



Leaching of vanadium and chromium from converter vanadium slag intensified with surface wettability

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Abstract: Technology intensified with surface wettability was introduced to leach vanadium and chromium from converter vanadium slag without roasting. Parameters affecting the leaching efficiency of vanadium and chromium were investigated: sulfuric acid concentration, MnO₂-to-slag mass ratio, liquid-to-solid ratio, leaching time, leaching temperature, and sodium dodecyl sulfate (SDS)-to-slag mass ratio. The leaching efficiencies of vanadium and chromium were 33.46 % and 20.02 % higher in the presence of MnO₂ and SDS, respectively, compared to the control. The leaching efficiencies of vanadium and chromium were 68.93 % and 30.74 %, respectively, under the optimum conditions: sulfuric acid concentration 40 wt%, MnO₂-to-slag mass ratio 10.0 wt%, liquid-to-solid ratio 5:1 mL/g; 12 h; 90 °C; and SDS-to-slag mass ratio 0.25 wt%. The analysis of the reaction mechanism in the leaching process indicates that MnO₂ combined with protons (H⁺) could oxidize low-valent vanadium and chromium; SDS could change the chemical behavior and decrease the surface tension of the aqueous solution to favor MnO₂ oxidation.

Key words: vanadium; chromium; leaching; surfactant; MnO₂

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1 Introduction

Vanadium is an important strategic metal that is used in the manufacture of iron and steel and non-ferrous metals, and in the petrochemical industry because of its excellent physicochemical properties, such as tensile strength, hardness, and fatigue resistance [1–5]. In China, vanadium slag is typically produced by oxidation in the steelmaking process, representing a major vanadium resource [6–8]. Technologies have been developed to extract vanadium from converter vanadium slag, including

direct leaching, roasting leaching, sub-molten salt leaching, and electro-oxidation leaching [9–12]. Roasting and leaching methods involve calcium salt roasting–acidic leaching and sodium salt roasting–water leaching processes. These processes require high temperatures and involve energy consumption. In particular, the sodium salt roasting–water leaching process causes serious environmental pollution in the form of poisonous gas and wastewater production [13–17]. Sub-molten salt technology has been applied to extracting valuable metals from treated amphoteric ores. However, this method requires high-

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concentration alkalinity (usually greater than 50 %), high reaction temperature, and concentrated solutions [18–20]. Acid oxidation leaching technology is a comparatively simple method to leach vanadium and chromium from converter vanadium slag [21, 22]. This technology results in lower poisonous and harmful gas emissions from the leaching process, and its operation is relatively simple. However, the process was only used to leach vanadium directly from converter vanadium slag, whereas other states of vanadium were hardly leached using this method. In general, this process requires high concentrations of acid and corrosion-resistant equipment. Therefore, it is necessary to develop a clean and high recovery technology to extract vanadium and chromium from converter vanadium slag.

Previous studies showed that the easiest and most effective method to improve leaching efficiency was to add fortifier to the leaching process [2]. The additive components react with the slag in different ways to destroy the spinel phase structure. MnO_2 exhibits strong oxidation ability, such that low valence vanadium and chromium can combine with H^+ to damage the structure of the converter vanadium slag. This reduces the amount of acid required for the leaching process, and improves the leaching efficiency of vanadium and chromium. In addition, according to the standard electrode potential (298 K) table, in acidic solution, $\phi_{\text{MnO}_2/\text{Mn}^{2+}}^{\theta} = +1.23 \text{ V}$, $\phi_{\text{VO}_2^+/ \text{V}^{3+}}^{\theta} = +0.359 \text{ V}$, $\phi_{\text{VO}_2^+ / \text{V}^{3+}}^{\theta} = +0.680 \text{ V}$. This indicates that MnO_2 could oxidize low-valent vanadium to high-valent vanadium oxide [21]. At the same time, surfactants can change the surface tension of the leaching solution and wetting properties of the variable leaching system [23]. This represents a clean and efficient way to leach vanadium and chromium with MnO_2 and surfactants as enhancers of the leaching process.

The present work focused on strengthening the use of interface chemistry and controlling the chemical behavior of vanadium and chromium in aqueous solution. Through acid oxidation leaching with surfactants to extract vanadium and chromium from converter vanadium slag, the effects of sulfuric acid concentration, MnO_2 -to-slag mass ratio, liquid-to-solid ratio, leaching time, leaching

temperature, and sodium dodecyl sulfate (SDS)-to-slag mass ratio on the leaching efficiency of vanadium and chromium were studied.

2 Materials and methods

2.1 Materials

The converter vanadium slag used in this study was obtained from Panzhihua, Sichuan Province, China. The vanadium and chromium present in the slag were low valence states, such as FeCr_2O_4 and Fe_2VO_4 , as shown in Figure 1. Its chemical composition was analyzed by X-ray fluorescence, as shown in Table 1. X-ray diffractions (XRD) studies were performed using a Shimadzu XRD-6000 diffractometer with a step size of 0.05° and an angular range of 5° to 90° . Scanning electron microscopy (SEM) images were recorded on a Shimadzu SSX-550 instrument and the applied X-ray fluorescence was obtained on a Shimadzu XRF-1800 instrument. The ultraviolet-visible (UV-vis) spectroscopy data were tested with Beijing Purkinje General Instrument TU-1901.

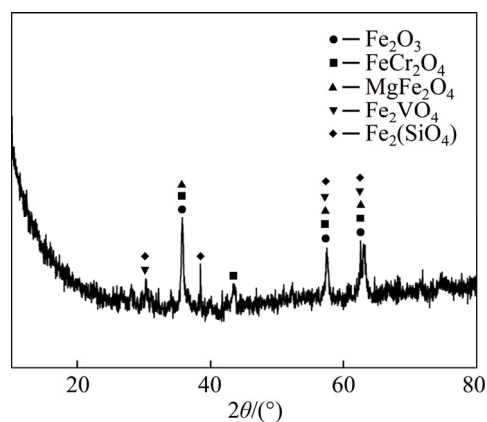


Figure 1 XRD pattern of converter vanadium slag

Table 1 Composition of converter vanadium slag (wt%)

V_2O_5	Cr_2O_3	Cr/V	TFe	FeO	CaO
9.7	10.2	1.28	19.2	24.7	2.04
MgO	SiO_2	Al_2O_3	MnO	TiO_2	
13.89	25.76	10.3	1.61	2.78	

All reagents used were of analytical grade, including sulfuric acid, MnO_2 and the surfactants used for leaching, as well as the ammonium ferrous sulfate, urea, potassium permanganate, sodium nitrite, and N-phenylanthranilic acid used for chemical analysis.

2.2 Methods

2.2.1 Leaching process

The leaching experiments were conducted in a 300 mL beaker that was heated in a water bath, with the temperature maintained within ± 0.1 °C. A predetermined amount of sulfuric acid and deionized water was added to the reactor to produce a homogeneous slurry under constant stirring (550 r/min to 560 r/min). The slurry was heated to a predetermined temperature and the converter vanadium slag and MnO_2 were subsequently added to the beaker.

After the scheduled leaching time, the filtrate was separated from the slag by vacuum filtration. Titration with ammonium ferrous sulfate was used to determine the concentration of vanadium and chromium in the filtrate. Then, the leaching efficiencies of vanadium and chromium were calculated using the following formulae, respectively:

$$\eta_V = \frac{C_V \times V}{m \times \omega_V} \quad (1)$$

$$\eta_{Cr} = \frac{C_{Cr} \times V}{m \times \omega_{Cr}} \quad (2)$$

where η_V is the leaching efficiency of vanadium; η_{Cr} is the leaching efficiency of chromium; V is the volume of the leaching liquid, L; C_V is the vanadium concentration in the leaching liquid, g/L; C_{Cr} is the chromium concentration in the leaching liquid, g/L; m is the mass of the converter vanadium slag, g; ω_V is the vanadium content in the converter vanadium slag; and ω_{Cr} is the chromium content in the converter vanadium slag.

The error analysis was based on the relative standard deviation (RSD) and the error sources included instrumental and human errors.

2.2.2 Surface tension measurements

A surface tension meter was used to determine the surface tension of the leaching filtrate, with an accuracy of 0.01 (N/m). During the process of measurement, the temperature was maintained at 25 °C. The measurement precision was improved by averaging the test values of repeated measurements.

3 Results and discussion

3.1 Effect of sulfuric acid concentration

To determine the effect of sulfuric acid

concentration on the leaching efficiency of vanadium and chromium, various concentrations of sulfuric acid were studied at 90 °C for 12 h with a liquid-to-solid ratio of 5:1 mL/g. The results are shown in Figure 2.

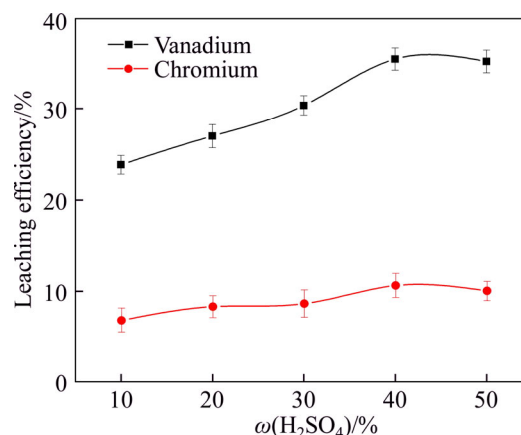


Figure 2 Effect of concentration of sulfuric acid on leaching efficiency of vanadium and chromium

Compared with other sulfuric acid concentrations, 40 wt% sulfuric acid was the most effective for leaching vanadium and chromium. At lower sulfuric acid concentrations, the rate at which protons (H^+) destroyed the spinel lattice was slow, and as the sulfuric acid concentration increased, the rate of reaction also increased as sufficient protons (H^+) were available to destroy the spinel lattice. However, the leaching efficiency of vanadium and chromium slightly reduced as the concentration of sulfuric acid increased from 40 wt% to 50 wt%. When the sulfuric acid concentration was higher than 40 wt%, impurities such as silicon and calcium sulfate covered the surface of the slag particles, reducing the rate of reaction [13]. Hence, based on the above results, 40 wt% sulfur acid was chosen as the optimal condition for further experiments.

3.2 Effect of mass ratio of MnO_2 -to-slag

To investigate its effect on vanadium and chromium leaching efficiency, the mass ratio of MnO_2 -to-slag was varied from 5.0 wt% to 15.0 wt% as the concentration of sulfuric acid was held constant at 40 wt%, and the leaching temperature and time were maintained at 90 °C and 12 h, respectively, with a liquid-to-solid ratio of 5:1 mL/g. The corresponding results are shown in Figure 3.

The leaching efficiencies of vanadium and chromium were 14.07% and 6.11% higher in the presence of MnO_2 , respectively, compared to the

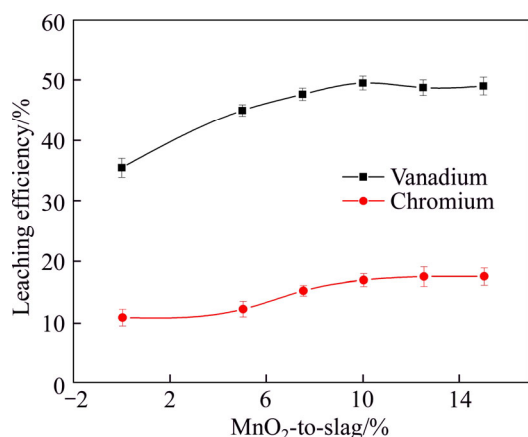


Figure 3 Effect of MnO₂-to-slag mass ratio on leaching efficiency of vanadium and chromium

control. During the leaching process, protons (H⁺) entered the slag particles and destroyed the silicate phase in the converter vanadium slag, exposing low-valent vanadium to the solution. In addition, when V(III), which possessed reducibility, came in contact with MnO₂, the process of oxidation to high-valent vanadium was enhanced, thereby increasing the speed at which low-valent vanadium was oxidized to high-valent vanadium. Thus, MnO₂ could combine with the protons (H⁺) to damage the slag structure and increase the leaching efficiency of vanadium and chromium [21].

When the mass ratio of MnO₂-to-slag was increased from 0 wt% to 15.0 wt%, the leaching efficiency of vanadium and chromium increased gradually. When the mass ratio of MnO₂-to-slag was higher than 10.0 wt%, the leaching efficiency of vanadium and chromium was largely unchanged and considering the cost and quantity of the filtered slag, the mass ratio of MnO₂-to-slag of 10.0 wt% was chosen as the optimum.

3.3 Effect of leaching time

To determine the effect of leaching time on the efficiencies of vanadium and chromium, different leaching times were studied. The concentration of sulfuric acid remained at 40 wt%, the MnO₂-to-slag mass ratio was 10.0 wt%, the liquid-to-solid ratio was 5:1 mL/g, and the leaching temperature was 90 °C.

The results shown in Figure 4 indicate that the leaching efficiencies of vanadium and chromium increased significantly when the leaching time was less than 12 h, and longer durations did not significantly improve the leaching efficiency. When

the leaching time was less than 12 h, increasing leaching time promoted contact between the slag and sulfuric acid solution. As the leaching time increased, silicon and calcium sulfate in the slag were leached, and these impurities covered the surface of slag particles and slowed the rate of subsequent reactions. Moreover, with increasing amounts of silicon and calcium sulfate, the viscosity of the pulp solution may increase and the mass transfer resistance at the acid-slag interface could decrease [13, 24]. Hence, the leaching time of 12 h was sufficient for leaching appreciable amounts of vanadium and chromium.

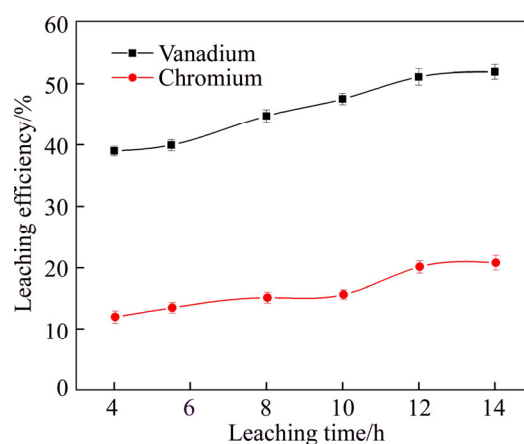


Figure 4 Effect of leaching time on leaching efficiency of vanadium and chromium

3.4 Effect of leaching temperature

To determine the effect of temperature on the leaching efficiencies of vanadium and chromium, the temperature was varied from 50 °C to 90 °C. The concentration of sulfuric acid remained constant at 40 wt%, the MnO₂-to-slag mass ratio was 10.0 wt%, liquid-to-solid ratio was 5:1 mL/g, and leaching time was 12 h.

Figure 5 shows that the leaching efficiency of vanadium and chromium increased gradually with increasing leaching temperature. That was likely because the increased temperature increased the number of activated molecules, accelerated molecular motion and collision between vanadium and chromium slag particles, and ensured full contact among the reactants. At the same time, the elevated temperature accelerated oxidation, and decreased the viscosity of the medium, increasing the diffusion rate and favoring increased reactions [5, 21]. Thus, the optimum temperature was determined to be 90 °C in this study, considering the costs and energy requirements [2, 24].

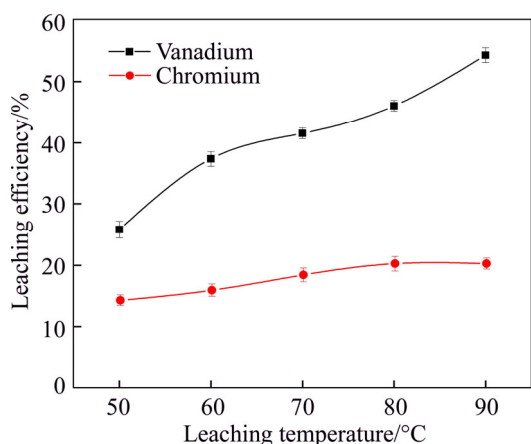


Figure 5 Effect of leaching temperature on leaching efficiency of vanadium and chromium

3.5 Effect of liquid-to-solid ratio

To determine the effect of the liquid-to-solid ratio on the leaching efficiencies of vanadium and chromium, the liquid-to-solid ratio was varied from 3:1 to 7:1 mL/g, with sulfuric acid concentration remaining constant at 40 wt% and the temperature at 90 °C. The mass ratio of MnO₂-to-slag was 10.0 wt%, and the leaching time was 12 h. The results are shown in Figure 6.

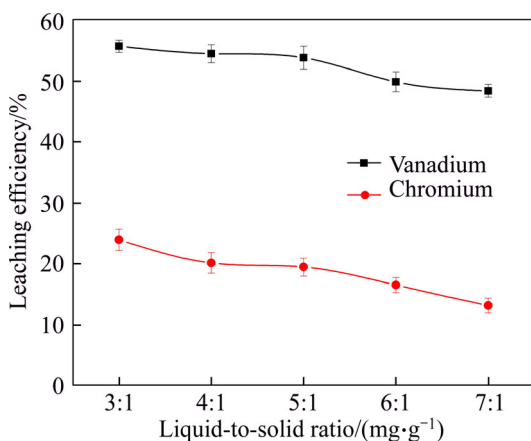


Figure 6 Effect of liquid-to-solid ratio on leaching efficiency of vanadium and chromium

From the above analysis, the effect of liquid-to-solid ratio on the leaching efficiencies of vanadium and chromium is clearly more significant than the other factors. Under the conditions of constant total leaching agent and increasing liquid-to-solid ratio, the volume of the liquid phase increased as the concentration of the leaching agent decreased. The chemical reaction equilibrium disfavored the leaching reaction, reducing the leaching efficiencies of vanadium and chromium

[11]. When the liquid-to-solid ratio was 3:1 mL/g, the concentration of leaching agent was too high, which increased the difficulty of separating the leachate from the slag.

Meanwhile, a high liquid-to-solid ratio produced a large amount of industrial waste water, which resulted in significant environmental pollution. Moreover, the liquid-to-solid ratio of 5:1 mL/g was easy to filter, and the leaching efficiencies of vanadium and chromium were relatively high at this ratio. Therefore, the optimum ratio was set as 5:1 mL/g in this work.

3.6 Effect of surfactant types

According to the nature of the polar groups, surface-active agents can be divided into anionic, cationic, zwitterionic, and non-ionic surfactants. According to its properties and results from previous studies [23, 25], the preliminary experiment investigated these four types of surfactants.

To determine the effect of surfactant type on the leaching efficiencies of vanadium and chromium, four types of surfactant were investigated. The sulfuric acid concentration, MnO₂-to-slag mass ratio, liquid-to-solid ratio, leaching temperature, and leaching time were held constant at 40 wt%, 10.0 wt%, 90 °C, 5:1 mL/g, and 12 h, respectively.

Figure 7 shows that different types of surfactants had different effects on the leaching process. SDS intensified the leaching efficiencies of vanadium and chromium while the others hindered the leaching process.

As shown in Table 2, the addition of different

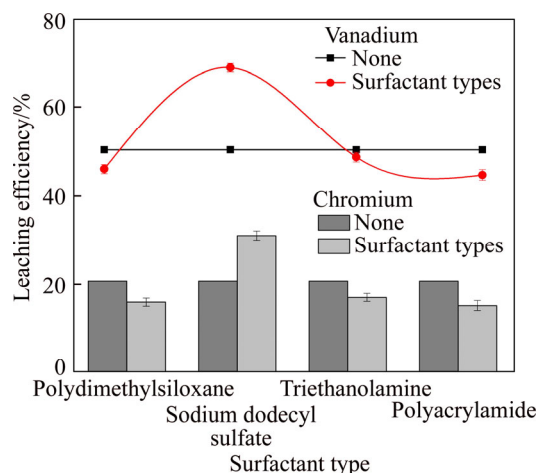


Figure 7 Effect of different surfactant types on leaching efficiencies of vanadium and chromium

Table 2 Lixivium of average surface tension by adding different surfactants ($T=25.0\text{ }^{\circ}\text{C}$)

Surfactant type	Average surface tension of leaching filtrate/($\text{N}\cdot\text{m}^{-1}$)
None	73.48
Polydimethylsiloxane (nonionic)	75.21
Sodium dodecyl sulfate (anionic)	59.65
Triethanolamine (cationic)	75.15
Polyacrylamide (zwitterion)	75.09

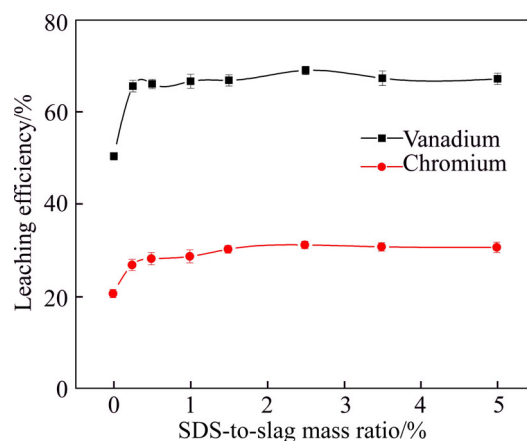
surfactant types resulted in different surface tensions. The addition of SDS reduced the average surface tension of the leachate, whereas the others increased the surface tension. The addition of SDS in the leaching process reduced the average surface tension of the leaching solution and decreased the adhesion strength between the liquid and solid phases. These factors likely reduced the moisture content of the slag to facilitate the filtration process. When the moisture content of the slag was reduced, the soluble vanadium present in the residual slag decreased. This phenomenon may increase the content of vanadium in the leaching solution and raise the leaching efficiency of vanadium [23]. Thus, SDS was selected for further study.

3.7 Effect of SDS-to-slag mass ratio

To determine its effect on the leaching efficiencies of vanadium and chromium, the mass ratio of SDS-to-slag was varied from 0 wt% to 5.0 wt% while the concentration of sulfuric acid remained at 40 wt% and the temperature at $90\text{ }^{\circ}\text{C}$. The leaching time was 12 h, the MnO_2 -to-slag ratio was 10.0 wt% and the liquid-to-solid ratio was 5:1 mL/g.

Figure 8 shows that increased SDS-to-slag mass ratio did not significantly change the leaching efficiency of vanadium and chromium. Table 3 shows that the average surface tension of the leaching filtrate was reduced by the addition of SDS.

During the leaching process of vanadium and chromium from the converter vanadium slag, the addition of SDS changed the properties of surface wettability to intensify leaching. SDS considerably reduced the surface tension of the leaching solution. This uniformly distributed the slag in the leaching solution, helping to maintain optimal contact with the leaching agent. This effect can be attributed to the surfactant, which can adsorb on the surface of

**Figure 8** Effect of SDS-to-slag mass ratio on leaching efficiencies of vanadium and chromium**Table 3** Effect of SDS-to-slag mass ratio on surface tension ($T=25.0\text{ }^{\circ}\text{C}$)

SDS dosage/%	Average surface tension of leaching filtrate/($\text{N}\cdot\text{m}^{-1}$)
0	74.48
0.25	62.84
0.5	62.64
1.0	62.35
1.5	60.66
2.5	59.65
3.5	59.37
5.0	59.17

slag grain, changing its wettability, and increasing the contact between the slag and leaching agent. Meanwhile, the surfactant exhibited the characteristics of parent molecular structure, which produced oriented adsorption at the interface of the solution, reducing the surface tension of the leaching solution, and enhancing the wettability and permeability of the solution. Thus, the leaching efficiency of vanadium was effectively improved. At the same time, the addition of the surfactant reduced the moisture content of the slag which aided filtration [23, 25].

4 Reaction mechanism

Based on the acid oxidation reaction that occurred, the mechanism of acid oxidation leaching is depicted in Figure 9.

UV-vis spectroscopy is a powerful tool that can be used to elucidate the ion valence state of vanadium and chromium. According to previous

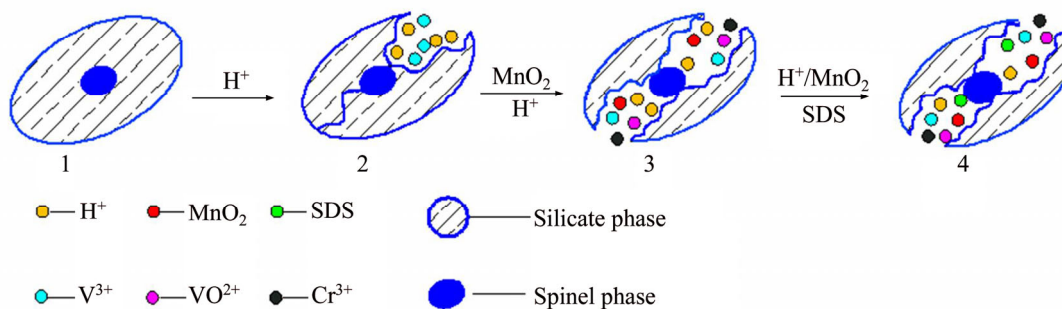


Figure 9 Mechanism of vanadium leaching

studies, V(IV) and Cr(VI) absorb at about 760 nm and 373 nm, respectively [2], as shown in Figure 10. Vanadium and chromium were present in the forms of V(IV) and Cr(VI), and through the sulfuric acid leaching process, the pH of the leaching solution was less than 2. Vanadium was present in the leaching solution in the VO²⁺ ion.

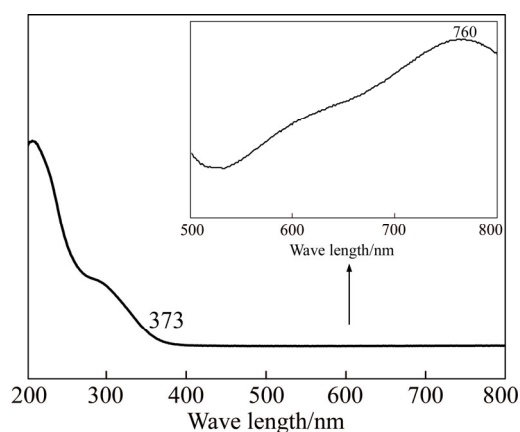
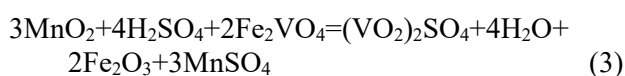


Figure 10 UV-vis spectra of leaching solution

During the leaching process, protons (H⁺) damaged the silicate phase, exposing the spinel phase wrapped in the silicate phase in the process. Then, MnO₂ oxidized the low-valent vanadium and chromium to improve the respective leaching efficiencies of vanadium and chromium. The main reaction occurring in the leaching process is as follows:



The addition of the surfactant SDS changed the chemical behavior of vanadium and chromium in the aqueous solution, which enhanced the efficiency of MnO₂ oxidation and subsequent leaching [21, 26].

Surfactants reduced the surface tension of the leaching solution and changed the wetting properties in the variable leaching system. Surfactants had high surface activity and good diffusion and permeability, allowing them to quickly penetrate into the inner surface of the slag particle pores where they were adsorbed.

Adsorption of SDS on the surface of the slag in the leaching solution had a significant impact on the wetting effect. The surface of the converter vanadium slag generally exhibits significant electronegativity [23, 27]. For anionic surfactants, the nonpolar group was adsorbed on the surface of the converter vanadium slag. The polar group was oriented toward the leaching solution, which allowed the protons (H⁺) to enter the inner areas of the converter vanadium slag and promoted the leaching process. This mechanism is shown in Figure 11.

The results shown in Figure 12(a) indicate that the original converter vanadium slag particles were irregular in shape and unevenly distributed. From Figure 7(b), the irregular shapes were broken into molten particles, the sheet-like structures were broken into small irregular pieces, and the surface became rough after leaching. The silicate and spinel phases were partially broken after acid oxidation leaching.

5 Conclusions

1) Within the scope of the critical micelle concentration, the addition of SDS considerably affected the chemical properties of the pulp surface, increasing the hydrophilicity, and decreasing the surface tension of the leaching solution and moisture content of the slag. Hence, the addition of

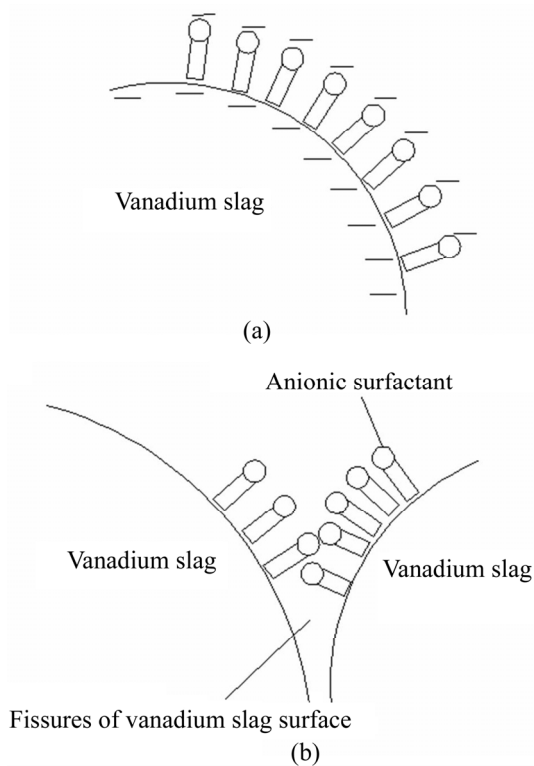


Figure 11 Adsorption of anionic surfactant on slag surface: (a) On slag surface; (b) In slag fissures

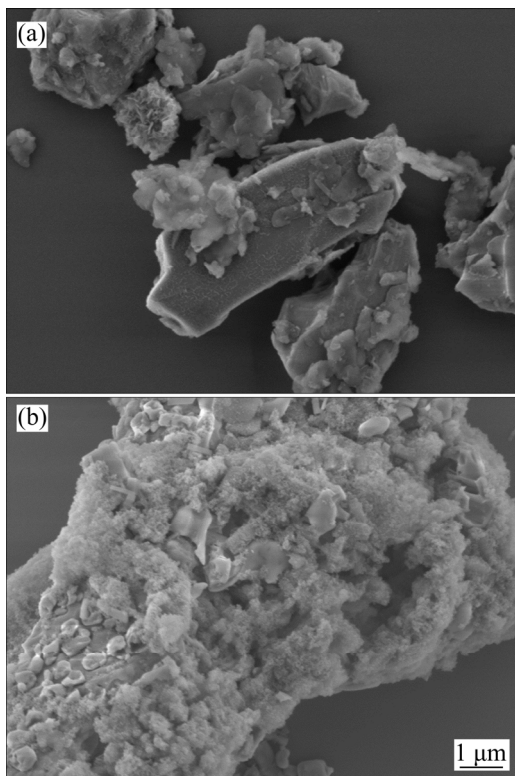


Figure 12 SEM images of converter vanadium slag before and after leaching: (a) Converter vanadium slag; (b) Converter vanadium slag after acid leaching with MnO_2 and SDS

SDS could improve the leaching efficiencies of vanadium and chromium.

2) Vanadium and chromium existed in low valence in the converter vanadium slag and were not directly leached to a great extent. The addition of MnO_2 combined with protons (H^+) damaged the slag structure and greatly improved the leaching efficiency of vanadium from the converter vanadium slag.

3) The leaching efficiencies of vanadium and chromium from the converter vanadium slag were 68.93 % and 30.74 %, respectively, under the optimal conditions: sulfuric acid concentration of 40 wt%; MnO_2 -to-slag mass ratio of 10.0 wt%; liquid-to-solid ratio of 5:1 mL/g; time of 12 h; temperature of 90 °C; and SDS-to-slag mass ratio of 0.25 wt%.

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中文导读

表面浸润强化转炉钒渣中钒和铬的浸出

摘要: 介绍一种从转炉钒渣中提取钒和铬的表面浸润强化浸出技术, 考察硫酸浓度、二氧化锰 (MnO₂) 的添加量、液固比、浸出时间、浸出温度以及十二烷基硫酸钠 (SDS) 的添加量对钒和铬浸出率的影响。结果表明: 在浸出过程中添加 MnO₂ 和 SDS, 钒和铬的浸出率比未添加时分别高 33.46% 和 20.02%, 在硫酸浓度 40 wt%、反应时间 2.0 h、液固比 5:1 mL/g、反应温度 90 °C、MnO₂ 添加量为 10.0 wt% 以及 SDS 的添加量 0.25 wt% 时, 钒和铬的浸出率分别为 68.93% 和 30.74%。浸出过程反应机理表明, MnO₂ 协同 H⁺ 离子可以氧化低价钒和铬, SDS 能改变溶液表面化学行为, 降低溶液表面张力, 有利于 MnO₂ 的氧化。

关键词: 钒; 铬; 浸出; 十二烷基硫酸钠; 二氧化锰