

Article

Vanadium Extraction by Acid Leaching from Vanadium Slag Produced by Microwave-Assisted Calcification Roasting: Leaching Behavior and Optimization

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Abstract

Vanadium slag is an important secondary vanadium resource. Although microwave-assisted calcification roasting improves the leachability of vanadium-bearing phases, further extraction can still be limited during acid leaching, making optimization of the leaching process essential for efficient vanadium recovery. Using this slag, leaching was evaluated at different temperatures, times, liquid-to-solid ratios (L/S), sulfuric acid concentrations, and agitation speeds. A Box–Behnken design (BBD) was used to optimize leaching parameters within the selected ranges. Residue phase composition and microstructure were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive spectroscopy (EDS). Leaching efficiency increased with temperature, L/S, and acid concentration, but plateaued above 60 °C, 6 mL·g^{−1}, and 14 wt.%, respectively; increases beyond 50 min or 200 rpm gave marginal improvements. Analysis of variance (ANOVA) of the BBD model ranked the statistical effects of the four linear terms within the investigated BBD range as sulfuric acid concentration > L/S > leaching time > temperature. Within the selected BBD parameter ranges, optimization yielded 64.95 °C, 55.21 min, 6.56 mL·g^{−1}, and 15.17 wt.% sulfuric acid, with agitation fixed at 200 rpm. Validation gave an average leaching efficiency of 92.92%, with a relative error of 0.205% compared with the model prediction. After leaching, Mn₂V₂O₇ was undetected. The residue mainly contained irregular particles, 10–30 μm acicular or plate-like CaSO₄·2H₂O crystals, and minor residual vanadium-bearing CrVO₃ and CaVH₂Si₄O₁₂ phases. Surface CaSO₄·2H₂O deposition and refractory-phase encapsulation of vanadium-bearing constituents increased mass-transfer resistance and limited further leaching. This study clarified the relative effects of the investigated leaching conditions on vanadium leaching efficiency within the design range and the interactions among these conditions, and provided microstructural evidence related to the factors limiting further vanadium leaching, thereby providing theoretical guidance for the efficient extraction of vanadium from vanadium slag.

Keywords: vanadium slag; vanadium extraction; calcification roasting; acid leaching



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1. Introduction

Vanadium slag, generated during vanadium extraction from vanadium-bearing hot metal, is an important secondary vanadium resource and a major feedstock for producing

vanadium and its compounds [1,2]. Owing to its complex phase assemblage and closely intergrown minerals, vanadium occurs mainly as low-valence species in structurally stable vanadium-bearing spinel, some of which is encapsulated by silicate phases, hindering efficient vanadium leaching and recovery [2,3]. Therefore, disrupting the vanadium-bearing spinel structure, oxidizing low-valence vanadium, and converting it into readily leachable phases are essential for efficient vanadium recovery from slag [2,4].

During calcification roasting, calcium-bearing additives react with vanadium slag, converting low-valence vanadium into acid-soluble calcium vanadates through oxidation [5,6]. Compared with conventional sodium roasting, calcification roasting avoids large additions of sodium salts, thereby reducing sodium-bearing wastewater and the associated environmental burden [7,8]. Microwave-assisted calcification roasting was employed to enhance heat and mass transport, reduce energy consumption, and facilitate the conversion of vanadium-bearing phases. As a result of the volumetric, phase-selective absorption of microwave energy, microwave heating promotes spinel oxidation and structural breakdown while favoring the generation of calcium vanadate phases, which enhances the reactivity of the roasted vanadium slag [9–11]. However, roasting activation only favors subsequent extraction; efficient vanadium recovery from the roasted vanadium slag still depends on phase dissolution, interfacial reactions, and mass transfer during acid leaching [8,12].

Sulfuric acid is commonly used to leach calcification-roasted vanadium slag, with most vanadium dissolution occurring during the initial stage [8]. Appropriate leaching conditions promote the decomposition of vanadium-bearing phases, enhance acid penetration and diffusion within roasted vanadium slag particles, and improve liquid–solid interfacial mass transfer [12]. However, excessive sulfuric acid concentration, prolonged leaching, or an unsuitable liquid-to-solid ratio may intensify CaSO_4 crystallization and silicic acid polymerization and gelation, causing particle-surface coating or pore blockage, increasing mass-transfer resistance, and suppressing further vanadium dissolution [13]. Previous studies have been conducted to enhance vanadium leaching efficiency. Gao et al. investigated the strengthening effect of microwave irradiation during the calcification roasting of high-chromium vanadium slag, systematically examining how roasting temperature, the $\text{CaO}/\text{V}_2\text{O}_5$ ratio, and roasting time affected vanadium extraction, and further interpreted the characteristics of microwave-assisted calcification roasting based on the observed changes in phase composition and microstructure [14]. Wang et al. applied response surface methodology to perform multivariable optimization of the composite roasting–acid leaching process for vanadium extraction from vanadium slag, quantitatively evaluated the effects of different roasting parameters and their interactions on the subsequent vanadium-leaching performance, and determined the corresponding optimized roasting conditions [15]. Previous studies respectively clarified the enhancement of vanadium extraction from vanadium slag by microwave calcification roasting and the applicability of multivariate statistical optimization to conventional roasting–acid leaching systems. Building on these studies, the present work further investigated the effects of leaching conditions on vanadium leaching efficiency from microwave-calcification-roasted vanadium slag and their interactions, determined a local optimum within the investigated range, and analyzed the possible factors limiting further vanadium leaching during acid leaching based on the phase composition and microstructural characteristics.

Therefore, in this study, microwave-assisted calcification-roasted vanadium slag was selected as the feedstock, with vanadium leaching efficiency examined across different leaching temperatures, leaching times, liquid-to-solid ratios, sulfuric acid concentrations, and agitation speeds. Response surface methodology was subsequently applied to establish a quadratic regression model describing the dependence of vanadium leaching efficiency on these leaching parameters. Analysis of variance was used to evaluate the significance

of the model terms and compare the contributions of the selected leaching variables. The model-predicted local optimum within the investigated BBD space was then verified experimentally. The leaching residue was characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) to identify changes in phase composition and examine the morphology and distribution of CaSO_4 -containing products. The above findings can provide a theoretical basis for the efficient extraction of vanadium from vanadium slag.

2. Experimental

2.1. Characteristics of the Roasted Vanadium Slag

The microwave-assisted calcification-roasted vanadium slag contained 32.60 wt.% total Fe (TFe), 12.53 wt.% V_2O_5 , 5.11 wt.% CaO, and 15.60 wt.% SiO_2 . This composition corresponded to a $\text{CaO}/\text{V}_2\text{O}_5$ mass ratio of 0.41. The detailed chemical composition of the microwave-assisted calcification-roasted vanadium slag is presented in Table 1. This vanadium slag was sourced from the production site of a certain iron and steel enterprise in China. XRD analysis showed that the main crystalline phases identified in the roasted vanadium slag included Fe_2O_3 , Fe_2TiO_5 , $\text{Ca}(\text{Fe,Mg})\text{Si}_2\text{O}_6$, and the vanadium-bearing phases $\text{Mn}_2\text{V}_2\text{O}_7$, CrVO_3 , and $\text{CaVH}_2\text{Si}_4\text{O}_{12}$ (Figure 1). X-ray diffraction (XRD) analysis was performed using a SmartLab X-ray diffractometer (Rigaku Corporation, Akishima, Tokyo, Japan).

Table 1. Chemical composition of the microwave-calcified vanadium slag (wt.%).

Component	V_2O_5	TFe	TiO_2	SiO_2	MnO	CaO	MgO	Al_2O_3	Cr_2O_3	<i>p</i>
Content	12.53	32.60	11.64	15.60	8.03	5.11	2.43	3.32	4.71	0.142

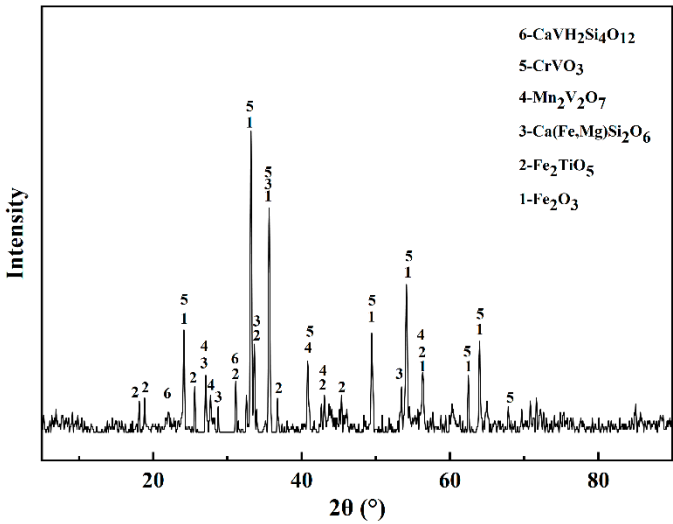


Figure 1. XRD analysis of the roasted vanadium slag.

2.2. Experimental Procedure

A predetermined mass (m_0) of microwave-assisted calcification-roasted vanadium slag was weighed for each leaching experiment. All chemical reagents used in this study were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China) and were of analytical grade unless otherwise specified. The required volume of sulfuric acid solution was calculated from the roasted vanadium slag mass and the selected liquid-to-solid ratio, after which a solution of the specified acid concentration was prepared. The roasted vanadium slag and leaching solution were placed in a covered beaker and heated in a

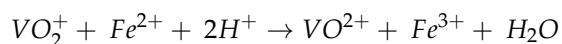
thermostatic water bath. The slurry was stirred at the required speed, and leaching time was counted once the target temperature had been reached. After the specified period, heating and stirring were stopped. The beaker was removed from the water bath and left to cool. The slurry was then filtered, and the residue was washed repeatedly with deionized water to obtain the vanadium-bearing filtrate and the leaching residue. The latter was dried in an oven to constant weight, brought to room temperature, and finally weighed, with its mass denoted as m_r . Vanadium leaching efficiency was subsequently determined, and the residue was retained for further characterization and analysis.

The vanadium leaching efficiency in this study was determined using the potassium permanganate oxidation–ferrous ammonium sulfate titration method, with reference to the titration procedures reported by Li et al. and Zhang et al. [16,17]. The specific procedure was as follows: the dried leaching residue was decomposed by Na_2O_2 fusion and subsequently treated with H_2SO_4 to ensure that vanadium was fully transferred into the solution for analysis. Under acidic conditions, KMnO_4 was added to oxidize vanadium to V(V) . Urea was then added, followed by NaNO_2 to remove excess KMnO_4 . N-phenylanthranilic acid was used as the indicator, and the solution was titrated to the endpoint with a standardized ferrous ammonium sulfate solution at a concentration of $c_t \text{ mol}\cdot\text{L}^{-1}$. The consumed titrant volume, V_t (L), was recorded. The entire resulting solution was used for titration, and the vanadium leaching efficiency was calculated according to Equations (1) and (2). The experimental procedure was conducted with reference to YB/T 547.1-2014 [18].

$$\eta_r = \frac{m_0 w_{V,0} - m_r w_{V,r}}{m_0 w_{V,0}} \times 100\% \quad (1)$$

$$m_r w_{V,r} = c_t V_t M_V \quad (2)$$

The titration equation is as follows:



where η_r denotes the vanadium leaching efficiency determined by the residue-based mass-balance method, %; m_0 is the initial mass of the roasted vanadium slag used for leaching, g; m_r is the mass of the leaching residue, g; $w_{V,0}$ is the initial elemental V mass fraction of the roasted vanadium slag, dimensionless, with a value of 0.0702 corresponding to 7.02 wt.% V; $w_{V,r}$ is the elemental V mass fraction in the leaching residue, dimensionless; c_t is the molar concentration of the ferrous ammonium sulfate titrant, $\text{mol}\cdot\text{L}^{-1}$; V_t is the volume of titrant consumed, L; and M_V is the molar mass of vanadium, $50.94 \text{ g}\cdot\text{mol}^{-1}$.

Single-factor experiments were first conducted to evaluate the effects of the main leaching parameters. Leaching temperature was varied from 40 to 80 °C (40, 50, 60, 70, and 80 °C), leaching time from 10 to 70 min (10, 20, 30, 40, 50, 60, and 70 min), liquid-to-solid ratio from 1 to 7 $\text{mL}\cdot\text{g}^{-1}$ (1, 2, 3, 4, 5, 6, and 7 $\text{mL}\cdot\text{g}^{-1}$), sulfuric acid concentration from 4 to 18 wt.% (4, 6, 8, 10, 12, 14, 16, and 18 wt.%), and agitation speed from 0 to 500 rpm (0, 100, 200, 300, 400, and 500 rpm). Unless otherwise specified, the reference conditions were a leaching temperature of 60 °C, a leaching time of 50 min, a liquid-to-solid ratio of 6 $\text{mL}\cdot\text{g}^{-1}$, a sulfuric acid concentration of 14 wt.%, and an agitation speed of 200 rpm. When one factor was varied, the remaining factors were maintained at their reference values. All single-factor leaching experiments were independently repeated three times under identical conditions. The experimental results are reported as the mean values of the three measurements.

Based on the single-factor experiments, a four-factor, three-level Box–Behnken design (BBD) was employed within the response surface methodology framework using Design-Expert 11.0. Leaching temperature (X_1), leaching time (X_2), liquid-to-solid ratio (X_3), and

sulfuric acid concentration (X_4) were selected as the independent variables, while vanadium leaching efficiency was taken as the response (y). The agitation speed was fixed at 200 rpm. Each factor was investigated at three coded levels (-1 , 0 , and $+1$). The corresponding actual levels were 50, 60, and 70 °C for leaching temperature; 40, 50, and 60 min for leaching time; 5, 6, and 7 mL·g⁻¹ for the liquid-to-solid ratio; and 12, 14, and 16 wt.% for sulfuric acid concentration, respectively. The BBD consisted of 29 experimental runs, including five replicates at the center point. The factor levels and corresponding coded values are summarized in Table 2.

Table 2. Factors, levels, and corresponding coded values used in the optimization of the leaching conditions.

Factor	Symbol	Coded Levels		
		−1	0	1
Leaching temperature/°C	X_1	50	60	70
Leaching time/min	X_2	40	50	60
Liquid-to-solid ratio/(mL·g ⁻¹)	X_3	5	6	7
Sulfuric acid concentration/wt. %	X_4	12	14	16

Inductively coupled plasma optical emission spectrometry (ICP-OES; iCAP 7400, Thermo Fisher Scientific, Waltham, MA, USA) was also used in this study to validate all measurement results within the investigated experimental conditions. The relative differences in vanadium leaching efficiency obtained by the two methods were all no greater than 0.1%, supporting the reliability of the experimental measurements within the parameter ranges investigated in this study. Finally, the leaching residue generated under the local optimum within the BBD space was analyzed by XRD and SEM-EDS (GeminiSEM 300, Carl Zeiss Microscopy GmbH, Oberkochen, Germany; XFlash 6 | 30 EDS detector, Bruker Nano GmbH, Berlin, Germany).

3. Results and Discussion

3.1. Effect of Leaching Parameters on Vanadium Leaching Efficiency

3.1.1. Leaching Temperature

Figure 2 shows the effect of leaching temperature on vanadium leaching efficiency. As shown in the figure, when the leaching temperature increased from 40 °C to 50 °C and 60 °C, the vanadium leaching efficiency increased from 85.98% to 90.90% and 92.62%, respectively, corresponding to increases of 4.92 and 1.72 percentage points for each 10 °C increase. When the temperature was further increased to 70 °C and 80 °C, the vanadium leaching efficiency reached 92.74% and 92.89%, respectively, corresponding to increases of only 0.12 and 0.15 percentage points for each additional 10 °C increase. These results indicate that increasing temperature is favorable for improving vanadium leaching efficiency, but its promoting effect gradually weakens as the temperature increases. Once the temperature reaches 60 °C, the rapid-increase stage of vanadium leaching efficiency has essentially ended, and further increases in temperature result in only very limited improvement. The reduced temperature sensitivity may be related to the formation and deposition of calcium sulfate during leaching. Calcium sulfate crystallization at the reaction interface or on particle surfaces can increase diffusion resistance and hinder the transport of dissolved species between the bulk solution and the reaction interface [19,20]. In addition, previous studies in related acidic V(V) systems have suggested that elevated temperatures may affect the stability of V(V) species and promote their polymerization and precipitation [21,22].

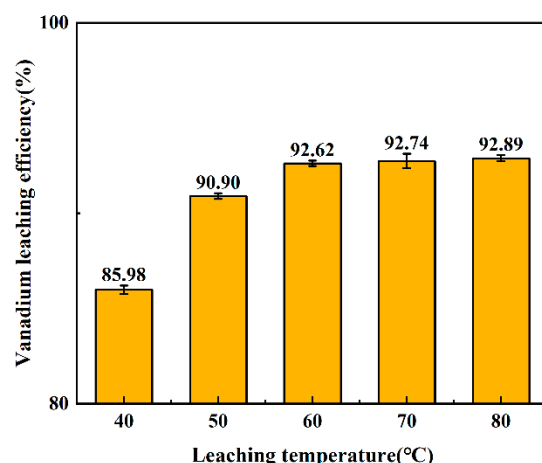


Figure 2. Effect of leaching temperature on vanadium leaching efficiency.

3.1.2. Leaching Time

Figure 3 shows the effect of leaching time on vanadium leaching efficiency. With increasing leaching time, the vanadium leaching efficiency initially increased markedly and then gradually approached a stable level. When the leaching time was extended from 10 min to 20 min and 30 min, the vanadium leaching efficiency increased from 80.02% to 85.71% and 89.45%, respectively, corresponding to increases of 5.69 and 3.74 percentage points. This indicates that extending the reaction time during the initial stage of leaching markedly promoted vanadium leaching. When the leaching time was further extended to 40 min and 50 min, the vanadium leaching efficiency increased to 91.29% and 92.35%, respectively, while the increases between adjacent time intervals decreased to 1.84 and 1.06 percentage points. This indicates that the promoting effect of extending the leaching time gradually weakened as the leaching process proceeded. Further extending the leaching time to 60 min increased the vanadium leaching efficiency by only 0.16 percentage points, reaching 92.51%. When the leaching time was further extended to 70 min, the vanadium leaching efficiency slightly decreased to 92.34%, indicating that extending the leaching time beyond 50 min provided no obvious further improvement in leaching efficiency. Therefore, 50 min was considered a suitable leaching time.

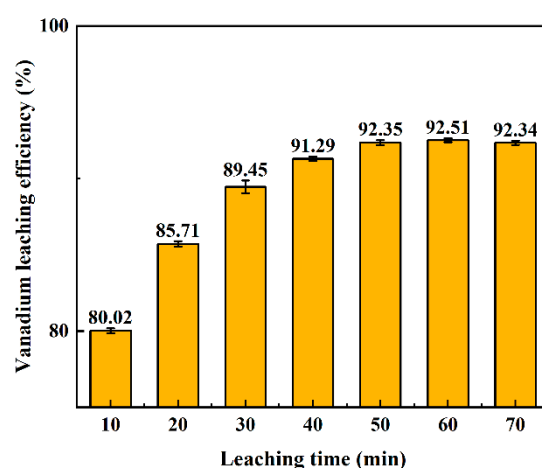


Figure 3. Effect of leaching time on vanadium leaching efficiency.

3.1.3. Liquid-to-Solid Ratio

Figure 4 shows the effect of the liquid-to-solid ratio on vanadium leaching efficiency. As the liquid-to-solid ratio increased, the vanadium leaching efficiency continuously increased, while the increment between adjacent liquid-to-solid ratio levels gradually de-

creased. When the liquid-to-solid ratio increased from 1 to 2 mL·g⁻¹, the vanadium leaching efficiency increased by 3.19 percentage points. When it increased from 2 to 3 mL·g⁻¹, the increment decreased to 1.91 percentage points. Further increases from 3, 4, and 5 mL·g⁻¹ to 4, 5, and 6 mL·g⁻¹ resulted in increases in vanadium leaching efficiency of 1.31, 0.63, and 0.55 percentage points, respectively. These results indicate that the promoting effect of increasing the liquid-to-solid ratio on vanadium leaching gradually weakened. When the liquid-to-solid ratio was further increased from 6 to 7 mL·g⁻¹, the vanadium leaching efficiency increased only from 92.10% to 92.13%, corresponding to an increase of only 0.03 percentage points. This indicates that further increasing the liquid-to-solid ratio provided almost no additional improvement in vanadium leaching efficiency, and the system essentially approached a stable level at approximately 6 mL·g⁻¹. At lower liquid-to-solid ratios, the limited amount of leaching solution and the relatively high slurry concentration may restrict solid–liquid contact and mass transfer. As the liquid-to-solid ratio increases, these limitations are gradually alleviated, which is favorable for the further leaching of vanadium-bearing phases. However, once the liquid-to-solid ratio reaches approximately 6 mL·g⁻¹, the promoting effect of further increasing the amount of leaching solution becomes very limited. Therefore, under the investigated conditions, 6 mL·g⁻¹ can be considered a suitable liquid-to-solid ratio.

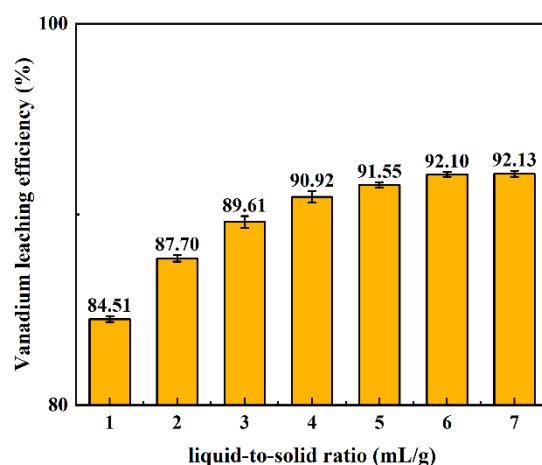


Figure 4. Effect of the liquid-to-solid ratio on vanadium leaching efficiency.

3.1.4. Sulfuric Acid Concentration

Figure 5 shows the effect of sulfuric acid concentration on vanadium leaching efficiency. The vanadium leaching efficiency increased from 77.52% at 4 wt.% to 82.14% at 6 wt.%, 86.34% at 8 wt.%, 89.65% at 10 wt.%, 91.65% at 12 wt.%, and further to 92.25% at 14 wt.%. The corresponding increases between adjacent concentration levels were 4.62, 4.20, 3.31, 2.00, and 0.60 percentage points, respectively, indicating that the promoting effect of increasing sulfuric acid concentration on vanadium leaching gradually weakened as the concentration increased. When the sulfuric acid concentration was further increased to 16 and 18 wt.%, the vanadium leaching efficiency increased by only 0.17 and 0.11 percentage points, respectively. This trend is mainly associated with the rising H⁺ concentration, which shifts the reaction equilibrium toward dissolved species. At the same time, proton-assisted decomposition of calcium vanadate is intensified, promoting its dissolution in the sulfuric acid medium. Consequently, dissolution of the vanadium-bearing phases is enhanced, facilitating vanadium transfer from the solid phase to the leach solution and increasing vanadium leaching efficiency [23]. Considering that the increase in vanadium leaching efficiency became markedly smaller when the sulfuric acid concentration exceeded 14 wt.%,

and that higher acid concentrations may also increase the dissolution of impurities, 14 wt.% was selected as the reference sulfuric acid concentration for the subsequent experiments.

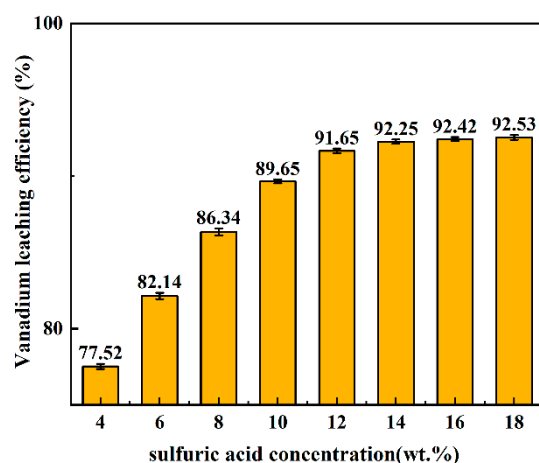


Figure 5. Effect of sulfuric acid concentration on vanadium leaching efficiency.

3.1.5. Agitation Speed

Figure 6 shows the variation in vanadium leaching efficiency with agitation speed. Relative to the unstirred condition, agitation at 100 rpm produced a marked increase in efficiency. A smaller additional gain was observed at 200 rpm, whereas higher speeds caused little further change. Without agitation, incomplete particle suspension and local settling may have restricted contact between the roasted vanadium slag and the sulfuric acid solution. Stirring improved particle dispersion, renewed the liquid film surrounding the solids, and facilitated mass transport at the liquid–solid interface. These effects favored the dissolution of acid-soluble vanadium-bearing phases. The nearly constant efficiency above 200 rpm suggests that external liquid-film transport was no longer a major limitation within this agitation range.

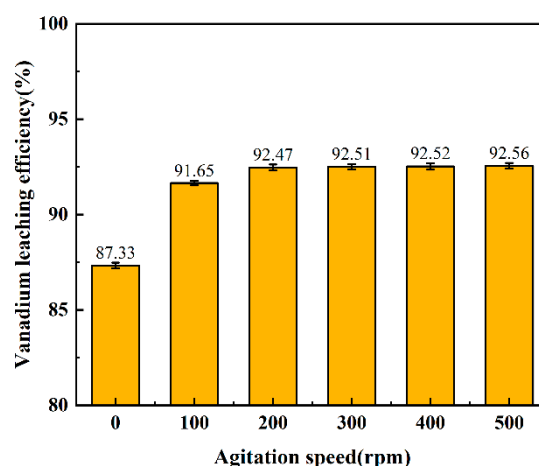


Figure 6. Effect of agitation speed on vanadium leaching efficiency.

3.2. Analysis and Discussion of Factors Affecting Vanadium Leaching

3.2.1. Response Surface Experimental Design

Drawing on the single-factor results, leaching temperature, leaching time, liquid-to-solid ratio, and sulfuric acid concentration were identified as the principal variables governing vanadium leaching efficiency. These four variables were subsequently investigated using a four-factor, three-level Box–Behnken design (BBD), with vanadium leaching

efficiency as the response. The experimental matrix and corresponding measured responses are presented in Table 3.

Table 3. Experimental design and corresponding results.

No.	Leaching Temperature/°C	Leaching Time/Min	Liquid-to-Solid Ratio/(mL·g ^{−1})	Sulfuric Acid Concentration/wt. %	Vanadium Leaching Efficiency/%
1	60	60	7	14	92.35
2	60	40	6	12	89.62
3	60	50	5	12	89.65
4	70	50	7	14	91.59
5	50	50	6	12	90.06
6	70	50	6	16	92.48
7	60	50	6	14	91.89
8	60	50	6	14	92.16
9	70	60	6	14	91.87
10	70	50	5	14	90.39
11	60	50	5	16	90.36
12	60	50	6	14	92.28
13	50	50	7	14	91.58
14	60	60	6	12	90.28
15	60	50	6	14	92.38
16	70	40	6	14	91.35
17	70	50	6	12	91.39
18	60	50	7	12	90.13
19	60	60	5	14	90.26
20	60	50	7	16	91.88
21	50	50	5	14	90.36
22	60	40	6	16	90.28
23	60	60	6	16	91.89
24	50	40	6	14	89.85
25	60	40	7	14	90.09
26	60	40	5	14	89.93
27	60	50	6	14	92.10
28	50	60	6	14	90.86
29	50	50	6	16	91.76

3.2.2. Regression Model and Analysis of Variance

After fitting a full quadratic model to the experimental data and subsequently simplifying the model, the quadratic regression model shown in Equation (3) was obtained. As indicated by Equation (3), the coefficients of the linear terms for leaching temperature, leaching time, liquid-to-solid ratio, and sulfuric acid concentration are all positive, indicating that, near the center of the investigated experimental range, increasing the levels of these factors generally favors vanadium leaching. The coefficients of the quadratic terms for leaching time, liquid-to-solid ratio, and sulfuric acid concentration are all negative, and these quadratic effects are statistically significant. This indicates that these factors have significant nonlinear effects on vanadium leaching efficiency; that is, as their levels increase, their promoting effects gradually weaken, and the vanadium leaching efficiency may decrease after a certain level is reached.

$$y = 92.16 + 0.38X_1 + 0.53X_2 + 0.56X_3 + 0.63X_4 - 0.12X_1X_2 - 0.15X_1X_4 + 0.48X_2X_3 + 0.24X_2X_4 + 0.26X_3X_4 - 0.23X_1^2 - 0.85X_2^2 - 0.85X_3^2 - 0.70X_4^2 \quad (3)$$

Table 4 summarizes the main statistical indicators of the quadratic model. The coefficient of determination (R^2) of the model is 0.9361, while the adjusted coefficient of determination (R^2_{adj}) and predicted coefficient of determination (R^2_{pred}) are 0.8806 and 0.7354, respectively. The difference between R^2_{adj} and R^2_{pred} is less than the commonly

used criterion of 0.2, indicating acceptable agreement between the two statistics [24]. The R^2_{pred} value indicates reasonable predictive capability of the model within the investigated BBD space, supporting its application for prediction and local optimization within the investigated factor ranges. The relatively low PRESS value of 6.90 further indicates that the model has good predictive performance. In addition, the coefficient of variation is only 0.37%, indicating low dispersion of the experimental results and good stability and repeatability of the data.

Table 4. Fit statistics of the reduced quadratic model.

Std. Dev.	Mean	CV/%	Press	R^2	R^2_{Adj}	R^2_{pred}	Adequate Precision
0.33	91.07	0.37	6.90	0.9361	0.8806	0.7354	10.7025

Table 5 presents the analysis of variance (ANOVA) results for the quadratic model. The model has an F-value of 16.89 and a p -value of less than 0.0001, indicating that the established regression model is statistically significant. The p -value for lack of fit is 0.0967 (>0.05), indicating that the lack of fit is not significant [25]. All four linear terms, X_1 – X_4 , are statistically significant. Among the retained interaction terms, only X_2X_3 is significant ($p = 0.0111$), whereas X_1X_2 , X_1X_4 , X_2X_4 , and X_3X_4 are not significant. Among the quadratic terms, X_2^2 , X_3^2 , and X_4^2 show significant effects, whereas X_1^2 is not significant. Within the investigated BBD range, the F-values of the four linear terms X_1 , X_2 , X_3 , and X_4 are 15.87, 30.62, 33.36, and 42.40, respectively. Based on these F-values, their relative statistical effects decrease in the following order: sulfuric acid concentration (X_4) $>$ liquid-to-solid ratio (X_3) $>$ leaching time (X_2) $>$ leaching temperature (X_1).

Table 5. Analysis of variance for the quadratic model.

Source	Sum of Squares	df	Mean Square	F-Value	p -Value
Model	24.41	13	1.88	16.89	<0.0001
X_1	1.76	1	1.76	15.87	0.0012
X_2	3.4	1	3.4	30.62	<0.0001
X_3	3.71	1	3.71	33.36	<0.0001
X_4	4.71	1	4.71	42.40	<0.0001
X_1X_2	0.06	1	0.06	0.5401	0.4737
X_1X_4	0.093	1	0.093	0.8370	0.3747
X_2X_3	0.9312	1	0.9312	8.38	0.0111
X_2X_4	0.2256	1	0.2256	2.03	0.1747
X_3X_4	0.2704	1	0.2704	2.43	0.1397
X_1^2	0.3511	1	0.3511	3.16	0.0958
X_2^2	4.65	1	4.65	41.81	<0.0001
X_3^2	4.73	1	4.73	42.56	<0.0001
X_4^2	3.2	1	3.2	28.82	<0.0001
Residual	1.67	15	0.1111	/	/
Lack of fit	1.53	11	0.1389	3.99	0.0967
Pure error	0.1393	4	0.0348	/	/
Corrected total	26.07	28	/	/	/

Figure 7a compares the experimental values with the model-predicted values. The data points are generally distributed close to the 45° reference line, indicating good agreement between the predicted and experimental values. In Figure 7b, the externally studentized residuals are randomly distributed on both sides of zero, and all values fall within the critical range of -3.86262 to 3.86262 , with no obvious outliers observed. In addition, the residuals do not exhibit any obvious systematic trend. Together with the model-fitting

statistics presented in Table 4, these results indicate that the quadratic model has reasonable fitting and predictive performance and can be used to predict vanadium leaching efficiency within the investigated BBD range.

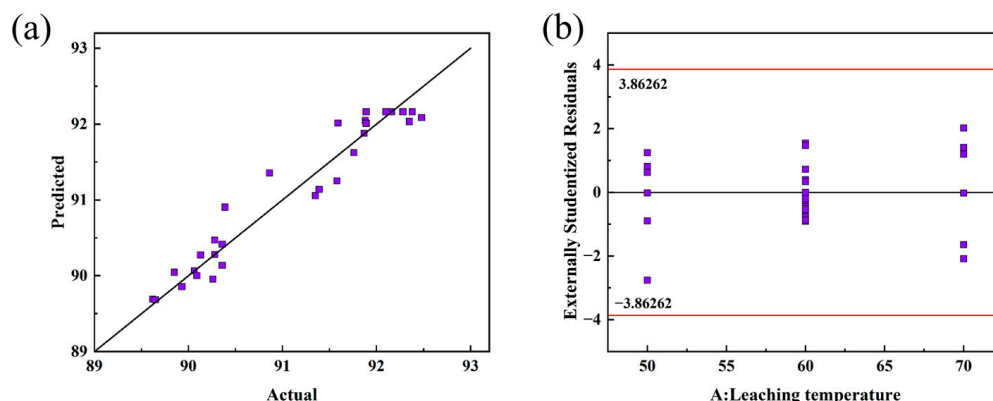


Figure 7. (a) Comparison between the actual and predicted vanadium leaching efficiencies obtained using the reduced quadratic model; (b) externally studentized residuals versus leaching temperature.

3.2.3. Response Surface Analysis

Design-Expert 11.0 was used to generate the contour plots and three-dimensional response surfaces presented in Figures 8–13. These plots show how the predicted response varies over each two-factor domain. A steeper surface generally indicates greater sensitivity to changes in the corresponding variables. An elliptical contour often suggests a stronger interaction, whereas an approximately circular contour is usually associated with a weaker interaction [26–28]. This interpretation is qualitative. The statistical significance of each interaction should therefore be confirmed using the *p*-value of the corresponding interaction term in the analysis of variance.

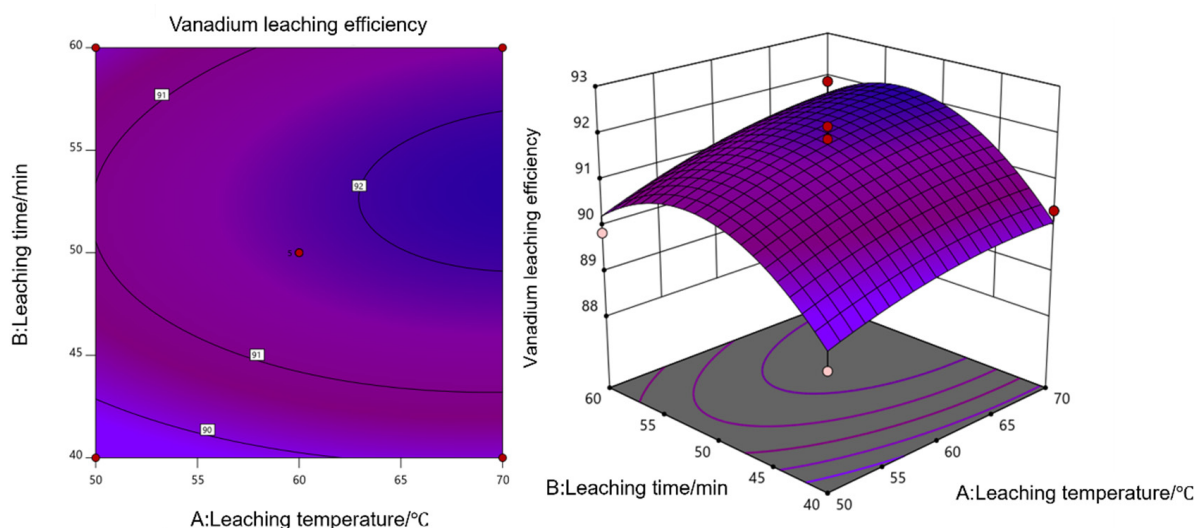


Figure 8. Contour plot and response surface plot showing the interactive effects of leaching temperature and leaching time on the vanadium leaching efficiency. Red and pink dots denote design points with actual responses above and below the predicted values, respectively.

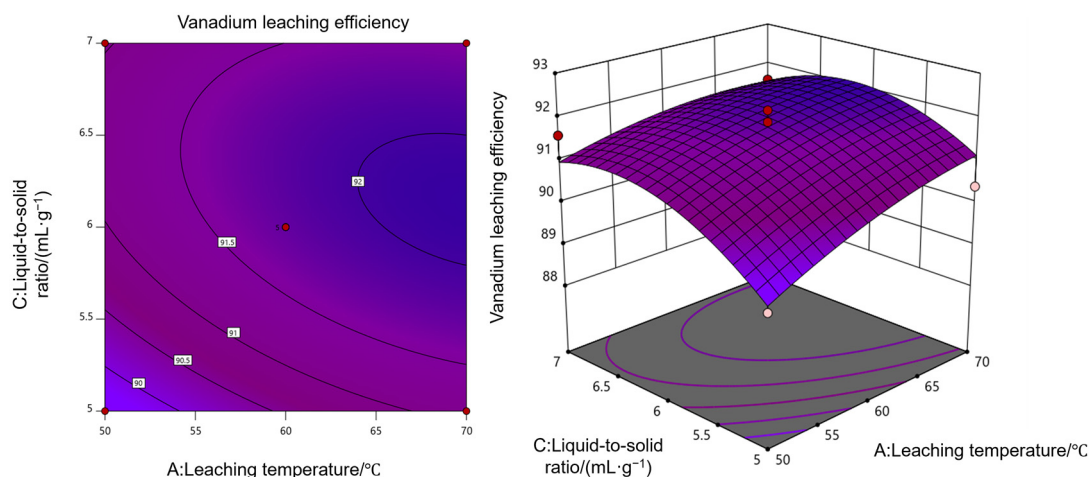


Figure 9. Contour plot and response surface plot showing the interactive effects of leaching temperature and liquid-to-solid ratio on the vanadium leaching efficiency. Red and pink dots denote design points with actual responses above and below the predicted values, respectively.

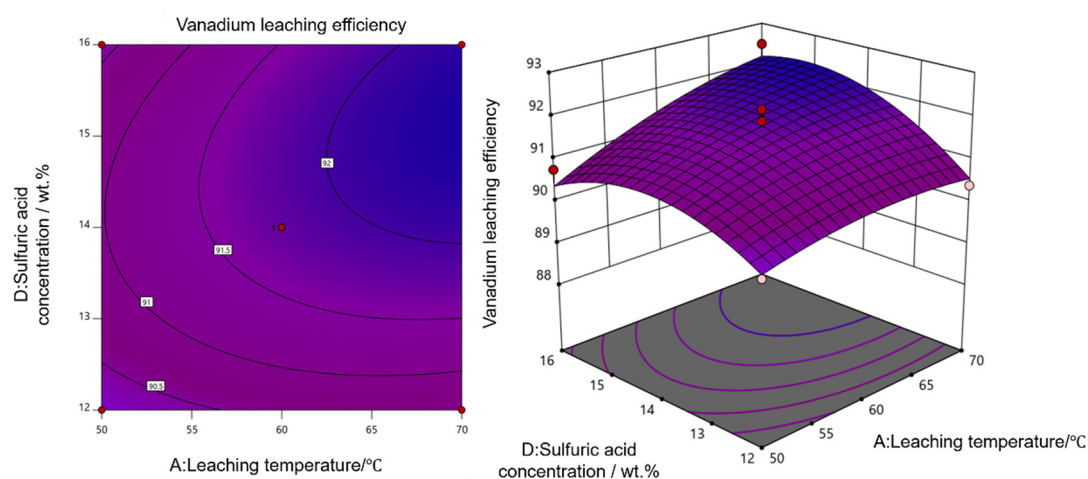


Figure 10. Contour plot and response surface plot showing the interactive effects of leaching temperature and sulfuric acid concentration on the vanadium leaching efficiency. Red and pink dots denote design points with actual responses above and below the predicted values, respectively.

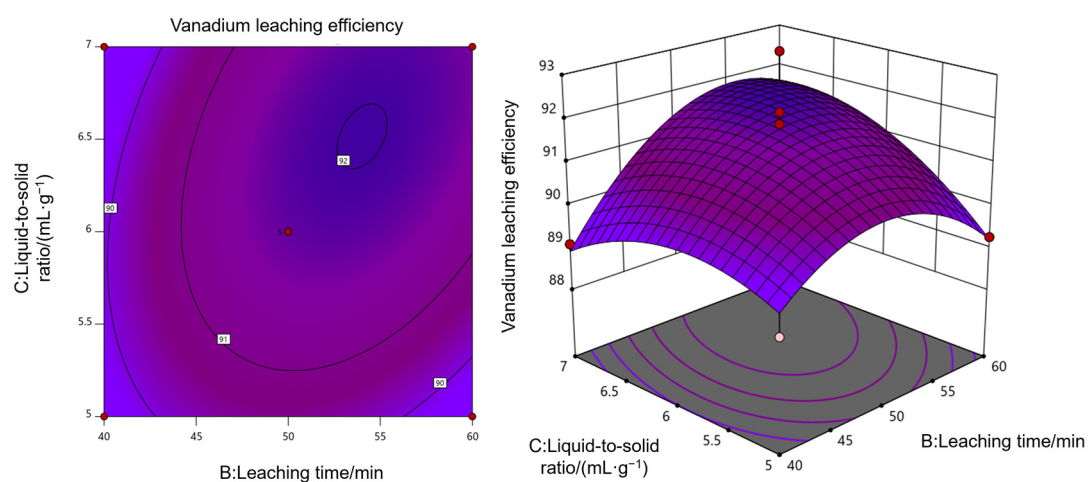


Figure 11. Contour plot and response surface plot showing the interactive effects of leaching time and liquid-to-solid ratio on the vanadium leaching efficiency. Red and pink dots denote design points with actual responses above and below the predicted values, respectively.

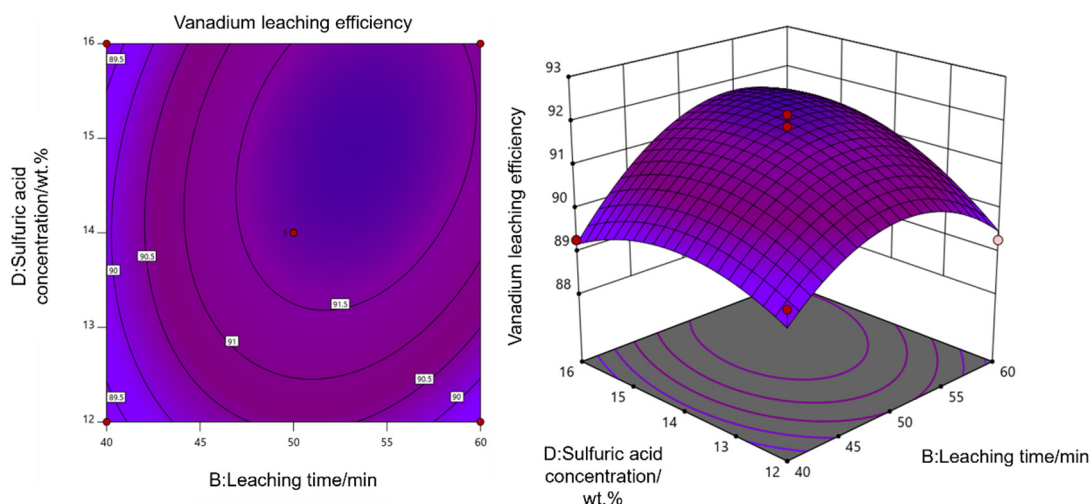


Figure 12. Contour plot and response surface plot showing the interactive effects of leaching time and sulfuric acid concentration on the vanadium leaching efficiency. Red and pink dots denote design points with actual responses above and below the predicted values, respectively.

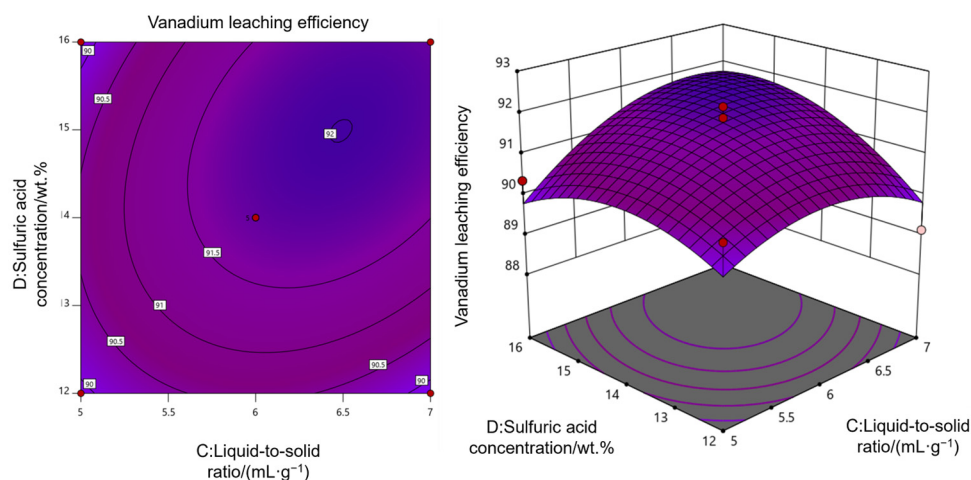


Figure 13. Contour plot and response surface plot showing the interactive effects of liquid-to-solid ratio and sulfuric acid concentration on the vanadium leaching efficiency. Red and pink dots denote design points with actual responses above and below the predicted values, respectively.

Figure 8 displays the contour map and three-dimensional surface for leaching temperature and leaching time. During this analysis, the liquid-to-solid ratio and sulfuric acid concentration were held at their center-point values of 6 mL·g⁻¹ and 14 wt.%. The predicted vanadium leaching efficiency varies along each axis, showing that temperature and time each contribute to the response while the other two factors remain unchanged. Within the ranges of relatively low temperature and short leaching time, the response surface exhibits a comparatively steep slope, indicating that raising leaching temperature and extending leaching time markedly promote vanadium dissolution and leaching from roasted vanadium slag. As leaching temperature rises above 50 °C, vanadium leaching efficiency generally increases, although the magnitude of the improvement gradually diminishes. With an increase in leaching time from 40 min to approximately 50–55 min, the vanadium leaching efficiency initially increases, whereas a further extension of the leaching time results in a subsequent decrease. Relatively high vanadium leaching efficiencies are mainly obtained at leaching temperatures of approximately 60–70 °C and leaching times of approximately 50–55 min. Analysis of variance demonstrates that the linear terms for leaching temperature and leaching time are statistically significant, with leaching time

exerting a stronger influence than leaching temperature. Moreover, the quadratic term of leaching time is highly significant, whereas the interaction between leaching temperature and leaching time is statistically insignificant ($p = 0.4737$), indicating that the curvature observed in the response surface is predominantly attributable to the nonlinear effect of leaching time.

Figure 9 displays the contour map and the surface describing how vanadium leaching efficiency changes across variations in leaching temperature and liquid-to-solid ratio. During this analysis, leaching time remained at 50 min, while sulfuric acid concentration was maintained at 14 wt.%. As illustrated, vanadium leaching efficiency initially increases and subsequently approaches a plateau as leaching temperature and liquid-to-solid ratio rise. Greater curvature along the liquid-to-solid-ratio axis indicates a more pronounced nonlinear effect of this variable, whereas variation along the temperature axis is comparatively gradual. Relatively high vanadium leaching efficiencies are mainly obtained at leaching temperatures of 65–70 °C and liquid-to-solid ratios of 6.0–6.5 mL·g^{−1}. Combined with the analysis of variance, these results demonstrate that the interaction between leaching temperature and liquid-to-solid ratio is statistically insignificant and that the ratio exerts a stronger influence on vanadium leaching efficiency than leaching temperature.

Figure 10 maps the response in vanadium leaching efficiency across the two-factor domain defined by leaching temperature and sulfuric acid concentration, using a contour plot together with a three-dimensional surface. Leaching time remained at 50 min; the liquid-to-solid ratio was held at 6 mL·g^{−1}. Across the 50–70 °C interval, the efficiency followed a generally upward trend, but the overall variation was modest. By comparison, vanadium leaching efficiency rises markedly as the sulfuric acid concentration moves upward from 12 wt.% toward the middle of the tested range. A slight decline then appears at higher acid levels. This reversal reveals a clear nonlinear response to sulfuric acid concentration. The most favorable region lies at 60–70 °C and 14–15 wt.% sulfuric acid. Analysis of variance confirms significant linear contributions from leaching temperature and acid concentration, with the latter exerting the greater influence. Their interaction term, however, is not significant ($p = 0.3747$). The coupling between these variables is therefore weak. Overall, this response pattern agrees well with the trend obtained in the single-factor experiments.

Figure 11 maps the predicted vanadium leaching efficiency across the leaching-time and liquid-to-solid-ratio domain using contour and three-dimensional surface plots. The remaining factors were held at their center points: 60 °C for leaching temperature and 14 wt.% for sulfuric acid concentration. A clear coupling between time and liquid-to-solid ratio emerges from the surface. This interpretation is supported by the analysis of variance, in which the interaction term X_2X_3 is significant ($p = 0.0111$). Thus, the contribution of leaching time changes according to the liquid-to-solid ratio. Extending the leaching period beyond 40 min improves the response until approximately 50–55 min; prolongation to 60 min then causes a decline. The liquid-to-solid ratio follows a similar turning pattern. Raising it from 5 to roughly 6.0–6.5 mL·g^{−1} substantially increases the efficiency, but values approaching 7 mL·g^{−1} reduce the response. The resulting dome-shaped surface reflects pronounced nonlinear behavior for these two variables [29]. Relatively high vanadium leaching efficiencies are primarily obtained at leaching times of approximately 50–55 min and liquid-to-solid ratios of approximately 6.0–6.5 mL·g^{−1}.

Figure 12 characterizes the vanadium-leaching response across the factor space spanned by leaching time and sulfuric acid concentration, using a contour map and a three-dimensional surface. Throughout this analysis, leaching temperature was constrained to 60 °C, whereas the liquid-to-solid ratio remained at 6 mL·g^{−1}. As leaching time extends beyond 40 min and sulfuric acid concentration rises above 12 wt.%, vanadium leaching effi-

ciency initially increases and subsequently decreases. Relatively high vanadium leaching efficiency is obtained at a leaching time of approximately 52–56 min and a sulfuric acid concentration of approximately 14.5–15.5 wt.%; further increases in either variable gradually reduce the response. Because pronounced curvature is observed along both the leaching time and sulfuric acid concentration axes, both variables are considered to exert strong nonlinear effects on vanadium leaching efficiency. Combined with the analysis of variance, the results show that the interaction between leaching time and sulfuric acid concentration is statistically insignificant and that sulfuric acid concentration exerts a slightly stronger influence on vanadium leaching efficiency than leaching time.

Figure 13 maps the predicted vanadium leaching efficiency across the factor domain defined by liquid-to-solid ratio and sulfuric acid concentration, using contour and three-dimensional response-surface representations. Leaching temperature and leaching time were held at their center-point values of 60 °C and 50 min. The response changes markedly along either factor axis. Vanadium leaching efficiency rises sharply as the liquid-to-solid ratio moves from 5 toward approximately 6.0–6.5 mL·g^{−1}, but declines once the ratio approaches 7 mL·g^{−1}. Vanadium leaching efficiency shows a similar rise–fall pattern as the sulfuric acid concentration changes. It increases between 12 wt.% and approximately 14–15 wt.%, but declines at 16 wt.%. Together with the response observed for liquid-to-solid ratio, this behavior produces a dome-shaped surface. The curvature is consistent with significant quadratic effects associated with the two variables [29]. Higher efficiencies occur mainly at liquid-to-solid ratios of 6.0–6.5 mL·g^{−1} and sulfuric acid concentrations of 14–15 wt.%. Analysis of variance confirms significant linear contributions from both variables, with sulfuric acid concentration exerting the greater influence. Their quadratic terms are also significant, whereas the interaction between them remains weak.

3.2.4. Model Validation and Local Optimization of Leaching Conditions

Numerical optimization of the reduced quadratic model using Design-Expert 11.0 identified a model-predicted local optimum within the investigated BBD space at 64.95 °C, 55.21 min, a liquid-to-solid ratio of 6.56 mL·g^{−1}, and a sulfuric acid concentration of 15.17 wt.%, with a predicted vanadium leaching efficiency of 92.73%. Three replicate validation experiments were conducted under the above conditions, yielding vanadium leaching efficiencies of 92.89%, 92.92%, and 92.96%, with an average value of 92.92%. The experimental results are summarized in Table 6. Under the validation conditions, the reduced model predicted a vanadium leaching efficiency of 92.73%, corresponding to a relative error of 0.205% compared with the mean value of the replicate experiments. These results indicate good reliability of the vanadium leaching efficiency measurements and also demonstrate that the reduced quadratic regression model has good local predictive capability within the identified high-leaching-efficiency region.

Table 6. Experimental results obtained under the model-predicted local optimum conditions.

No.	Experimental Conditions				Vanadium Leaching Efficiency /%
	Leaching Temperature /°C	Leaching Time /min	Liquid-to-Solid Ratio /(mL·g ^{−1})	Sulfuric Acid Concentration /wt. %	
1	64.95	55.21	6.56	15.17	92.89
2	64.95	55.21	6.56	15.17	92.92
3	64.95	55.21	6.56	15.17	92.96

3.3. Phase Composition and Morphological Characterization of the Leaching Residue

Figure 14 summarizes the phase composition and morphology of the leaching residue. Figure 14a shows the XRD pattern of the leaching residue after acid leaching. The main crystalline phases in the leaching residue included $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, Fe_2O_3 , SiO_2 , Fe_2TiO_5 , CrVO_3 , and $\text{CaVH}_2\text{Si}_4\text{O}_{12}$. Compared with the roasted vanadium slag shown in Figure 1, $\text{Mn}_2\text{V}_2\text{O}_7$ was no longer detected after leaching, indicating that this phase was largely dissolved during sulfuric acid leaching. The characteristic peaks of $\text{Ca}(\text{Fe,Mg})\text{Si}_2\text{O}_6$ also became indistinct in the leaching residue, whereas diffraction peaks corresponding to SiO_2 and pronounced $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ appeared after leaching. This indicates that some Ca-containing silicate phases decomposed during acid leaching, accompanied by the formation and precipitation of gypsum. The precipitated $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ may deposit on the surfaces of solid particles and increase mass-transfer resistance, thereby limiting the further dissolution of residual vanadium-bearing phases. Fe_2O_3 and Fe_2TiO_5 remained present after leaching, indicating that their dissolution was limited under the current leaching conditions. Small amounts of CrVO_3 and $\text{CaVH}_2\text{Si}_4\text{O}_{12}$ were also present in the roasted vanadium slag, and their presence in the leaching residue is mainly attributed to the incomplete dissolution of the original vanadium-bearing phases. These results indicate that $\text{Mn}_2\text{V}_2\text{O}_7$ is relatively readily leached in sulfuric acid solution, whereas part of the vanadium remains in CrVO_3 and $\text{CaVH}_2\text{Si}_4\text{O}_{12}$ phases that are more difficult to dissolve completely.

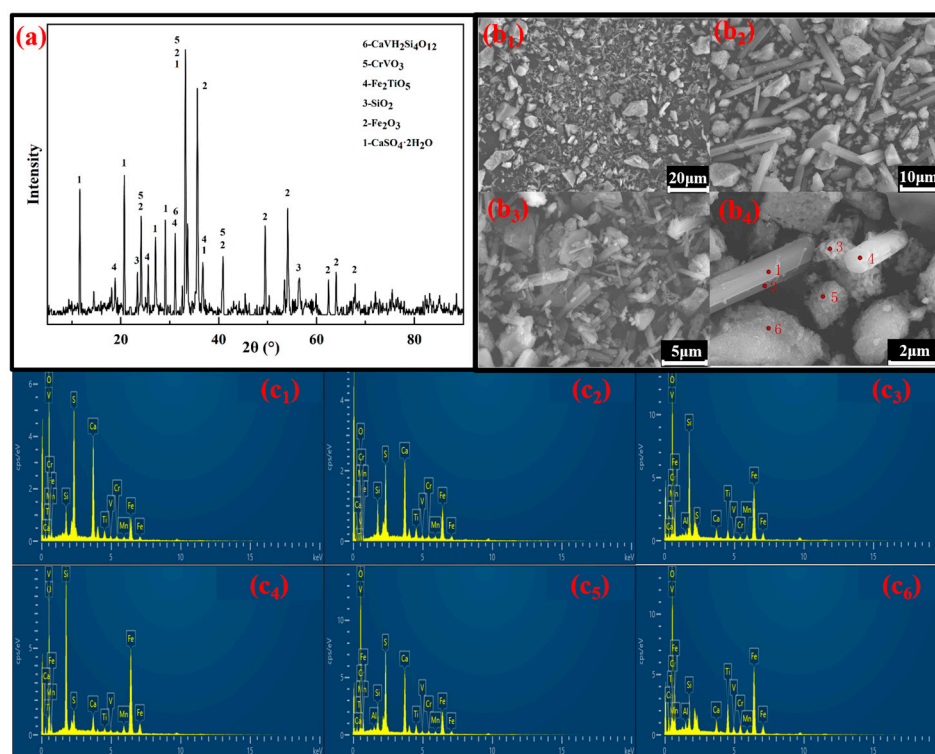


Figure 14. Phase assemblage and morphological features of the leaching residue obtained from calcification-roasted vanadium slag: (a) XRD profile of the leaching residue; (b₁–b₄) SEM micrographs of the leaching residue, points 1–6 indicate the areas selected for elemental analysis; and (c₁–c₆) EDS spectra collected from the corresponding regions of the leaching residue.

Figure 14(b₁–b₄) show SEM images of the leaching residue with scale bars of 20, 10, 5, and 2 μm, respectively, providing progressively higher-resolution views of the residue morphology. Figure 14(b₁) contains irregular particles of different sizes, together with acicular and elongated plate-like crystals. Cracks are visible on several particle surfaces. These features may result from the partial dissolution of acid-soluble phases, which weakened

the original roasted vanadium slag structure. Large quantities of acicular and elongated plate-like crystals were observed in Figure 14(b₂,b₃). Based on the corresponding scale bars, these crystals were approximately 10–30 µm in length and about 1 µm in width. In combination with the CaSO₄·2H₂O phase identified by XRD, these crystals are inferred to be gypsum formed during the leaching process. According to the presence of calcium-bearing mineral phases, such as Ca(Fe,Mg)Si₂O₆, in the roasted vanadium slag shown in Figure 1, it is inferred that calcium-bearing silicates and other acid-soluble calcium-containing phases were attacked by sulfuric acid and released Ca²⁺ into the leachate. The released Ca²⁺ subsequently combined with SO₄^{2−}, after which CaSO₄·2H₂O nucleated and crystallized on the surfaces of the roasted vanadium slag particles. Simultaneously, acid-soluble vanadium-bearing phases, such as Mn₂V₂O₇, may have been decomposed, resulting in the transfer of vanadium from the solid phase into the leachate. The interlaced growth of the acicular and plate-like crystals on the particle surfaces may block internal pores and consequently increase the diffusional resistance of the leaching products.

Figure 14(c₁–c₆) and Table 7 present the EDS results obtained from different regions of the leaching residue. The results show that Points 1, 2, and 5 contain relatively high concentrations of O, Ca, and S, together with certain amounts of Fe. The Ca/S mass ratios at Points 1 and 5 are approximately 1.33 and 1.27, respectively, which are close to the theoretical Ca/S mass ratio of CaSO₄·2H₂O. This result further supports the XRD analysis and indicates that these regions are mainly composed of calcium sulfate crystals, possibly accompanied by minor amounts of iron oxides. Points 3 and 4 are enriched mainly in Fe and Si, together with lower concentrations of several other elements, indicating that these regions are predominantly composed of refractory phases such as Fe-rich silicates. Based on the XRD results and the relative contents of the detected elements, Point 6 is inferred to consist mainly of Fe₂O₃ and Fe₂TiO₅, together with small amounts of Si-containing phases, and may also contain small amounts of residual CrVO₃ and CaVH₂Si₄O₁₂. The persistent presence of residual CrVO₃ and CaVH₂Si₄O₁₂ indicates that a small amount of vanadium remains in incompletely leached solid phases. In addition, the relatively refractory Fe- and Si-containing matrix and the deposition of CaSO₄ crystals on particle surfaces may hinder acid penetration and mass transfer, thereby further limiting the dissolution of residual vanadium-bearing phases. The absence of an obvious S signal at Point 6 may mainly be because this point is located in a Fe- and Ti-rich residual particle region rather than in a sulfate-rich crystalline region.

Table 7. Elemental compositions of selected regions in the leaching residue produced under the model-predicted local optimum conditions.

EDS Analysis Point	Elemental Content (wt.%)									
	O	Si	Fe	Ca	Ti	V	Mn	Cr	S	Al
1	53.26	2.43	10.91	16.63	1.98	0.68	0.97	0.67	12.46	N.D.
2	39.60	4.57	20.90	17.84	3.47	1.01	1.26	1.21	10.15	N.D.
3	41.28	14.80	34.32	1.90	2.77	0.76	2.26	0.88	0.31	0.72
4	29.09	19.16	42.17	2.80	0.89	0.67	3.00	N.D.	2.22	N.D.
5	55.52	3.02	13.33	12.58	2.47	0.56	1.11	1.06	9.94	0.40
6	40.97	4.33	38.05	2.06	8.11	0.95	1.54	2.86	N.D.	1.14

Note: N.D. indicates that the corresponding element was not detected in the EDS spectrum.

4. Conclusions

This study combined single-factor experiments, Box–Behnken design, and leaching-residue characterization to investigate the sulfuric acid leaching behavior of microwave-assisted calcification-roasted vanadium slag. A local optimum within the investigated BBD range was determined, and the phase composition and microstructural characteristics of the leaching residue were analyzed. Future work may further consider actual industrial operating conditions to provide theoretical support for industrial application. The main conclusions of this study are as follows:

- (1) For the vanadium slag obtained by microwave-assisted calcification roasting, vanadium leaching efficiency increased as leaching temperature rose, whereas further improvement above 60 °C was negligible. It initially increased with leaching time, stabilized after 50 min, and then decreased slightly. The liquid-to-solid ratio had a moderate effect on vanadium leaching efficiency, with little additional improvement observed above 6 mL·g^{−1}. Sulfuric acid concentration showed a pronounced effect in the lower-concentration region, with vanadium leaching efficiency increasing from 77.52% at 4 wt.% to 92.25% at 14 wt.%, whereas further increases above 14 wt.% produced only marginal improvement. Agitation markedly enhanced liquid–solid contact and mass transfer relative to the unstirred condition, whereas agitation speeds exceeding 200 rpm yielded little further improvement.
- (2) Within the investigated BBD space, ANOVA of the reduced quadratic model indicated that the statistical effects of the four linear terms followed the order sulfuric acid concentration > liquid-to-solid ratio > leaching time > leaching temperature, while only the interaction between leaching time and liquid-to-solid ratio was statistically significant. Within the investigated BBD design range, the model identified a local optimum at 64.95 °C, 55.21 min, a liquid-to-solid ratio of 6.56 mL·g^{−1}, and a sulfuric acid concentration of 15.17 wt.%, with a predicted vanadium leaching efficiency of 92.73%. The validation experiments yielded an average leaching efficiency of 92.92%, with a relative error of 0.205% compared with the model prediction, supporting the reliability of the model prediction at the identified local optimum within the investigated BBD design space.
- (3) The leaching residue contained irregular particles together with acicular and plate-like crystals. The crystals were attributed mainly to CaSO₄·2H₂O, whereas the irregular particles were associated with residual Fe- and Si-containing phases, including Fe₂O₃, SiO₂ and Fe₂TiO₅. Mn₂V₂O₇ was no longer detected after leaching, suggesting substantial dissolution or transformation of this phase; however, minor CrVO₃ and CaVH₂Si₄O₁₂ remained in the residue. The deposition of CaSO₄·2H₂O on particle surfaces and the retention of vanadium in refractory phases likely increased transport resistance, thereby limiting further vanadium leaching.

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