching [7,8], intensified leaching by electric field [1,3,9], ultrasonically assisted leaching [10,11]. However these are typically associated with shortcomings including long operating times, high costs, complicated processes etc. for manganese recovery.

The application of microwave assisted leaching technology in metallurgy and mineral extraction has been widely reported over the past decades [12]. Selective mineral liberation, controllable and faster heating process, are the main driving force for microwave heating being attractive in extractive industries, usually considered as an efficient and greener technique [13,14]. Studies on extraction of metals from industrial residues using microwave energy have shown it to be significantly faster and in some cases, resulted in enhanced metal dissolution as compared to conventional heating technologies [15-18]. However [19], enhanced extraction of manganese from EMR using a combination of microwave irradiation and citric acid leaching was rarely reported.

At present, the most commonly used experimental design methods are orthogonal design and uniform design, both of which adopt linear mathematical model to fit data, requiring less experiments but poor predictability. Response Surface Methodology (RSM) is by far the most popular statistical and mathematical tool since it has some functions include experimental design, modeling, model detection, statistical analysis and so on, the optimum level of each factor and interactions among parameter can be identified through RSM [20]. There are

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three types of response surface design methods, including Central Composite Design, Box-Behnken Design and Plackett-Burman Designs. In particular, the Box-Behnken Design (BBD) is used widely in establishing the second-order RSM, and is one the most popular experimental designs used for process variables [21].

Thus, in order to improve leaching efficiency and to reduce sulfuric acid consumption, the optimization of Mn extraction from EMR using microwave assisted leaching with citric acid using RSM is attempted in the present work. Parameters such as temperature, duration, H₂SO₄ concentration, dosage of citric acid were assessed using statistical design Box-Behnken method.

2 Materials and methods

2.1 Materials

The EMR used in this work was supplied from Guizhou Wuling Manganese Industry Co. Ltd., and before use its composition was analysed by X-ray fluorescence (XRF, XRF-1800, Shimadzu, Japan), with the results presented in Table 1. The results show the main constituents to be SiO₂, SO₃, Al₂O₃, CaO and Fe₂O₃ amounting to approximately 98% of the total composition.

Figure 1 shows the XRD pattern of the EMR (D/Max 2200, Rigaku, Japan), indicates presence of quartz (SiO₂),

gypsum (CaSO₄·2H₂O), groutite (Mn³⁺O(OH)), ammonium iron sulfate (NH₄)₃Fe(SO₄)₃, jacobsite (MnFe₂O₄) as the major minerals. The particle size distribution of the EMR is presented in Figure 2 (Master Sizer 2000, Malvern, UK.). It can be seen the average particle size of EMR is 21 μm and that 90% of the particles are smaller than 47 μm while 10% are smaller than 10 μm.

2.2 Experimental procedure and apparatus

The leaching tests were carried out in a self-made microwave reactor which had a temperature and stirring speed control system (Figure 3).

Figure 2: Grain size distribution curves of EMR.

Table 1: The main constituents of EMR used in this study.

Compound	SiO ₂	SO ₃	CaO	Al ₂ O ₃	MnO	Fe ₂ O ₃	K ₂ O	MgO
Weight%	29.74	29.59	14.71	6.86	6.47	5.85	2.72	1.86

Figure 1: X-ray diffraction pattern of EMR.

Figure 3: Schematic diagram of microwave reactor (1 – reflux condenser, 2 – three-necked flask, 3 – mechanically controlled stirrer, 4 – stirring controller, 5 – digital thermometer, 6 – LCD screen, 7 – temperature setting knob, 8 – power switch, 9 – emergency switch, 10 – microwave power setting knob).

First, the sulfuric acid solution was poured into a three-necked 500 mL flask reactor. Then, a certain amount of EMR sample was added to it. Following this, the flask was placed into the microwave reactor and the temperature raised to the desired value. The agitation speed and microwave power were kept constant at 300 rpm and 200 W respectively for all experiments. After the required duration was met and the solid and liquid separated, the concentration of manganese in filter liquor was determined by atomic absorption spectrometry (Z2000HITACHI). The percentage of manganese leached (E_m) was calculated according to the following equation:

$$E_m = cV/M_0 \times 100\% \quad (1)$$

where c and M_0 are the mass of Mn in the leaching solution and the original sample, respectively; V is the volume of the leaching solution after filtration.

2.3 Experimental design

At present work, four important parameters such as leaching temperature (X_1 , °C), leaching duration (X_2 , min), H_2SO_4 concentration (X_3 , mol·L⁻¹), and dosage of citric acid (X_4 , mL/g) were chosen as the independent variables. The low, middle, and high levels of each variable were identified as -1, 0, and +1 (Table 2).

The above stated upper and lower limits of the variables were selected based on preliminary experiments in addition to information gathered from literature sources. In order to evaluate the effects of independent variables on percentage of Mn extracted, batch tests were carried out. The coded values of the four significant parameters were generated based on Eq. 2:

$$x_i = \frac{X_i - X_0}{\Delta X_i} \quad (2)$$

Table 2: Levels and codes used in Box-Behnken design.

Variable	Symbol		Coded level		
	Uncoded	Coded	Low	Center	High
			-1	0	1
Temperature (°C)	X_1	A	40	60	80
Time (min)	X_2	B	20	40	60
H_2SO_4 concentration (mol·L ⁻¹)	X_3	C	0.2	0.5	0.8
Dosage of citric acid (mg/g)	X_4	D	2	6	10

where x_i is defined to be a dimensionless value of an independent variable, X_i is the real value of variable, X_0 is the real value of an independent factor at the center point, and ΔX_i is the step change.

The statistical models and data analysis were given using Design-Expert 8.0.6 software (trial version). The design is depended on the combination of 2^k factorial design with an incomplete block design. Total numbers of experiments required in a Box-Behnken design can be calculated according to the Eq. 3 [21]:

$$N = 2k(k - 1) + cp \quad (3)$$

where N is the number of experiments, k is the factor number ($k = 4$), and (cp) is the replicate number of the central point. It resulted in 24 experiments with three repetitions at the center point to estimate the pure error. The generalized form of model equation relating the X 's and Y is given by Eq. 4:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i=1}^k \sum_{j=1}^k \beta_{ij} X_i X_j + \dots + e \quad (4)$$

where Y is the response, β_0 is a constant coefficient, X_i and X_j are the input factors, β_i , β_{ii} and β_{ij} are linear, quadratic and interaction coefficients, respectively, e is the random error. The quality of the fit of the model would be evaluated by ANOVA.

3 Results and discussion

3.1 Data analysis

The number of experiments generated utilizing Box-Behnken design are listed in Table 3, which comprehensively provides the process conditions at which experiments were conducted along with the outcome of the experiment 'Percentage Mn recovery'.

Different empirical models were attempted to relate the process variables and independent variable subjecting it to statistical analysis. Among the models tested the quadratic model was found to be the best and the regression model is expressed as Eq. 5:

$$Y = -22.02 + 1.86 X_1 + 0.62 X_2 + 64.37 X_3 + 0.15 X_4 + 1.79 \times 10^{-3} X_1 X_2 + 0.68 X_1 X_3 + 0.03 X_1 X_4 + 0.37 X_2 X_3 + 0.01 X_2 X_4 + 0.24 X_3 X_4 - 0.02 X_1^2 - 9.34 \times 10^{-3} X_2^2 - 95.29 X_3^2 - 0.13 X_4^2 \quad (5)$$

Table 3: Box-Behnken experimental design and response value.

Std No.	Coded level of variables				Actual level of variables				Observed recovery (%)
	A	B	C	D	X_1	X_2	X_3	X_4	
1	-1	-1	0	0	40	20	0.5	6	65.02
2	1	-1	0	0	80	20	0.5	6	82.53
3	-1	1	0	0	40	60	0.5	6	73.23
4	1	1	0	0	80	60	0.5	6	93.61
5	0	0	-1	-1	60	40	0.2	2	67.95
6	0	0	1	-1	60	40	0.8	2	83.18
7	0	0	-1	1	60	40	0.2	10	72.67
8	0	0	1	1	60	40	0.8	10	89.04
9	-1	0	0	-1	40	40	0.5	2	69.89
10	1	0	0	-1	80	40	0.5	2	84.77
11	-1	0	0	1	40	40	0.5	10	70.42
12	1	0	0	1	80	40	0.5	10	93.66
13	0	-1	-1	0	60	20	0.2	6	65.54
14	0	1	-1	0	60	60	0.2	6	69.13
15	0	-1	1	0	60	20	0.8	6	78.65
16	0	1	1	0	60	60	0.8	6	91.08
17	-1	0	-1	0	40	40	0.2	6	64.24
18	1	0	-1	0	80	40	0.2	6	71.58
19	-1	0	1	0	40	40	0.8	6	69.22
20	1	0	1	0	80	40	0.8	6	92.78
21	0	-1	0	-1	60	20	0.5	2	77.05
22	0	1	0	-1	60	60	0.5	2	85.32
23	0	-1	0	1	60	20	0.5	10	80.57
24	0	1	0	1	60	60	0.5	10	92.04
25	0	0	0	0	60	40	0.5	6	88.97
26	0	0	0	0	60	40	0.5	6	88.08
27	0	0	0	0	60	40	0.5	6	89.91

where Y is the percentage Mn extraction, A is the reaction temperature, B is the reaction time, C is the H_2SO_4 concentration, D is the dosage of citric acid.

The adequacy of model to utilize the statistical methods was established by using diagnostic plot (normal % probability versus studentized residuals, studentized residuals versus run number). The probability plot authenticates data to be considered as of normal distributed while the studentize plot helps to identify the outliers.

Figure 4a shows all the data falling on line except one authenticating that the sample data is from a normally distributed population. Figure 4b shows possibility of the one of the data could be an outlier. Figure 4c authenticates the validity of the model equation (Eq. 5), as the model prediction is very close to the experimental data [22]. The adequacy of the model was investigated using the sequential model sum of squares and the model summary statistics are illustrated in Table 4.

As seen from Table 4, the value of regression coefficient for quadratic model is 0.9893. The R^2_{adj} and

predicted R^2 are 0.9767 and 0.9404 for the quadratic model, respectively. The high R^2 shows the proximity of the model prediction to the experimental data. In general, the difference between adjusted R^2 and predicted R^2 is considered to be good fitness within the range of 0~0.200 for the model [23]. At present work, the difference between the predicted R^2 and the adjusted R^2 for the given quadratic mode is only 0.0363. Adequate precision is a crucial criterion to judge the signal to noise ratio. A ratio greater than 4 is preferable, and the ratio of 29.122 (adequate precision) indicates an adequate signal [24]. Based on all the favorable facts detailed above it can be concluded that the model adequately fits the experimental data.

Table 5 shows the analysis results obtained from the ANOVA test for fitting quadratic model. The Model F-value 78.98 implies that the model is significant. There is only a 0.01% chance that such a large "Model F-value" will appear due to noise.

The significance of each coefficient was investigated based on F-test and P-test also as shown in Table 5. According to the F values of A , B , C and D , the influence

Figure 4: Diagnostic plot the model for manganese leaching: (a) normal % probability versus studentized residuals, (b) studentized residuals versus run number, (c) predicted versus actual data.

Table 4: Adequacy of the model.

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value Prob > F	Remarks
Mean vs Total	1.712×10^5	1	1.712×10^5	17.46	-----	
Linear vs Mean	1999.50	4	499.87	0.55	< 0.0001	
2FI vs Linear	107.73	6	17.95	52.48	0.7629	
Quadratic vs 2FI	493.80	4	123.45	1.37	< 0.0001	Suggested
Cubic vs Quadratic	20.68	8	2.58	----	0.4042	Aliased
Residual	7.55	4	1.89	----		
Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	5.35	0.7605	0.7169	0.6723	861.49	
2FI	5.71	0.8015	0.6774	0.5629	1149.15	
Quadratic	1.53	0.9893	0.9767	0.9404	156.72	Suggested
Cubic	1.37	0.9971	0.9813	0.6766	850.18	Aliased

Table 5: Regression analysis of BBD criterion data for leaching of manganese.

Source	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value Prob. > F	Significance
Model	2601.03	14	185.79	78.98	< 0.0001	Significant
A	952.48	1	952.48	404.90	< 0.0001	Significant
B	252.54	1	252.54	107.36	< 0.0001	Significant
C	718.27	1	718.27	305.34	< 0.0001	Significant
D	76.20	1	76.20	32.39	0.0001	Significant
AB	2.06	1	2.06	0.88	0.3679	
AC	65.77	1	65.77	27.96	0.0002	Significant
AD	17.47	1	17.47	7.43	0.0184	Significant
BC	19.54	1	19.54	8.30	0.0138	Significant
BD	2.56	1	2.56	1.09	0.3174	
CD	0.32	1	0.32	0.14	0.7166	
A ²	231.62	1	231.62	98.46	< 0.0001	Significant
B ²	74.50	1	74.50	31.67	0.0001	Significant
C ²	392.28	1	392.28	166.76	< 0.0001	Significant
D ²	24.40	1	24.40	10.37	0.0073	Significant
Residual	28.23	12	2.35	-----	-----	
Lack of Fit	26.55	10	2.66	3.17	0.2635	Not significant
Pure Error	1.67	2	0.84	-----	-----	
Cor Total	2629.26	26	-----	-----	-----	

Table 6: Significance rank of different factors on manganese extraction under microwave heating.

Rank	Parameters
1	Temperature
2	H ₂ SO ₄ concentration
3	Time
4	Dosage of citric acid

degree of the four variables on manganese extraction by microwave assisted leaching are illustrated in Table 6.

A values of “Prob > F” less than 0.0500 imply proposed model is significant, while a values greater than 0.1000 indicate the proposed model is non-significant [25]. In this case A, B, C, D, AC, AD, BC, A², B², C², D² are significant model terms. It indicates the linear effects and square effects of temperature, H₂SO₄ concentration, dosage of citric acid and time are significant. In addition, the interactive effects of temperature and H₂SO₄ concentration (P = 0.0002), temperature and dosage of citric acid (P = 0.0184) and time and H₂SO₄ concentration (P = 0.0138) have an adequate influence on the extraction of manganese by microwave assisted leaching. Compared to the value of pure error, the “Lack of Fit F-value” of 3.17 indicates that the Lack of Fit is not significant. At the same time, there is a 26.35% chance that a “Lack of Fit F-value” this large could appear owing to noise, non-significant lack of fit is desired to ensure the high quality of the fit of the model [26].

3.2 Contour plots and response surface

The effects of variables on the manganese leaching efficiency is assessed by plotting 3D surface curves against any two independent variables, while keeping other variables at their center value. The 3D response surface plots and the contour plots from the interactions between the variable are shown in Figures 5 and 6.

Figure 5a demonstrates the combined effect of temperature with reaction time. It is clear from Figure 5a that % leaching increases significantly with increasing temperature. An increase in temperature from 40°C to 80°C increased the percentage leaching remarkably from 65.02% to 93.61%. The corresponding contour plot is shown in Figure 6a. The combination of the response surface along with the contour plot helps to identify the combination of time and temperature that could provide the maximum percentage recovery. The combined factors reach an asymptote at % Mn recovery higher than 90%. An increase in the percentage Mn extraction with increase in temperature could be attributed to the higher rates of mass transfer due to increased diffusion coefficient. While an increase with increase in time could be due to the time available to complete the extraction.

Figure 5b shows the combined effect of H₂SO₄ concentration and leaching temperature on the % leaching. As can be seen from Figure 5b, manganese recovery increased with increasing both: temperature and H₂SO₄ concentration. However, at higher H₂SO₄

Figure 5: Response surface plots show the effect of temperature (X_1), time (X_2), H_2SO_4 concentration (X_3) and dosage of citric acid (X_4) on % manganese extraction with microwave irradiation.

concentration effect of temperature on the percent recovery of Mn was significant, while at lower H_2SO_4 concentration only a minor effect was observed. An increase in the concentration of H_2SO_4 would increase the rate of reaction and due to reduced the mass transfer resistance aiding diffusion of H_2SO_4 to the site of Mn containing mineral

phase, facilitating higher % leaching. Figures 5b and 6b help to identify the combination of factors that maximize the % Mn recovery.

Figure 5c illustrates the combined effect of citric acid dose with temperature. The effect of increase in citric acid concentration seems to be insignificant at low

Figure 6: Contour plots show the effect between temperature (X_1), time (X_2), H_2SO_4 concentration (X_3) and dosage of citric acid (X_4) on manganese extraction.

temperatures while at high temperatures it exhibits an increase with increase in concentration. Again, Figures 5c and 6c provide the combination factors that offer the maximum of % Mn recovery.

Figures 5d and 5e show the effect of H_2SO_4 and citric acid dose in combination with time on the percentage manganese recovery respectively. The effect of these parameters was discussed in detail earlier. The combination of H_2SO_4 concentration as well as time is found to have significant influence on the percentage Mn recovery while the combination of the citric acid dose and the time has only marginal effect on the percentage Mn recovery. Figures 6d and 6e provide the combination of factors that could maximize the % Mn recovery [27].

The interactive effect of H_2SO_4 concentration with citric acid dose is shown in Figure 5f. It can be observed that an increase in both H_2SO_4 concentration as well as citric acid dose increases the percentage leaching. An increase in the amount of sulfuric acid affects the dissolution rate of manganese in leaching of EMR much more than the citric acid dose. Although the increase in the citric acid dose doesn't seem to increase the extraction rate significantly, the increase in citric acid dose promotes conversion of high valence of Mn into low valence of

Mn, inhibiting the leaching of other gangue minerals. In other words, citric acid offers not only high manganese recovery and selectivity of leaching but also contributes to reduced acid consumption rendering the process more environmentally benign. Above mentioned result is also revealed by the corresponding contour plots presented in Figure 6f.

3.3 Optimization

Although the effect of process variables and combinations thereof that could maximize the percentage Mn recovery is evident from plots developed, the optimal combination of factors that would maximize the percentage recovery may be identified with the help of process optimization. Within the range of parameters assessed in the present work, the process optimizer identified the optimal conditions to maximize the percentage Mn recovery as a temperature of 77°C , time of 55 min, H_2SO_4 concentration of $0.76 \text{ mol}\cdot\text{L}^{-1}$, dosage of citric acid of 3.51 mg/g, corresponding to a maximum Mn recovery of 94.25% with the desirability of 1. The pictorial representation of the optimized process parameters as shown the software is provided in Figure 7.

Figure 7: Ramps of the numerical optimization.

Table 7: The main constituents of EMR (wt%) after leaching with different methods.

Method	SiO ₂	SO ₃	CaO	Al ₂ O ₃	MnO	Fe ₂ O ₃	K ₂ O	MgO
Conventional heating	33.20	26.03	15.68	7.76	3.32	7.34	3.23	1.02
Microwave assisted leaching	37.20	32.40	13.49	7.83	0.44	2.47	3.51	0.74

In order to verify the results of predicted optimum conditions and understand the role of microwave irradiation, the experiments were carried out at identified optimized process conditions under conventional heating and microwave fields, the results are shown in Table 7.

Table 7 revealed that about a half of the manganese retain in the leached residue under conventional leaching process. On the contrary, microwave-assisted leaching ensures the perfect recovery of manganese from EMR. This can be explained that the different microwave absorber character between manganese-bearing mineral and other components result in intergranular and transgranular thermal stress cracking, increasing the contact area between lixiviant and ores. Therefore, microwave-assisted leaching led to the higher recovery efficiency. Moreover, the experiment at identified optimized process condition was carried out in triplicates and the average value of 93.28% for Mn leaching efficiency is sufficiently agree with the predicted value. Hence, this validation confirms the adequacy of the developed quadratic model for Mn leaching.

4 Conclusion

In the present work, the effect of temperature, time, concentration of sulphuric acid and dosage of citric acid were assessed to identify the optimal conditions to maximize the percentage Mn recovery from EMR using BBD of RSM. The proposed quadratic model was used successfully to fit the experimental data, with high values of R^2 and R^2_{adj} of 0.9767 and 0.9404, respectively. ANOVA showed that the effect of temperature and concentration of H₂SO₄ to be the most significant parameters a compare dot time and dosage of critic acid. The best conditions for manganese leaching were identified to be a temperature of 76°C, time of 55 min, H₂SO₄ concentration of 0.76 mol·L⁻¹, dosage of citric acid of 3.51 mg/g with the maximum % Mn recovery being 94.25%. With microwave assisted leaching the manganese leaching rate was about 40% higher than that found with conventional leaching. The high recovery at favorable process conditions economically favor the process for scale-up and eventual commercial adoption.

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References

- [1] Shu J.C., Liu R.L., Liu Z.H., Du J., Tao C.Y., Electrokinetic remediation of manganese and ammonia nitrogen from electrolytic manganese residue. *Environ. Sci. Pollut. R.*, 2015, 22, 16004-16013.
- [2] Shu J.C., Liu R.L., Liu Z.H., Chen H.L., Du J., Tao C.Y., Solidification/stabilization of electrolytic manganese residue using phosphate resource and low-grade MgO/CaO. *J. Hazard. Mater.*, 2016, 317, 267-274.
- [3] Shu J.C., Liu R.L., Liu Z.H., Chen H.L., Tao C.Y., Enhanced extraction of manganese from electrolytic manganese residue by electrochemical. *J. Electroanal. Chem.*, 2016, 780, 32-37.
- [4] Li C.X., Zhong H., Wang S., Xue J.R., Leaching behavior and risk assessment of heavy metals in a landfill of electrolytic manganese residue in Western Hunan, China. *Hum. Ecol. Risk Assess.*, 2014, 20(5), 1249-1263.
- [5] Chen H.L., Differences analysis of minerals compositions and toxicity characteristics between the fresh electrolytic manganese residue and the stockpiling residue. *Journal of Guizhou Normal University (Natural Science)*, 2016, 34, 32-36 (in Chinese).
- [6] Olívia S.H.S., Cornelio F.C., Gilmore A.S., Gláudio G.S., Manganese ore tailing: Optimization of acid leaching conditions and recovery of soluble manganese. *J. Environ. Manage.*, 2015, 147, 314-320.

- [7] Duan N., Zhou C.B., Chen B., Jiang W.F., Xin B.P., Bioleaching of Mn from manganese residues by the mixed culture of *Acidithiobacillus* and mechanism. *J. Chem. Technol. Biot.*, 2011, 86, 832-837.
- [8] Xin B.P., Chen B., Duan N., Zhou C.B., Extraction of manganese from electrolytic manganese residue by bioleaching. *Bioresource Technol.*, 2011, 102, 1683-1687.
- [9] Zhang X.R., Liu Z.H., Wu X.B., Du J., Tao C.Y., Electric field enhancement in leaching of manganese from low-grade manganese dioxide ore: Kinetics and mechanism study. *J. Electroanal. Chem.*, 2017, 788, 165-174.
- [10] Li H., Zhang Z.H., Tang S.P., Li Y.N., Zhang Y.K., Ultrasonically assisted acid extraction of manganese from slag. *Ultrason. Sonochem.*, 2008, 15, 339-343.
- [11] OuYang Y.Z., Peng X.W., Cao J.B., Li Z.P., Deng X.D., Ultrasonic leaching of electrolytic manganese residue with additive. *Environmental Protection of Chemical Industry*, 2007, 27, 257-259 (in Chinese).
- [12] Chang J., Zhang L.B., Yang C.J., Ye Q.X., Chen J., Peng J.H., et al., Kinetics of microwave roasting of zinc slag oxidation dust with concentrated sulfuric acid and water leaching. *Chem. Eng. Process.*, 2015, 97, 75-83.
- [13] Liu B.G., Peng J.H., Zhang L.B., Zhou J.W., Srinivasakannan C., Preparation of V_2O_5 from ammonium metavanadate via microwave intensification. *Russ. J. Non-Ferr. Met.*, 2017, 58, 600-607.
- [14] Pinto I.S.S., Soares H.M.V.M., Microwave-assisted selective leaching of nickel from spent hydrosulphurization catalyst: A comparative study between sulphuric and organic acids. *Hydrometallurgy*, 2013, 140, 20-27.
- [15] Wen T., Zhao Y.L., Xiao Q.H., Ma Q.L., Kang S.C., Li H.Q., et al., Effect of microwave assisted heating on chalcopyrite leaching of kinetics, interface temperature and surface energy. *Results Phys.*, 2017, 7, 2594-2600.
- [16] Suoranta T., Zugazua O., Niemelä M., Perämäki P., Recovery of palladium, platinum, rhodium and ruthenium from catalyst materials using microwave-assisted leaching and cloud point extraction. *Hydrometallurgy*, 2015, 154, 56-62.
- [17] Maryam S.S., Vanpeteghem G., Neto I.F., Soares H.M., Selective leaching of Zn from spent alkaline batteries using environmentally friendly approaches. *Waste Manage.*, 2017, 60, 696-705.
- [18] Kim E., Horckmans L., Spooren J., Selective leaching of Pb, Cu, Ni and Zn from secondary lead smelting residues. *Hydrometallurgy*, 2017, 169, 372-381.
- [19] Astuti W., Hirajima T., Sasaki K., Okibe N., Comparison of effectiveness of citric acid and other acids in leaching of low-grade Indonesian saprolitic ores. *Miner. Eng.*, 2016, 85, 1-16.
- [20] Shaghghi-Moghaddam R., Jafarizadeh-Malmiri H., Mehdikhani P., Alijanianzadeh R., Jalalian S., Optimization of submerged fermentation conditions to overproduce bioethanol using two industrial and traditional *Saccharomyces cerevisiae* strains. *Green Process. Synth.*, 2019, 8(1), 157-162.
- [21] Tanong K., Coudert L., Mercier G., Blais J.F., Recovery of metals from a mixture of various spent batteries by a hydrometallurgical process. *J. Environ. Manage.*, 2016, 181, 95-107.
- [22] Saikia R., Goswami R., Bordoloi N., Senapati K. K., Pant K. K., Kumar M., et al., Removal of arsenic and fluoride from aqueous solution by biomass based activated biochar: optimization through response surface methodology. *J. Environ. Chem. Eng.*, 2017, 5, 5528-5539.
- [23] Jiang H.L., Yang J.L., Shi Y.P., Optimization of ultrasonic cell grinder extraction of anthocyanins from blueberry using response surface methodology. *Ultrason. Sonochem.*, 2017, 34, 325-331.
- [24] Liu H., Zhang Y.M., Huang J., Liu T., Xue N.N., Shi Q.H., Optimization of vanadium (IV) extraction from stone coal leaching solution by emulsion liquid membrane using response surface methodology. *Chem. Eng. Res. Des.*, 2017, 123, 111-119.
- [25] Nazari E., Rashchi F., Saba M., Mirazimi S.M.J., Simultaneous recovery of vanadium and nickel from power plant fly ash: Optimization of parameters using response surface methodology. *Waste Manage.*, 2014, 34, 2687-2696.
- [26] Kul M., Oskay K.O., Şimşir M., Sübütaş H., Kirgezen H., Optimization of selective leaching of Zn from electric arc furnace steelmaking dust using response surface methodology. *T. Nonferr. Metal. Soc.*, 2015, 25, 2753-2762.
- [27] Madakkaruppana V., Piusb A., Sreenivas T., Giri N., Sarbajna C., Influence of microwaves on the leaching kinetics of uraninite from a low grade ore in dilute sulfuric acid. *J. Hazard. Mater.*, 2016, 313, 9-17.