



Separation and recovery of vanadium from a sulfuric-acid leaching solution of stone coal by solvent extraction using trialkylamine



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ABSTRACT

Trialkylamine-N235, diluted with sulfonated kerosene containing tri-butyl phosphate, was used to separate vanadium and impurities as Fe, Al, Mg, K, Ca, P, and Si from a leaching solution generated by the sulfuric-acid leaching of vanadium-containing stone coal. Variations in aqueous pH exhibited good extraction of vanadium at an equilibrium pH of 1.6. Vanadium was successfully extracted by a three-stage countercurrent process using 20% N235 and 5% tri-butyl phosphate at an organic-to-aqueous (A/O) phase ratio of 2:1. The loaded organic was then scrubbed with 0.5 mol/L H₂SO₄, and most impurities were scrubbed by three-stage scrubbing. 1.0 mol/L NaOH was used to strip of vanadium from the scrubbed organic solution at an O/A phase ratio of 2:1, where >99% of vanadium was stripped in three stages. After regeneration by 1.0 mol/L H₂SO₄, the regenerated organic phase showed a good performance on vanadium extraction even after 5 cycles. Finally, a process flow sheet was established to recover of vanadium from a sulfuric-acid leaching solution of stone coal.

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1. Introduction

Vanadium is a significant strategic resource that is widely used in numerous fields because of its unique properties, such as hardness, fatigue resistance, tensile strength, specific physiological functions, good corrosion resistance at low temperature, and high melting point [1]. As vanadium applications continue to expand, high-purity vanadium products have become increasingly on demand, and studies on high-quality vanadium pentoxide preparation techniques are necessary. Vanadium-bearing stone coal is an important vanadium resource in China; therefore, vanadium extraction from stone coal is given considerably more attention in this country [2]. At present, acid leaching with H₂SO₄ has been widely adopted for vanadium leaching from stone coal because of the advantages of high vanadium recovery and low pollution [3,4]. So as to improve vanadium leaching rate, high H₂SO₄ concentration is commonly used in the acid leaching process. Therefore, numerous impurities, such as Fe, Al, and Mg ions, are also leached along with vanadium from stone coal, resulting in a complex vanadium-bearing leaching solution with high concentrations of elemental impurities and low pH value. The recovery and separation of vanadium from leaching solutions that contain massive impurities are difficult. Precipitation of the leaching solution at

pH of 4–5 to remove Al and Fe [5] would result in a substantial loss of vanadium because of vanadium hydrolysis precipitation within this pH range. Therefore, a solvent extraction system that can extract and separate vanadium from impurities should be developed. Solvent extraction presents advantages of being a well-established process, sturdiness, and favorable economics [6].

Numerous researchers have studied and developed solvent extraction for the separation of vanadium and other ions from acid leaching solutions [7–10] and alkaline solutions [11]. Selective extraction of vanadium using different extractants, such as D2EHPA [8,9], LIX 63 [7], PC88A [12], amines (N1923, Alamine 308, and Alamine 336) [5,13–16], Cyanex 923, and Cyanex 272 [17], from the solution of a multi-element system has been found in the literature. In the above-mentioned extraction approach, phosphate-based extraction agents exhibit the advantages of acidic system adaptability and ease of stripping. However, these extractants present the disadvantage of poor selectivity [18], as these extractants can co-extract other metals, such as Fe and Al, even at high acidity. Furthermore, due to the weak efficiency of organophosphorus extractants for the V(V) extraction, V(V) in the acid leaching solution of roasted stone coal should be reduced to V(IV) prior to extraction and then re-oxidized to V(V) prior to vanadium precipitation. These steps make the purification procedure more complicated and expensive [19]. Oxime base extractants (LIX) were found to separate vanadium in the more acidic range (pH < 1) and provide good vanadium separation. However, this

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extraction agent is expensive, extracting vanadium from a low-grade ore, such as stone coal, is not economical. Amine extractants are potential extractants that are able to extract vanadium from acidic, neutral, and alkaline solutions, as well as separate vanadium and Mo, Co, and Ni ions thoroughly from spent catalyst leaching solution. Kumar et al. [20] found that amine-based extractants exhibit better separation efficiency of vanadium and other metals, such as Ca and Fe, in comparison with phosphorus-based extractants. However, the literature focused on the extraction behaviors of vanadium and common impurities in acid leaching solution of stone coal using amine extractants is limited. Acidic solutions from stone coal contain more Al, Fe, P, and Si, and less Mo, Co, and Ni, which are extremely important for the practical application of amine extractants.

Therefore, in the current investigation, a separation scheme for vanadium and common ions was developed by solvent extraction using trialkylamine for the recovery of vanadium from stone coal acid leaching solution. The present study mainly aims to obtain better extraction efficiency of vanadium and separation possibilities from other associated elements, such as Fe, Al, Mg, K, Ca, Si, and P.

2. Experimental

2.1. Materials and apparatus

The trialkylamine N235 (R_3N , $R = C_8-C_{10}$, tertiary amine, commercialized in China) extractant was supplied by Luoyang Aoda Chemical Co., Ltd. and acidified by 1.0 mol/L H_2SO_4 (volume ratio = 1:1) prior to use. Tri-butyl phosphate (TBP) used as a phase modifier was procured from Xilong Chemical Co., Ltd. Sulfonated kerosene purchased from Shanghai Rare-Earth Chemical Co., Ltd. was used as diluent. H_2SO_4 , CaO, NaOH were of analytical grade. Acidic vanadium leaching solution was prepared by roasting stone coal at 850 °C for 1 h, followed by leaching with 15% (v/v) H_2SO_4 and 0.8% (wt) $NaClO_3$ (used as oxidant) at 95 °C and 6 h. The composition of this solution is listed in Table 1, and the pH value of the solution was around 0.4. Results of vanadium valence determination using the potentiometric titration method [21] shows that 99.2% of the vanadium in the leaching solution was V(V).

The concentrations of ions in the solution were analyzed by inductively coupled plasma-optical emission spectroscopy (ICP-OES, Optima 4300DV, Perkin-Elmer, USA). The pH of the solution was measured using a pH meter (Mettler Toledo S220, Switzerland).

2.2. Procedure of solvent extraction and stripping

The solution was adjusted to desired pH values by using 2 mol/L H_2SO_4 and 300 g/L CaO prior to solvent extraction. Extractants were composed of 20% N235 (v/v), 5% TBP (v/v), and 75% (v/v) sulfonated kerosene (except in the N235 concentration experiments). Solvent extraction and stripping experiments were performed by magnetically stirring (with stirring frequency of 600 r/min) the organic and aqueous phases (40 mL aqueous and 20 mL organic, except for A/O ratio experiments) in 250 mL conical flasks immersed in a temperature-controlled water bath at 25 °C for 10 min (except for extraction time experiments). After phase disengagement within 5 min, the organic and aqueous phases were

separated by using separatory funnels. Aqueous solutions (raffinate) included diluted rational multiples to analyze the concentrations of metals with ICP-OES, and the concentrations of ions in the organic phase were calculated by mass balance.

The loaded organic phase was prepared by the extraction of feed solutions. In stripping experiments, loaded organic phase and stripping reagents were mixed in conical flasks by magnetic stirring at a stirring frequency of 600 r/min and separated in separating funnels (within 10 min). After phase disengagement, the concentrations of metals in the aqueous phase were analyzed by ICP-OES.

2.3. Data treatment

Distribution ratios (D), separation coefficient (β), extraction (E), and stripping (S), were calculated by Eqs. (1)–(4), respectively

$$D = \frac{C_{org}}{C_{aq}} \quad (1)$$

$$E = \frac{D}{D + (V_{aq}/V_{org})} \times 100\% \quad (2)$$

$$\beta_{a,b} = \frac{D_a}{D_b} \quad (3)$$

$$S = \frac{C'_{aq} V'_{aq}}{C'_{org} V'_{org}} \times 100\% \quad (4)$$

where C_{org} is the concentration of vanadium or other ions present in the organic phase, C_{aq} is the concentration of vanadium or other ions in the raffinate, V_{aq} and V_{org} are the volumes of aqueous and organic phases used in the extraction, respectively, $\beta_{a,b}$ is the separation coefficient of ion a and ion b, C'_{org} is the vanadium or other ions content in the loaded organic phase, V'_{org} is the volume of the loaded organic phase, C'_{aq} is the concentration of vanadium or other ions content in stripping solution, and V'_{aq} is the volume of stripping solution.

3. Results and discussion

3.1. Solvent extraction

3.1.1. Effect of initial pH on the vanadium extraction

The effect of initial solution pH on vanadium extraction under the conditions of organic solution consist of 20% (v/v) N235 and

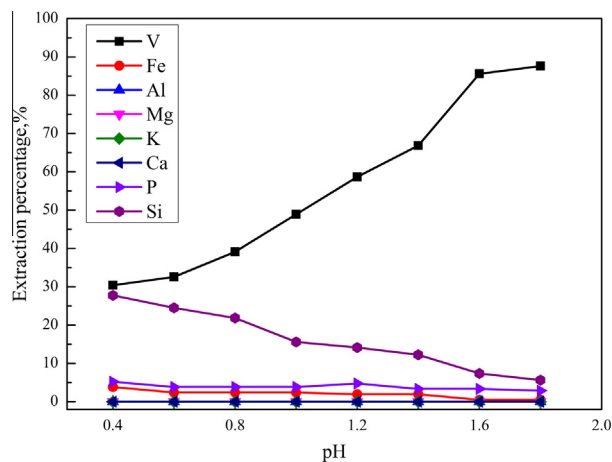


Fig. 1. Effect of pH on vanadium and other ions extraction.

Table 1
Composition of acid leaching solution.

Items	V	Fe	Al	Mg	K	Ca	P	Si
Concentration (g/L)	1.47	3.09	13.88	3.51	5.08	0.65	0.57	0.21

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