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Highly Efficient Recovery of Vanadium and Chromium: Optimized by Response Surface Methodology

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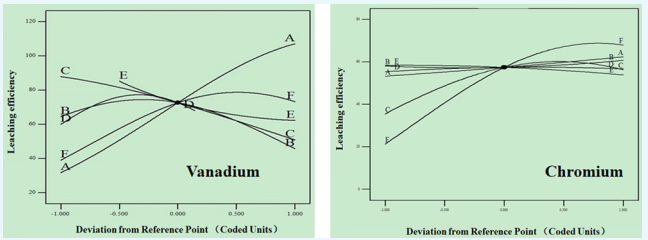
Supporting Information

ABSTRACT: Response surface methodology was applied to optimize the processing parameters (dosage of NaOH, dosage of H_2O_2, reaction temperature, liquid-to-solid ratio, stirring rate, and reaction time) that affected the leaching process of vanadium and chromium. The results indicated that the leaching process of vanadium was significantly affected by the dosage of NaOH and dosage of H_2O_2 used in the experiments, whereas the processing parameters affected the leaching efficiency of chromium in the following order: dosage of H_2O_2 (F) > reaction temperature (C) > dosage of NaOH (A) > reaction time (B) > stirring rate (D) > liquid-to-solid ratio (E). Almost 98.60% of vanadium and 79.82% of chromium were leached out during the leaching process.



1. INTRODUCTION

Vanadium and chromium are important national strategy resources and widely used in petrochemical industry, catalysts, and iron steel because of their excellent physicochemical properties.[1−8] To date, many technologies have been applied to leach out vanadium and chromium. Roasting-leaching is a common technology for vanadium extraction. Vanadium in low valence is oxidized to sodium vanadate with the addition of sodium salt in sodium-roasting technology.[9] Also, calcium-roasting technology has been applied to overcome some problems associated with sodium-roasting technology, such as the amount of harmful gases like Cl_2 and SO_2 and agglomeration during roasting process with sodium salts at low melting points. Lime or limestone was added to the slag, and vanadium in low valence was oxidized to CaV_2O_6, $Ca_2V_2O_7$, and $Ca_3V_2O_8$.[10−1410−14] The parameters affecting the leaching efficiency of vanadium including the dosage of CaO and roasting temperature and the roasting mechanism have attracted much more attention. Acid leaching technology was applied in leaching out vanadium from stone coal or was coupled with calcium-roasting technologies.[15−17] Liquid-phase oxidation technologies including electro-oxidation and sub-molten salt technology were also used to leach out vanadium.[18−22]

In many cases, the single-parameter experiment method was applied to investigate the parameters affecting the hydro-metallurgical process, in which only one parameter changed and others held constant. In these processes, only the effect of a single processing parameter was investigated, whereas the interactions among different parameters were ignored.[23−25] In recent studies,[21] the parameters affecting the leaching efficiency of vanadium and chromium including the dosage of NaOH, reaction time, stirring rate, reaction temperature, liquid-to-solid ratio, and dosage of H_2O_2 from vanadium−chromium residues were studied, whereas the effects of interactions among different parameters were ignored. Moreover, also which was the most significant parameter is still unknown. Response surface methodology (RSM)[26−30] is an efficient method that offers a large amount of information from a relatively small number of experiments, allowing the observation of both the effect of the independent variables on the response and their possible interactions. In addition, it has been extensively applied for the optimization study of test parameters and obtained response surfaces. Therefore, the aim of this article was mainly to optimize the leaching conditions of the leaching process of vanadium−chromium residues oxidized with H_2O_2 in alkaline medium. The optimal leaching conditions were optimized by RSM. The experimental results could provide the technical basis for the hydrometallurgical technologies for leaching of vanadium and chromium.

2. RESULTS AND DISCUSSION

2.1. Single-Parameter Experiments. The recent studies[21,22] showed that the six processing parameters including the dosage of NaOH, dosage of H_2O_2, reaction time, reaction temperature, stirring rate, and liquid-to-solid ratio affected the leaching efficiency of vanadium and chromium significantly. Vanadium and chromium could be easily leached out with the addition of H_2O_2 in alkaline medium.

2.2. Analysis of the Adequacy of the Fitted Model. Experiments were carried out to analyze the effect of processing parameters on the leaching efficiencies of vanadium

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and chromium. The design points and experimental results are presented in detail in Table S1.

2.2.1. Natural Logarithm Model for the Leaching Efficiency of Vanadium. The natural logarithm model, which was used to express the simulated results, for the leaching efficiency of vanadium after insignificant terms (p-values > 0.1) is presented in eq 1

$$\begin{aligned}\ln(\text{leaching efficiency}) &= 4.29 + 0.61*A - 0.17*B - 0.27*C - 0.38*D \\ &\quad - 0.26*E + 0.31*F + 0.16*A*B + 0.46*A*C \\ &\quad - 0.22*A*D + 0.12*A*E - 0.13*A*F - 0.25*B*C \\ &\quad - 0.029*B*D - 0.13*B*E - 0.11*B*F - 0.066*C \\ &\quad *D - 0.23*C*E - 0.23*C*F - 0.24*D*E - 0.081 \\ &\quad *D*F - 0.15*E*F - 0.22*A^2 - 0.29*B^2 - 0.082*C^2 \\ &\quad - 0.57*D^2 + 0.11*E^2 - 0.31*F^2 \end{aligned} \quad (1)$$

The results of the responses for the leaching efficiency of vanadium with analysis of variance are given in detail in Table 1. The F-value and p-value for the model were 7.12 and 0.0005, respectively, which indicated that the model was significant and could be used to describe the optimization process.

Table 1. Analysis of Variance (Results for Vanadium Model)[a]

source	S	Z	mean square	F-value	p-value probability
model	32.29	27	1.1	7.12	0.0005
residual	2.02	12	0.17		
lack of fit	1.81	7	0.26	6.33	0.0295
pure error	0.20	5	0.041		

[a] R^2 = 0.9412, R_{adj}^2 = 0.8090, pre-R^2 = −9.6804, adequate precision = 12.013.

2.2.2. Square-Root Model for the Leaching Efficiency of Chromium. The square-root model, which was used to express the simulated results, for the leaching efficiency of chromium after insignificant terms (p-values > 0.1) is presented in eq 2

$$\begin{aligned}\text{squares root}(\text{leaching efficiency}) &= 7.58 + 0.30*A + 0.088*B + 0.77*C + 0.0450*D \\ &\quad - 0.15*E + 1.81*F + 0.17*A*B - 0.22*A*C \\ &\quad + 0.16*A*D - 0.15*A*E - 0.031*A*F - 0.17*B*C \\ &\quad + 0.0005231*B*D + 0.041*B*E + 0.078*B*F \\ &\quad - 0.20*C*D - 0.067*C*E + 0.031*C*F - 0.046 \\ &\quad *D*E - 0.019*D*F - 0.13*E*F + 0.021*A^2 + 0.13 \\ &\quad *B^2 - 0.84*C^2 - 0.095*D^2 - 0.089*E^2 - 1.15*F^2 \end{aligned} \quad (2)$$

The results for chromium are given in detail in Table 2. The F-value for the model was 68.59. The corresponding p-value for the model was < 0.0001, which indicated that the model was significant and could be used to describe the optimization process.

2.3. Response Surfaces and Contour Plots. It could be seen from Figure 1 that the internally studentized residuals were almost in a straight line, which indicated that the normal probability was in a normal distribution. Also, the leaching efficiency of vanadium was more stable than that of chromium.

Table 2. Analysis of Variance (Results for Chromium Model)[a]

source	S	Z	mean square	F-value	p-value probability
model	127.24	27	4.71	68.59	<0.0001
residual	0.82	12	0.069		
lack of fit	0.41	7	0.058	0.69	0.6830
pure error	0.42	5	0.084		

[a] R^2 = 0.9936, R_{adj}^2 = 0.9791, pre-R^2 = 0.5136, adequate precision = 29.068.

The predicted results versus actual results are plotted in Figure 2. It was shown that the predicted leaching efficiency of chromium was more in agreement with the actual results than that of the leaching efficiency of vanadium. This also meant that the simulated model was valid and could be used to predict the actual results. Figure 3 shows the plot of the internally studentized residual responses versus the predicted responses; the internally studentized residual responses were random and had no obvious regulation. This indicated that the actual results were dependent on the processing parameters.

The perturbation plot in Figure 4 shows a comparison of all of the processing parameters. The leaching efficiency response results were drawn by changing only one processing parameter over its range while keeping the other processing parameters constant. For the leaching process of vanadium, the influence of processing parameters on the obtained leaching efficiency response followed the order: dosage of NaOH (A) > dosage of H_2O_2 (F) > reaction temperature (C) > liquid-to-solid ratio (E) > stirring rate (D) > reaction time (B). The comparatively sharp line of dosage of NaOH significantly affected the leaching efficiency. Note that the coefficient of the dosage of NaOH in eq 2 is extremely high. The increase of NaOH increased the reaction activity of the hydroxide ions and enabled the oxidation reactions to be thermodynamically more favorable.[21] It also intensified the oxidation solubility and mass transfer efficiency during the leaching process. eq 2 and the perturbation plot show that the reaction time and stirring rate exhibited parabola effects. The leaching efficiency increased linearly with the increase of the dosage of NaOH and dosage of H_2O_2 in the chosen design space.

While taking the leaching process of chromium into account, the influence of processing parameters on the obtained leaching efficiency response followed the order: dosage of H_2O_2 (F) > reaction temperature (C) > dosage of NaOH (A) > reaction time (B) > stirring rate (D) > liquid-to-solid ratio (E). The dosage of H_2O_2 (F) significantly affected the leaching efficiency of chromium, and the reaction temperature (C) exhibited curvature effects, whereas other parameters had no significant effects. The leaching efficiency of chromium was significantly affected by the dosage of H_2O_2,[21,2221,22] whereas it was hardly leached out in alkaline leaching without H_2O_2.[3131] In other words, the leaching process of chromium was dependent on the addition of H_2O_2.

Three-dimensional plots were applied to investigate the interaction among processing parameters. In such plots, two processing parameters were changed while keeping the other four processing parameters constant. The results given in detail in Figures S1−S8 show that the leaching efficiency of vanadium was increased with the increasing dosage of NaOH. Moreover, the leaching efficiency of chromium was significantly affected by the dosage of NaOH and dosage of H_2O_2.

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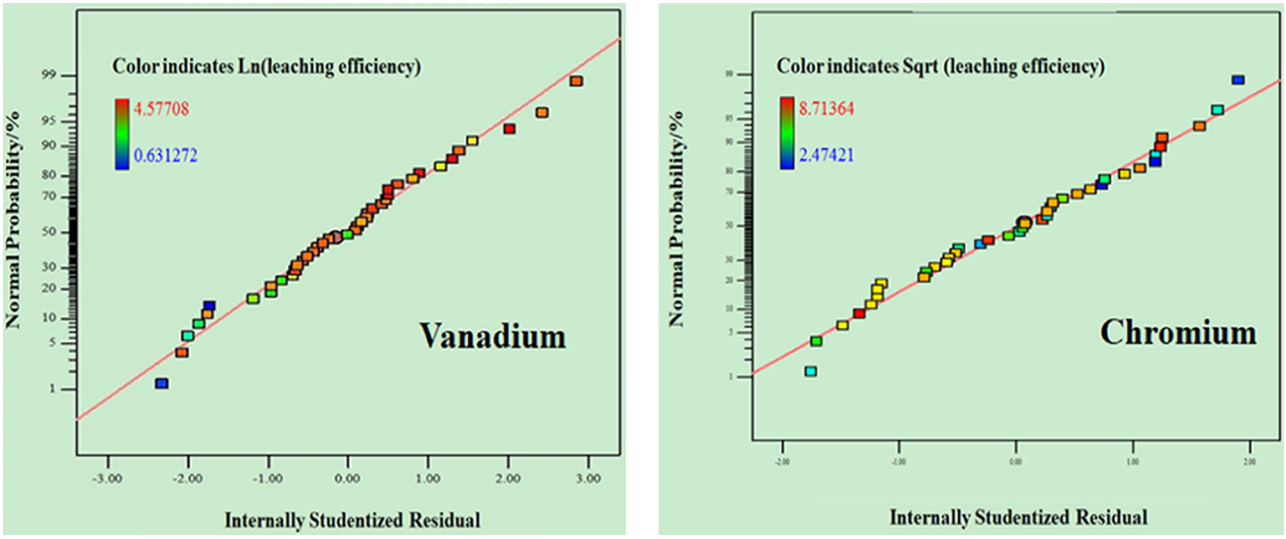

Figure 1. Plot of normal probability vs the internally studentized residuals.

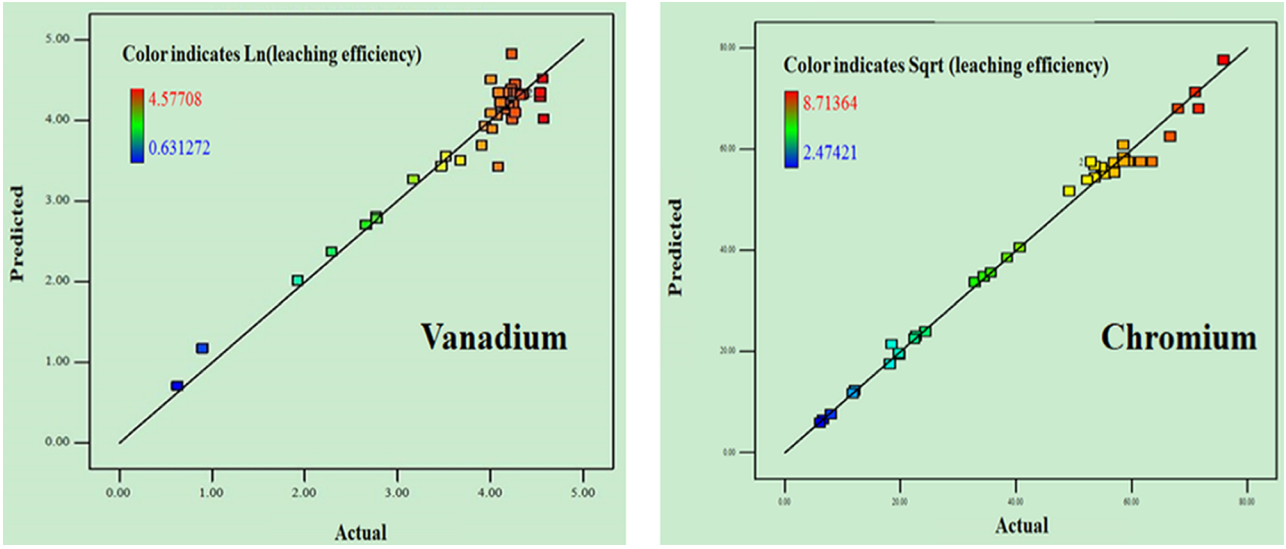

Figure 2. Predicted responses vs the actual values.

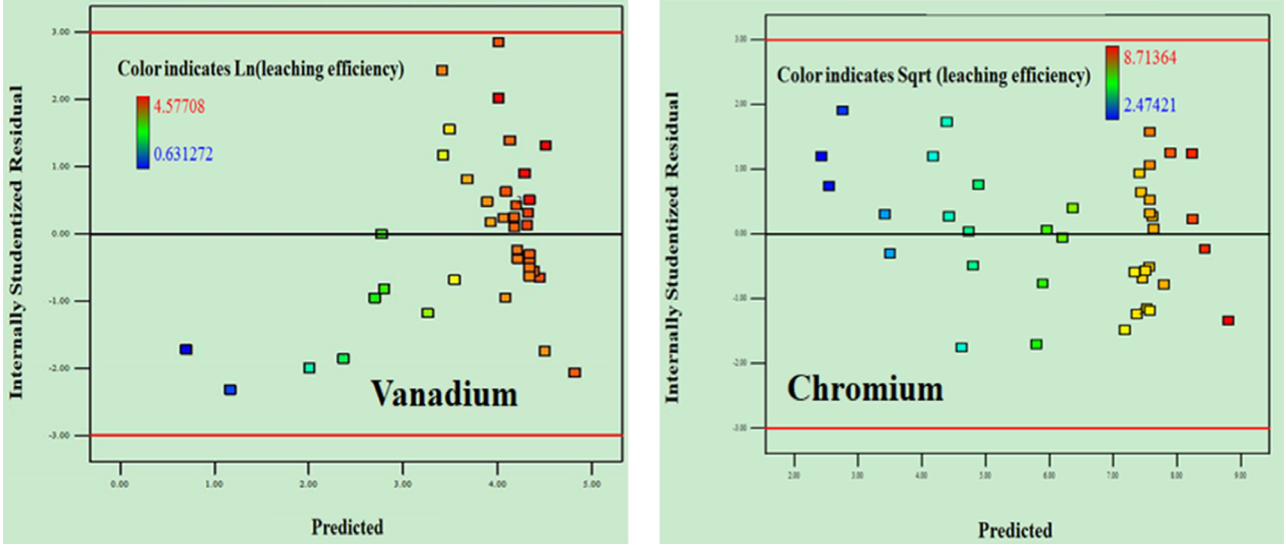

Figure 3. Internally studentized residuals vs the predicted responses.

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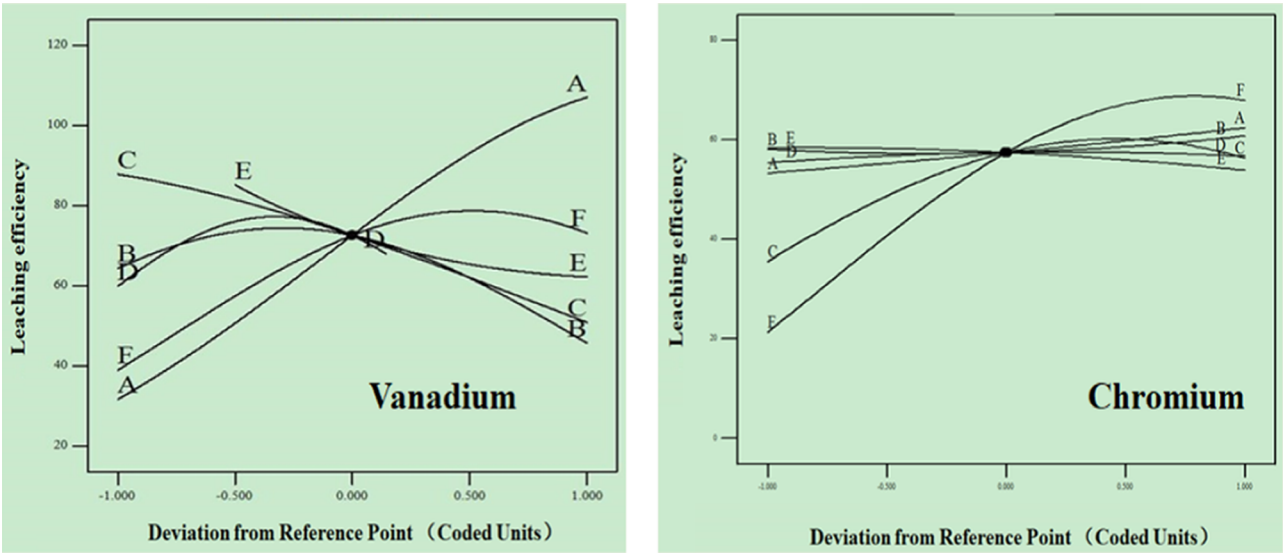

Figure 4. Perturbation plot for the leaching efficiencies of vanadium and chromium (A: dosage of NaOH; B: dosage of H_2O_2; C reaction time; D: reaction temperature; E: stirring rate; and F: liquid-to-solid ratio).

Table 3. Actual and Predicted Results under the Optimum Conditions Predicted via RSM

	optimum conditions				leaching efficiency/%			
	$m_{NaOH}/m_{residue}$	$V_{H_2O_2}/m_{residue}$	reaction time/min	reaction temperature/°C	stirring rate/rpm	liquid-to-solid-mass ratio	actual	predicted
vanadium	0.70	0.70	60	60	550	4	98.60	99.80
chromium	1.00	0.90	84	70	300	2.50	79.82	80.16

2.4. Optimization of Leaching Parameters and the Model Validation. The effects of a single parameter and interaction among parameters were discussed above. The optimal leaching conditions were also investigated. Optimum conditions were obtained when all of the processing parameters were selected "within the range" and the leaching efficiency of vanadium or chromium was maximum. The processing parameters and related responses are given in detail in Table 3 and Figure 5. The selected optimum conditions with a desirability value of 1.000 were predicted to reach 99.80 and 80.16% for the leaching efficiencies of vanadium and chromium, respectively. In Table 3, the results showed great agreement between the simulated predicted results and experimental results, which confirmed the validity of the model in simulating the leaching process of vanadium and chromium from vanadium−chromium residues.

3. CONCLUSIONS

In this study, the effects of six processing parameters (dosage of NaOH, dosage of H_2O_2, reaction temperature, liquid-to-solid ratio, stirring rate, and reaction time) on the leaching efficiencies of vanadium and chromium from vanadium−chromium residues were studied. The optimal processing parameters were obtained from RSM.

(1) The leaching efficiency of vanadium was significantly affected by the dosage of NaOH and dosage of H_2O_2 during the experiments. The leaching efficiency of vanadium was up to 98.60% at optimum technological parameters: dosage of NaOH of 0.70 g/g, dosage of H_2O_2 of 0.70 mL/g, reaction time of 60 min, reaction temperature of 60 °C, stirring rate of 550 rpm, and liquid-to-solid ratio of 4 mL/g.

(2) The optimal processing parameters were obtained from RSM, and the influence of processing parameters on the obtained rate response followed the order: dosage of H_2O_2 (F) > reaction temperature (C) > dosage of NaOH (A) > reaction time (B) > stirring rate (D) > liquid-to-solid ratio (E). Under the optimal conditions, the leaching efficiency of chromium was 79.82%: dosage of NaOH of 0.99 g/g, dosage of H_2O_2 of 0.90 mL/g, reaction time of 84 min, reaction temperature of 70 °C, stirring rate of 300 rpm, and liquid-to-solid ratio of 2.5 mL/g.

4. EXPERIMENTAL SECTION

4.1. Materials. The vanadium−chromium residue was collected from Pangang Group Co., Ltd., Chengdu, China. Also, it was precipitated from waste water containing vanadium and chromium in low valence from an iron and steel mill. The residue was dried and ground to fine particles before experiments. The chemical composition of the residue is given in detail in the references.[21]

4.2. Experimental Procedure. All experiments were conducted in a thermostatic mixing water bath pot. Before the experiments, a predetermined concentration of NaOH solution was added to the beaker, and then the residue was added as the appropriate temperature was reached. Then, H_2O_2 was manually added. The leachate was obtained by vacuum filtration after a required time. The detailed processes can be seen in the refs21, 22, 31. The concentrations of vanadium and chromium in the filtrate were determined by inductively coupled plasma-optical emission spectrometry.[18,32−34] The leaching efficiencies of vanadium (η_V) and chromium (η_{Cr}) are calculated in eqs 3 and 4, respectively, as follows

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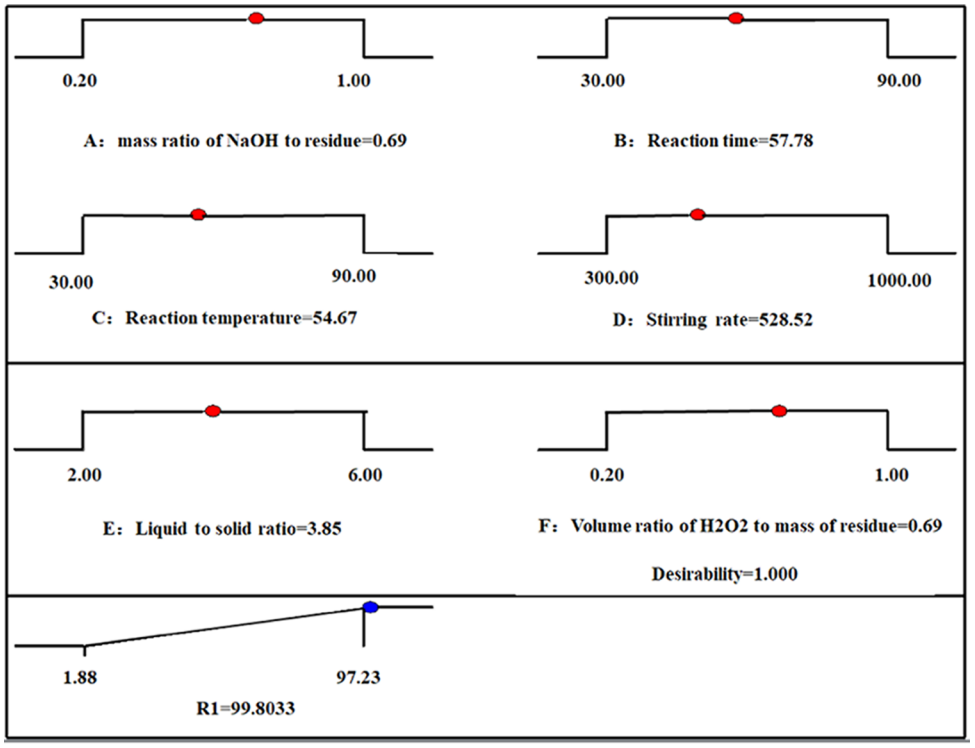
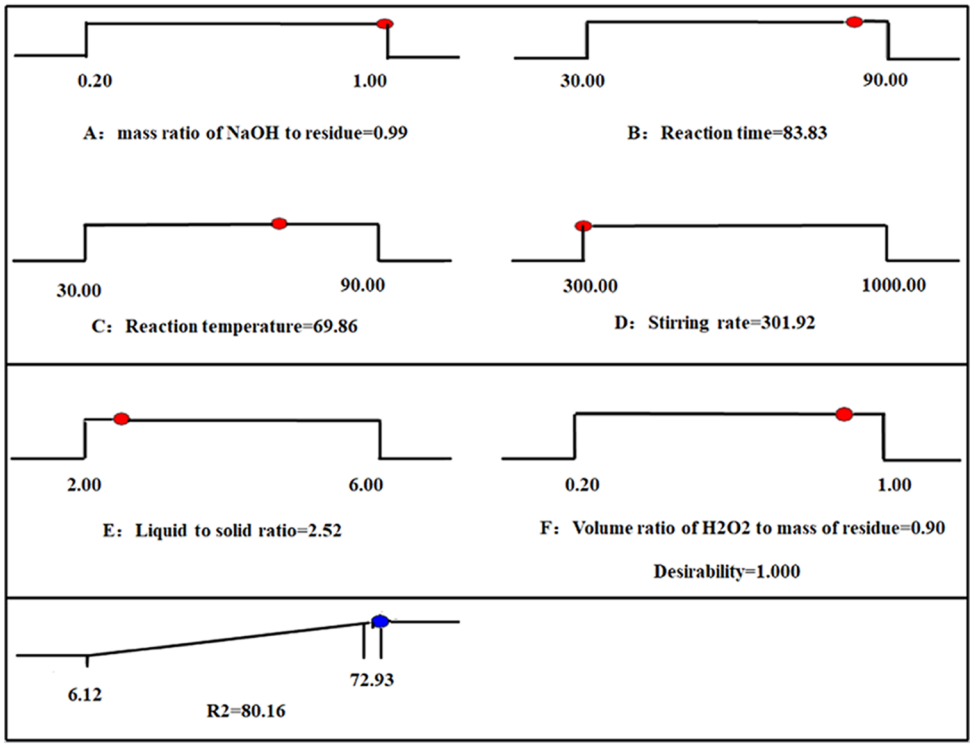


a Vanadium



b Chromium

Figure 5. Desirability ramp for numerical optimization.

$$\eta_{V} = \frac{V \cdot C_{V}}{m\omega_{V}} \times 100\% \quad (3)$$

$$\eta_{Cr} = \frac{V \cdot C_{Cr}}{m\omega_{Cr}} \times 100\% \quad (4)$$

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where C_V and C_{Cr} are the concentrations (g/L) of vanadium and chromium in the filtrate, respectively; V is the volume (mL) of the leachate; ω_V and ω_{Cr} are the mass fractions of vanadium and chromium in the residue, respectively; and m (g) is the mass of the vanadium−chromium residue used in the leaching experiments.

4.3. Experimental Optimization. The experiment design and optimization methods were according to the reference.[23] Central composite design (CCD) was applied to analyze the effect of processing parameters on the leaching efficiencies of vanadium and chromium from the vanadium−chromium residue. The operation processing parameters included the dosage of NaOH, dosage of H_2O_2, reaction time, reaction temperature, stirring rate, and liquid-to-solid ratio. The actual values for each processing parameter are given in detail in Table 4.

Table 4. Actual Values of Process Variables

		level		
variable	unit	−1	0	1
A: $m_{NaOH}/m_{residue}$	g/g	0.2	0.6	1.0
B: reaction time	min	30	60	90
C: reaction temperature	°C	30	60	90
D: stirring rate	rpm	300	500	700
E: liquid-to-solid-mass ratio	mL/g	3	4	6
F: $V_{H_2O_2}/m_{residue}$	mL/g	0.2	0.6	1.0

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.8b02708.

CCD experimental matrix and experimental results for this study (Table S1); response surface plots for factors (A to B, C, D, and E) (Figure S1); response surface plots for factors (B to C, D, E, and F) (Figure S2); response surface plots for factors (A to F and C to D, E, and F) (Figure S3); response surface plots for factors (D to E, F and E to F) (Figure S4); response surface plots for factors (A to B, C, D, and E) (Figure S5); response surface plots for factors (B to C, D, E, and F) (Figure S6); response surface plots for factors (A to F and C to D, E, and F) (Figure S7); response surface plots for factors (D to E, F and E to F) (Figure S8) (PDF)

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Notes

The authors declare no competing financial interest.

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