Leaching Kinetics of Vanadium from Calcium-Roasting High-Chromium Vanadium Slag Enhanced by Electric Field

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**ABSTRACT:** Vanadium exists as multivalent valences in high-chromium vanadium slag, and it is hard to leach out in low valence. Electro-oxidation technology has been applied to enhance the leaching process of calcium-roasting high-chromium vanadium slag. The effect of parameters that affect the leaching efficiency of vanadium including concentration of sulfur acid, current density, reaction temperature, and liquid-to-solid ratio was investigated. The results showed that vanadium in low valence could be oxidized and efficiently leached out enhanced with electricity. The leaching kinetics was analyzed, which indicates that the leaching rate was controlled by the surface chemical reaction with an apparent activation energy of 40.11 kJ/mol. On the basis of this process, vanadium could be efficiently leached out with a leaching efficiency of 92.14% under optimal conditions: concentration of sulfur acid of 40 vol %, current density of 750 A/m², reaction temperature of 90 °C, reaction time of 180 min, particle size under 75 μm, liquid-to-solid ratio of 4:1 mL/g, and stirring rate of 500 rpm. The relationship between the leaching efficiency and the parameters affecting the leaching process could be described as $1 - (1 - x)^{1/3} = K_0 \times \left[H_2SO_4\right]^{0.1390} \times \left[J\right]^{0.03354} \times \left[T\right]^{1.2847} \times \left[L/S\right]^{-0.2598} \times \exp^{40.11/8} \times t$.

1. INTRODUCTION

Vanadium and its compounds are widely used in petrochemical industry, catalyst, and iron and steel because of their excellent physicochemical properties. In China, the main resources for vanadium recovery are vanadium titanomagnetite, uranium-bearing sandstone, bauxite, stone coal, and so forth. A byproduct named high-chromium vanadium slag (HCVS) produced during smelting with high-chromium vanadium titanomagnetite has attracted more attention as it could be consumed in the acidic leaching process. The effect of parameters including concentration of H₂SO₄, current density, reaction temperature, and liquid-to-solid ratio has been investigated. The leaching kinetics behavior for leaching out vanadium has also been discussed.

In this paper, calcium-roasting and electro-oxidation technology have been coupled to treat HCVS in the acidic leaching process. The effect of parameters including concentration of H₂SO₄, current density, reaction temperature, and liquid-to-solid ratio has been investigated. The leaching kinetics behavior for leaching out vanadium has also been discussed.

Received: May 11, 2020
Accepted: June 26, 2020
Published: July 8, 2020

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2. RESULTS AND DISCUSSION

2.1. Leaching Process. The concentration of H₂SO₄ plays an important role during the leaching process as the experiments were conducted in acidic medium. The leaching efficiency of vanadium is significantly affected by the concentration of H₂SO₄. A series of experiments were investigated under the standard conditions: reaction temperature of 90 °C, particle size under 75 μm, liquid-to-solid ratio of 4:1 mL/g, and stirring rate of 500 rpm. The results shown in Figure 1 indicate that the leaching efficiency of vanadium increased with the increase in the concentration of H₂SO₄ as it ranged from 10 to 40 vol % (volume percentage of H₂SO₄). Vanadium in HCVS exists as multivalent valences; the vanadium in high valence was easily dissolved in the acidic medium and leached out, whereas the vanadium in low valence was hard to leach out, directly resulting in a low leaching efficiency (below 65%). Scanning electron microscopy (SEM) of HCVS before and after leaching (Figure 3) displayed that many villous particles were adsorbed on the surface of the leaching cake, which might block the pore channels and decrease the permeability of the slag particle mass, leading to the low leaching efficiency of vanadium. In order to improve the leaching efficiency, some enhancing technology is needed during the leaching process.

In order to enhance the leaching process and improve the leaching efficiency of vanadium, some oxidative leaching processes with MnO₂, H₂O₂, or KClO₃ were investigated, and a high leaching efficiency was achieved. Electro-oxidation technology as an environmental-friendly technology had been widely applied in enhancing the leaching process and achieved a high leaching efficiency of vanadium. In this study, we used electro-oxidation technology to improve the leaching process.

![Figure 1. Effect of acid concentration without electric field on the leaching efficiency of vanadium.](image1)

![Figure 2. Effect of parameters on the leaching efficiency of vanadium. (a) Current density; (b) acid concentration with electric field; (c) leaching temperature with electric field; (d) liquid-to-solid ratio with electric field.](image2)

![Figure 3. SEM of slag (a) before leaching and (b) after leaching.](image3)
paper, electro-oxidation technology has been used to enhance the leaching process to improve the leaching efficiency of vanadium at standard conditions: reaction temperature of 90 °C, liquid-to-solid ratio of 4:1 mL/g, concentration of H$_2$SO$_4$ of 40 vol %, particle size under 75 μm, and stirring rate of 500 rpm. The leaching efficiency of vanadium had a significant increase enhanced with electric field according to the results shown in Figure 2a. During the electro-oxidation leaching process, two kinds of “reactive oxygen” (hydroxyl radical chemisorbed oxygen atom) adhered on the anode surface (seen in eqs 1, 2 and Figure 4) within the electric field.\textsuperscript{32,36,38–40} The vanadium in low valence could be oxidized resulting in a high leaching efficiency (eqs 3 and 4). Nearly 92.14% vanadium could leach out at a current density of 750 A/m$^2$, which was much lower than 1000 A/m$^2$ for vanadium leaching with sodium-roasting alkaline leaching process.\textsuperscript{32} Further increase in the current density had no increase in the leaching efficiency of vanadium but a slight decrease due to anode corrosion.\textsuperscript{57} Thus, the current density of 750 A/m$^2$ was selected as the optimal condition for further experiments. The results in Figure 2b shows that the leaching efficiency of vanadium was much improved within the application of electric field in the same acid concentration compared with the results shown in Figure 1.

\begin{align*}
H_2O & \rightarrow H_2 + O_2 \tag{1} \\
H_2O & \rightarrow \cdot OH \tag{2} \\
Ca_2V_2O_7 + O_2/\cdot OH + H_2SO_4 & \rightarrow CaSO_4 + H_2O + (VO_2)_2SO_4 \tag{3}
\end{align*}

\[ Ca_2V_2O_7 + H_2SO_4 \rightarrow CaSO_4 + H_2O + (VO_2)_2SO_4 \tag{4} \]

The effect of reaction temperature on the leaching efficiency of vanadium was studied under the standard conditions: liquid-to-solid ratio of 4:1 mL/g, current density of 750 A/m$^2$, concentration of H$_2$SO$_4$ of 40 vol %, particle size under 75 μm, and stirring rate of 500 rpm. It can be seen from Figure 2c that the leaching efficiency of vanadium was promoted obviously as the reaction temperature increased and reached up to 92.14% at the reaction temperature of 90 °C. Increasing the reaction temperature could increase the activity of atoms and molecules, enforce the reaction intensity, and promote the reactions and thus was beneficial for leaching out vanadium.\textsuperscript{40} Higher reaction temperature did not lead to a high leaching efficiency of vanadium. Therefore, the reaction temperature of 90 °C was chosen as the optimal condition for further experiments.

During the leaching process, the volume of reaction medium affects the solution viscosity and the solid–liquid mass transfer. A series of experiments were conducted to evaluate an optimal liquid-to-solid ratio for a high leaching efficiency of vanadium. Other conditions, such as the concentration of H$_2$SO$_4$, current density, reaction temperature, particle size, and stirring rate, were kept constant at 40 vol %, 750 A/m$^2$, 90 °C, under 75 μm, and 500 rpm, respectively. The results are plotted in Figure 2d, and surprisingly, it was observed that the liquid–solid ratio played a negligible role in the dissolution of vanadium, as indicated by the slight increase of the vanadium leaching efficiency from 78.32 to 92.14% when the liquid-to-solid ratio increased from 2 to 4 mL/g. Such a result is possibly because the particle size selected for the experiments was small enough for the homogenous mixing of the slurry, and when the HCVS particles were in good contact with an excessive amount of leaching agent, the leaching process was mostly determined by the typical thermodynamic parameters including current density and reaction temperature but less by the kinetics parameters, including the liquid-to-solid ratio. For the sake of energy saving, 4 mL/g was chosen as the optimal liquid-to-solid ratio in the process.

From the above analysis, it was found that the effects of current density and reaction temperature were much more significant than other factors. The results suggested that the increase of reaction temperature and concentration of H$_2$SO$_4$ was beneficial for leaching out vanadium. The leaching efficiency of vanadium was up to 92.14% under the optimal conditions: concentration of H$_2$SO$_4$ of 40 vol %, current density of 750 A/m$^2$, reaction temperature of 90 °C, reaction...
time of 180 min, particle size under 75 μm, liquid-to-solid ratio of 4:1 mL/g, and stirring rate at 500 rpm.

2.2. Characterization of Slag. X-ray diffraction (XRD) patterns of original HCVS and roasted HCVS at different roasting temperatures were investigated to analyze the phase change, and the results are shown in Figure 5. It was visibly revealed that the main characteristic peaks were FeV2O4 and CaFe(Si2O6) in the original HCVS, which indicated that vanadium existed as V(III) in the slag. The X-ray photoelectron spectroscopy (XPS) results detailed in Table 1 show that vanadium in the slag mainly existed as V(III) and V(V), whereas V(IV) only accounted 18.44%. That is to say, some vanadium oxides in the HCVS were in an amorphous form which could not detected by XRD. During the calcium-roasting process, new phases such as Ca2V2O5 and Ca2V2O7 were formed (eqs 5-8). When the roasting temperature was below 450 °C, there were no new phases formed. The XPS results (Figure 6) show that 9.55% V(III) existed in the roasted HCVS, and V(IV) and V(V) accounted for 34.12 and 56.33%, respectively. Most of the low-valence vanadium was oxidized to V(IV) and V(V) during the calcium-roasting process and formed as Ca2V2O5 and Ca2V2O7. Then, all kinds of vanadium were leached out in the electro-oxidation acid leaching process, and almost all V(IV) was oxidized to V(V) and leached out. The whole recovery of vanadium was about 92.14%.

2.3. Kinetics Analysis. The leaching kinetics of vanadium from HCVS was investigated in order to know the controlling step during the leaching process. The shrink core model was widely used to describe the kinetics, where a reaction between solid and liquid reactants occurs on the outer surface of the solid. The kinetic model was described by three equations which are detailed in Table 2.

Table 1. Percentage of Various Valence Vanadium (%)

|                  | V(III) | V(IV) | V(V) |
|------------------|--------|-------|------|
| original HCVS    | 39.27  | 18.44 | 42.29|
| roasted HCVS     | 9.55   | 34.12 | 56.33|
| leaching solution| 8.42   | 91.58 |      |

In conclusion, the recovery of vanadium followed two steps: Most V(III) was oxidized to V(IV) and V(V) during the calcium-roasting process and formed as Ca2V2O5 and Ca2V2O7. Then, all kinds of vanadium were leached out in the electro-oxidation acid leaching process, and almost all V(IV) was oxidized to V(V) and leached out. The whole recovery of vanadium was about 92.14%.

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Table 2. Kinetic Models and Equations

| Controlling Step | Equations |
|------------------|-----------|
| Diffusion        | \( K_1 \times t = x \) |
| Product layer    | \( K_2 \times t = 1 - 2/3 \times x - (1 - x)^{2/3} \) |
| Surface reaction | \( K_3 \times t = 1 - (1 - x)^{2/3} \) |

where \( x \) is the leaching efficiency of vanadium; \( K_1, K_2, \) and \( K_3 \) are the apparent rate constants for each kinetic model, min\(^{-1}\); \( t \) is the reaction time, min.

The experimental results obtained from the leaching process were analyzed based on the three equations displayed in Table 2. The fitting results showed in Table 3 indicate that eq 11 fitted the experimental data well (correlation coefficients were large among the three equations and very close to 1), which indicated that eq 11 was more suitable to demonstrate the leaching process of vanadium from HCVS. Therefore, the controlling step of the leaching process was determined as the surface chemical reaction between the HCVS and the concentrated H\(_2\)SO\(_4\) solution.

The reaction rates and the apparent rate constant at different reaction temperatures (in Kelvin) were calculated, and the results are expressed in Figure 7a. Also, the apparent activation energy for leaching out vanadium was calculated based on the Arrhenius equation (eq 12, results shown in Figure 7b). The apparent activation energy was calculated as 40.11 kJ/mol, and the result was consistent with the model equation described in eq 11.63

\[ \ln K = \ln A - \frac{E_A}{RT} \]
where $E_a$ is the apparent activation energy, $A$ is the pre-exponential factor, and $R$ is the molar gas constant.

The leaching process was significantly affected by the parameters including the concentration of H$_2$SO$_4$, current density, reaction temperature, and liquid-to-solid ratio according to the results shown in Figure 2. The relationship between the apparent rate constant and these factors is provided as eq 13, and it could be determined as the fitting plot of $1 - (1 - x)^{1/3}$ versus time for different concentrations of H$_2$SO$_4$, current density ($J$), reaction temperature ($T$), and liquid-to-solid ratio ($L/S$).

### Table 3. Apparent Rate Constants $K_1$, $K_2$, and $K_3$ and Correlation Coefficients

| Temperature ($^\circ$C) | $K_1$ (min$^{-1}$) | $R^2$ | $K_2$ (min$^{-1}$) | $R^2$ | $K_3$ (min$^{-1}$) | $R^2$ |
|--------------------------|---------------------|-------|---------------------|-------|---------------------|-------|
| 50                       | 0.000862            | 0.9292| 0.000058            | 0.9488| 0.000348            | 0.9924|
| 60                       | 0.001690            | 0.9389| 0.000207            | 0.9324| 0.000791            | 0.9938|
| 70                       | 0.002540            | 0.9121| 0.000597            | 0.9115| 0.001560            | 0.9929|
| 80                       | 0.003900            | 0.9377| 0.000663            | 0.9545| 0.001480            | 0.9943|
| 90                       | 0.002050            | 0.9245| 0.000921            | 0.9800| 0.001950            | 0.9932|

**Figure 7.** Kinetics plots: (a) Plot of leaching kinetics of vanadium at various reaction temperatures; (b) natural logarithm of reaction rate constant vs reciprocal temperature.

**Figure 8.** Plot of $K$ as a function of reaction parameters: (a) acid concentration; (b) current density; (c) reaction temperature; (d) liquid-to-solid ratio.
The leaching kinetics equation is described as follows

\[
K = K_0 \times [H_2SO_4]^{0.1390} \times [J]^{0.03354} \times [T]^{2.8247} \times [L/S]^{0.2598} \times \exp\left(\frac{-E_a}{RT}\right) \times t
\]

(13)

After the transformation, 11 were obtained:

\[
1 - (1 - x)^{1/3} = K_0 \times [H_2SO_4]^{0.1390} \times [J]^{0.03354} \times [T]^{2.8247} \times [L/S]^{0.2598} \times \exp\left(\frac{-E_a}{RT}\right) \times t
\]

(14)

In K versus ln[H2SO4], ln[J], ln[T], and ln[L/S] plots (Figure 8) were established to determine the order of dependency with respect to the concentration of H2SO4, current density, reaction temperature, and liquid-to-solid ratio. The empirical reaction orders were calculated as 0.1390, 2.8247, and −0.2598. Therefore, the leaching kinetics equation is described as follows

\[
1 - (1 - x)^{1/3} = K_0 \times [H_2SO_4]^{0.1390} \times [J]^{0.03354} \times [T]^{2.8247} \times [L/S]^{0.2598} \times \exp\left(\frac{-E_a}{RT}\right) \times t
\]

(15)

3. CONCLUSIONS

Electro-oxidation technology was introduced to enhance the leaching process of vanadium from the calcium-roasting high-chromium vanadium slag. The following conclusions were obtained:

(1) Vanadium existed as multiple valences in the HCVS. After calcium roasting, most V(III) was oxidized to V(IV) and V(V) and existed as Ca2V2O5 or Ca2V3O7.

(2) Vanadium could be efficiently leached out during electro-oxidation acidic leaching process. On the basis of this process, vanadium could be effectively leached out with a leaching efficiency of 92.14% under the optimal conditions: concentration of H2SO4 of 40 vol %, current density of 750 A/m², reaction temperature of 90 °C, particle size under 75 μm, reaction time of 180 min, liquid-to-solid ratio of 4:1 mL/g, and stirring rate of 500 rpm.

(3) The leaching kinetics behavior for vanadium leaching out was analyzed. The result showed that the leaching process followed the shrink core model, and the leaching process was controlled by the surface chemical reaction, with the apparent activation energy of 40.11 kJ/mol. The relationship between the leaching efficiency and the parameters affecting the leaching process could be described as

\[
1 - (1 - x)^{1/3} = K_0 \times [H_2SO_4]^{0.1390} \times [J]^{0.03354} \times [T]^{2.8247} \times [L/S]^{0.2598} \times \exp\left(\frac{-E_a}{RT}\right) \times t
\]

4. EXPERIMENTAL SECTION

4.1. Materials. The HCVS used in the experiments was collected from Pangang Group Co., Ltd., Panzhihua, China. The slag was dried and ground to fine particles before each experiment. The detailed chemical composition of the slag is detailed in Table 4. All chemical reagents used in this study were of analytical grade and purchased from Kelong Chemical Reagent Co., Ltd., Chengdu, China and used without further purification.

4.2. Roasting Procedure. The HCVS was pretreated with calcium roasting before leaching. The slag was mixed with CaO at a mole ratio of n(CaO)/n(V2O5) = 1.1 and then roasted in the muffle furnace at different temperatures.

4.3. Leaching Procedure. All experiments were carried out in a 500 mL glass beaker in a thermostatic mixing water bath. The experimental setup is detailed in Figure 9. On the top of the beaker, there were two electrodes, and both of them were Pb–Ag–Ca–Sr alloy; the space between the anode and the cathode was 0.04 m, and the working area of the electrode was 8 cm². Before the experiments, a predetermined concentration of H2SO4 solution was added to the beaker, and then the roasted HCVS was added when the determined temperature was reached. Then, the power supply was turned on and adjusted to appropriate currents. The leaching solution was separated from the slag by vacuum filtration after the required reaction time.

4.4. Analysis Method. The concentration of vanadium in the leaching solution was determined by inductively coupled plasma-optical emission spectrometry (PerkinElmer Optima 6300DV), and the leaching efficiency of vanadium (x) was calculated with the following eq

\[
x = \frac{V \times C}{m \times \omega} \times 100\%
\]

(16)

where C is the concentration of vanadium in the leaching solution, g/L; V is the volume of the filtrate, mL; ω is the mass fraction of vanadium in the HCVS; m is the mass of HCVS used in the leaching experiments, g.

The element compositions of HCVS were examined with X-ray fluorescence (Shimadzu Lab Center XRF-1800, Japan), and the mineralogical compositions were determined by XRD (Shimadzu Lab Center XRD-6000, Japan). Vanadium valence distribution in the samples was analyzed by XPS (ESCALAB-250Xi, USA).

Table 4. Composition of the HCVS (wt %)

| composition | V2O5 | Cr2O3 | FeO | CaO | MgO |
|-------------|------|------|-----|-----|-----|
| counts (wt %) | 9.7  | 10.2 | 24.7 | 2.4 | 13.89 |
| composition | SiO2 | Al2O3 | MnO | TiO2 |
| counts (wt %) | 25.76 | 10.3 | 1.61 | 2.78 |

Figure 9. Experimental setup.
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Notes
The authors declare no competing financial interest.

ACKNOWLEDGMENTS
This work was supported by the National Natural Science Foundation of China (no. 51804062), the Science and Technology Research Program of Chongqing Municipal Education Commission (no. KJQN201901403), and the Chongqing Science and Technology Commission (no. cstc2018jcyjAX0018).

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