



# High-efficient extraction of vanadium and its application in the utilization of the chromium-bearing vanadium slag



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## HIGHLIGHTS

- Efficient extraction of vanadium using primary amine with hydrogen bond association mechanism.
- The impurity ions containing in the leaching solution was also considered.
- The extractant natures including viscosity and the dissolution loss were researched.
- The industrial experiments were performed based on the lab and pilot work.
- The vanadium was recycled as a high value product to be 99.5 wt.% vanadium pentoxide.

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## ABSTRACT

The high-efficient extraction of vanadium using primary amine and its application in the utilization of the chromium-bearing vanadium slag (V–Cr slag) were studied in this work. The extraction mechanism and the possible difficulties in the application were both considered. From the results, the extraction percentage of vanadium using free primary amine with the hydrogen bond association mechanism is higher than that in the acidic environment by primary amine salts. The inorganic ions and other impurities existing in the system were all considered to research for the continuous operation of the extraction. Furthermore, the viscosities of the fresh organic phase and the recycled organic phase were determined using the extractant not only N1923 but also LK-N21, where the viscosity of the latter system increased slightly during the recycled process of the extractant. Based on the above results, the pilot and the commercial experiment were carried out. In the large-scale experiment, the vanadium was extracted and the product of 99.5 wt.% vanadium pentoxide was obtained, and the mass loss of the extractant was only 0.1%.

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## 1. Introduction

Vanadium exists naturally in about 65 different minerals and in fossil fuel deposits, and was used widely in many industries [1,2] because of its good performance. It is mainly used to produce specialty steel alloys such as high-speed tool steels [3]. Its oxides, such as vanadium pentoxide, had an important industrial application as a catalyst for the production of sulfuric acid and selective catalytic reaction in the past decades [1]. Nowadays, the development and application of vanadium redox flow battery [4,5] is becoming a hot topic in hydrometallurgy, which expands the application scope of vanadium. Therefore, the hydrometallurgy of vanadium, especially the production of the high purity vanadium,

is still believed to have a good future [6] although the price of common vanadium is lower now.

The recovery of vanadium from the leaching solution of ore, fly ash or waste slag has been widely studied [7,8]. The extraction [9], precipitation [10], ion exchange [11] and membrane [12] methods were studied by many researchers. However, for the recovery of the high concentration of vanadium in leaching solution, ion exchange and membrane methods were not suitable due to their small capacities [13,14]. The vanadium is usually precipitated as the ammonium polyvanadate under high acidic concentration at 90 °C [15]. Vanadium was recovered using this method in most Chinese plants. However, the purity of vanadium oxide obtained by this ammonium polyvanadate is almost no more than 98.5% resulting in the lower value and the lower price of the product. It must be to find an effective way to recycle vanadium with higher purity, especially from the co-existing system of vanadium and chromium. Solvent extraction has been reported to separate

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vanadium from the solution containing vanadium and chromium [16]. Yu [17] put forward primary amine to extract vanadium selectively from chromium in the 80s of the last century. She used primary amine N1923 extracting vanadium in nearly neutral condition with equilibrium pH value of 7.0–8.0, and presumed the reaction mechanism to be solvation. However, this technology was not to industrialize because the viscosity of the extractant N1923 was too big to cycle continuously in the pilot experiment. Due to this difficulty, we [18] studied this system carefully and then found that the extracted complex increased the viscosity of the organic phase and the organic phase was partly oxidized by the hexavalent chromium. These results assisted that the impurity ions bearing in the leaching solution deteriorated the extraction cycle. Then, we [18] designed one of the primary amine extractants named LK-N21 to make the quantitative and thermodynamic calculation combined with our experiment. Here, the extractant LK-N21 is the mixture of some primary amines with 16–22 C atoms, and its boiling point is in the range from 5 to 325 °C. We also researched the separation performance of this extractant. Primary amine LK-N21 can extract more than 90% of V(V) which resulted in over 90% of Cr(VI) left in the leaching solution of the V–Cr slag under the optimal conditions. The interfacial crud was analyzed in detail and the prevention method [19] was also put forward [20,21]. Based on the above results, the technology route was drawn in Fig. 1.

In this paper, the work about the key technology of the utilization of the V–Cr waste slag was performed. The extraction mechanism of the reaction was studied comprehensively from various areas. In particular, the effects of the impurities on the extraction and the interface were carried out. Then the pilot experiment and the industrial experiment were performed in turn. The vanadium pentoxide of 99.5 wt.% was produced in the industrial-scale demonstration.

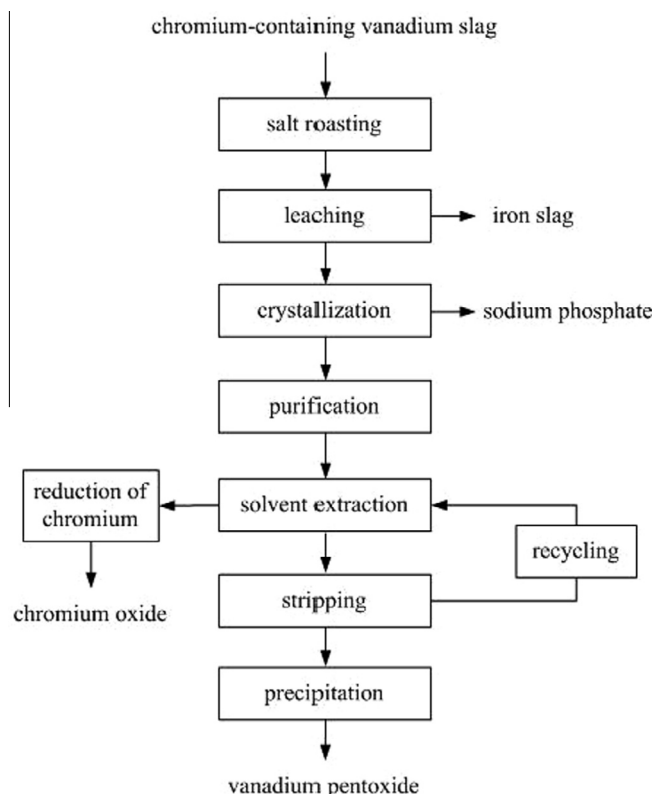


Fig. 1. Flow chart for the resourceful utilization of the chromium-bearing vanadium slag.

## 2. Experimental

### 2.1. Materials

In this work, the V–Cr waste slag was provided by the Huihong Nonferrous Metal Co., Ltd. (Liaoning Province, China), containing high levels of Na, V, Cr, a small amount of SiO<sub>2</sub> and low levels of inorganic salts of Fe and Al. Detailed compositions are listed in Table 1. The slag was roasted in the muffle furnace with sodium salts and dissolved in the water. The leaching solution was obtained after the solids in the system were filtered, with the components shown in Table 2. The extraction system is composed of primary amine LK-N21 modified by phase modifier LK-N21X diluted in kerosene. The chemical reagents used in this paper are listed in Table 3 without further purification. LK-N21 is the mixture of the primary amine with C<sub>16</sub>–C<sub>20</sub> with the molar concentration of –NH<sub>2</sub> being 3.15 mol/L. The modifier LK-N21X is a mixture of aliphatic alcohol and ester (analytical grade). The average density of LK-N21X is 0.831 g/cm<sup>3</sup> at 25 °C. The primary amine salts was obtained by pre-equilibrium with 10 vol.% H<sub>2</sub>SO<sub>4</sub> solution, and then scrubbed by a sodium sulfate solution (10 wt.%) until the aqueous phase was nearly neutral.

### 2.2. Experimental methods

The experimental methods were basically the same with the literature reported by us before [20]. The solvent extraction was performed by putting equal volume of the aqueous and organic phase into a separating funnel. The funnel was then fixed in an oscillator and shaken mechanically for 15 min at 20 °C. After that the mixture was allowed to stratify for 10 min. Then the organic phase was for recycling if needed. Recycling one time means the organic phase is stripped then contacted with the fresh leaching solution a second time.

### 2.3. Instrumental analysis

The metal ions in the aqueous phase were analyzed using an OPTIMA 6300DV (Perkin-Elmer, USA) ICP-OES. The aqueous solution sample was diluted with 1 vol.% hydrochloric acid solution, and then analyzed using the most sensitive wavelength corresponding to each element investigated. The anion concentration in the aqueous solution was determined by DX-500 ion chromatography (DIONEX, USA). Before analysis, the aqueous solution was diluted to the required volume by ultrapure water, and filtered by 0.22 μm cellulose membrane.

Table 1  
Weight percentages of major elements in V–Cr slag.

Element	Cr	V	P	Fe
Weight percentage, wt.%	3.4–5.7	3.26–5.2	24.7–26.6	59.9–68.5

Table 2  
Chemical components of the real feed solution originated from the V–Cr slag.

Element	Na	Al	SiO <sub>2</sub>	SO <sub>4</sub> <sup>2–</sup>	Cl <sup>–</sup>	Ca
Concentration, g/L	35.4	0.04	0.05	27.4	1.71	0.01
Element	Ti	V	Cr	Fe	Mo	Hf
Concentration, g/L	0.02	12.0	6.48	0.01	0.05	0.03

\* The anions were determined by ion chromatography, the others were measured by ICP-OES.

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