



# Enhanced leaching of vanadium slag in acidic solution by electro-oxidation



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## ABSTRACT

An improved process for the leaching of vanadium from vanadium slag assisted by electro-oxidation was developed to increase the leaching ratio of vanadium. Factors associated with the leaching process were investigated, and a high vanadium leaching ratio (>70%) was achieved under the optimal leaching condition of 40 wt.% H<sub>2</sub>SO<sub>4</sub>, 250 g/L solid concentration, anode current of 0.4 A, operating potential of 2.8–3.0 V, and electrode spacing of 20 mm at 75 °C for 4 h. X-ray diffraction patterns showed that the spinels and silicate were the major phases in the vanadium slag, and vanadium was in the form of FeV<sub>2</sub>O<sub>4</sub> (namely of FeO·V<sub>2</sub>O<sub>3</sub>). Scanning electron microscopy studies indicated that the spinels were broken into small pieces and vanadium was exposed on the surface, leading to facile oxidation and high vanadium leaching ratio, and demonstrated that electro-oxidation could significantly enhance the leaching process.

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## 1. Introduction

Vanadium is conventionally regenerated from recovered materials, such as vanadium titanium magnetite, spent catalyst, fly ashes from oil industry and vanadium slag (Moskalyk and Alfantazi, 2003). The roast-leaching process is the most applied recovery process (Holloway and Etsell, 2006, Holloway and Etsell, 2004; Shelwit and Alibrahim, 2006), which includes chloridizing roasting, water leaching, deposition, alkali melting, and thermal decomposition (Qi, 1999). Pelletization roasting–alkaline leaching–ion exchange–precipitation–calcining technology results indicated that the vanadium conversion rate achieved above 75% (Tan, 2008). The technique of extracting vanadium from stone coal is pelletization–oxidation roasting–alkali leaching with the roasting temperature 850 °C in a roasting time of 3 h. Its leaching temperature is 90 °C with a leaching agent concentration 2 mol/L and leaching L/S 3 at a leaching time of 2 h (He and Feng, 2007). But it has the disadvantages of more material and high energy consumption. Moreover, the process produces poisonous gases like HCl or Cl<sub>2</sub>, which can cause serious environmental pollution (Cai and Ling, 1993). The recently developed direct acid leaching technique (Bin, 2006; He et al., 2007; Mishra et al., 2007) has better recovery efficiency when assisted by ultrasound and additives (Amer, 1994; Shi et al., 2007; Yan, 1992), but would also generate much waste acid (Du et al., 2008; Ozawa et al., 2004; Qi, 1999; Zeng et al., 2002). In the direct acid leaching process (Fig. 1a), the generated protons (H<sup>+</sup>) react mostly with high valent

vanadium (VO<sub>2</sub>) by air oxidation, while most low valent vanadiums (V<sub>2</sub>O<sub>3</sub>) are not involved in the reaction. Hence the vanadium leaching ratio is usually low. Therefore, it is desirable to achieve high vanadium leaching ratio.

Enhancing the leaching process by employing electro-oxidation is an excellent alternative method because higher valent vanadium (VO<sub>3</sub>) can be generated from low valent vanadium (V<sub>2</sub>O<sub>3</sub>) by electro-oxidation (Fig. 1b) (Liu et al., 2014, 2015). Moreover, applied electricity is considered a clean energy source and the electro-oxidation process is an ideal hydrometallurgical process for leaching vanadium from vanadium slag without producing HCl and Cl<sub>2</sub> (Liu, 2011a, 2011b; Liu et al., 2010). The electro-oxidation process is widely used to treat copper sulfide, lead and zinc sulfide ore, bismuthinite, vulcanization silver, and pyrite (Antonijevic and Pacovic, 1992; Cao et al., 2009; Cruz et al., 2005; Souza et al., 2007). Based on previous studies on the oxidization of sodium hypochlorite and the electro-oxidation of molybdenite (Fu et al., 2005), suitable oxidization conditions were NaCl concentration of 4.0 mol/L, mass ratio of 20 (ore slurry liquid to solid), electric charge of 0.522 °C/g of Mo, original slurry at pH 8, anodic current density of 700 A m<sup>-2</sup> (cell potential 2.7–2.9 V) and temperature at around 25 °C. This yielded a Mo leaching ratio and current efficiency of 98% and 36%, respectively. The study also showed that a Mo leaching ratio of 88.5% can be achieved at 55 °C with 7 mol/L of H<sub>2</sub>SO<sub>4</sub>, 0.5 mol/L of MnSO<sub>4</sub>, 20 A h g<sup>-1</sup> of electricity, 15 mm electrode spacing, 800 A/m<sup>2</sup> anode current density, and rate of liquid to solid 40.

The present work focuses on the electro-oxidation hydrometallurgical leaching process with H<sub>2</sub>SO<sub>4</sub> for converter vanadium slag. The mechanism of the electro-oxidation hydrometallurgical process is discussed along with the factors associated with vanadium extraction.

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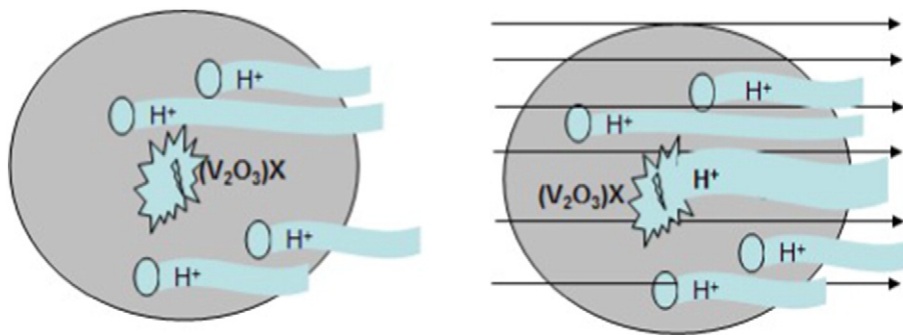


Fig. 1. Two vanadium slag phase destruction processes. a) Direct acid leaching; b) Electro-oxidation acid leaching

**Table 1**  
Main composition of vanadium slag (wt.%).

Element	Fe	Si	V	O	Ti	Mn
wt.%	27.39	9.95	8.57	27.57	8.08	7.05
Element	Mg	Ca	Re	Ga	Nb	
wt.%	4.06	1.93	0.04	0.01	0.03	

## 2. Experimental

### 2.1. Materials

The vanadium slag was obtained from Pan Gang Group Company, Sichuan Province, China. The main chemical compositions were determined by X-Ray Fluorescence and listed in Table 1. The mineralogical composition and the surface morphology of the samples were identified by X-ray diffraction (XRD) and scanning electron microscopy (SEM), as depicted in Figs. 2 and 9(A). The X-ray diffractions (XRD) studies were performed using a Shimadzu XRD-6000 Diffractometer with a step size of  $0.05^\circ$  and angular range of  $5\text{--}80^\circ$ . Scanning electron microscopy (SEM) images were recorded on a Shimadzu SSX-550. The applied X-ray fluorescence was obtained on a Shimadzu XRF-1800. As indicated in Fig. 2, vanadium slag contains much of spinels ( $\text{MnV}_2\text{O}_4$ ,  $\text{FeV}_2\text{O}_4$ ,  $\text{MnFe}_2\text{O}_4$ ) and minor amounts of free oxide ( $\text{VO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{SiO}_2$ ).

All aqueous solutions were prepared with distilled water. Other chemicals including  $\text{H}_2\text{SO}_4$ ,  $\text{MnSO}_4$ , urea, Tantalum,  $\text{KMnO}_4$ ,  $\text{NaNO}_2$  and  $\text{H}_3\text{PO}_4$  were of analytical grade and purchased from domestic vendors.

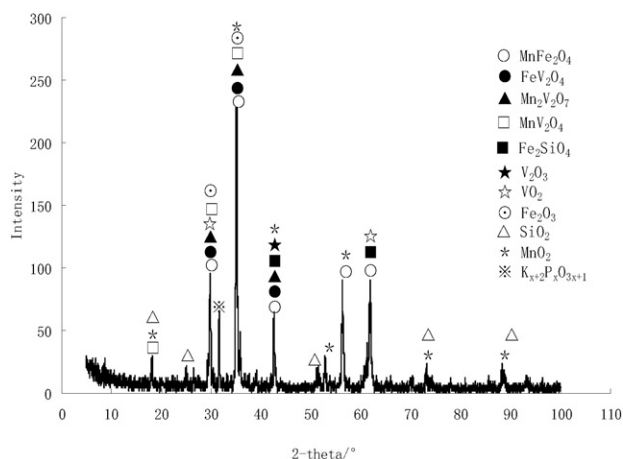


Fig. 2. XRD pattern of vanadium slag.

### 2.2. Methods

The leaching experiments were performed in a 500 mL beaker heated in a water bath. The bath was equipped with a plate-like dimensionally-stable-anode (DSA,  $10\text{ cm} \times 10\text{ cm}$ ) and a plate-like pure titanium cathode ( $10\text{ cm} \times 10\text{ cm}$ ), and the electrode spacing was 20 mm. The solution was stirred by a mechanical agitator. When the leaching agent sulfuric acid solution reached the desired temperature, vanadium slag ( $\sim 200$  meshes) and  $\text{MnSO}_4$  were added to the solution. After a scheduled leaching time, the leachate was separated from the residue by vacuum filtration. To systematically investigate the effects of various experimental conditions on the leaching processes, the experiments were conducted under different sulfuric acid concentration, leaching temperature, leaching time and electrical current. The scheme for the experimental setup is shown in Fig. 3.

The calculations for the leaching ratio were based on the following equation:

$$\eta_V = \frac{V \times C_V}{M_V} \times 100\%$$

Where  $\eta_V$  is the leaching ratio of vanadium,  $V$  is the filtrate volume,  $C_V$  is the concentrations of vanadium in the filtrate and  $M_V$  is the quantity of vanadium in the vanadium slag.

## 3. Results and discussion

### 3.1. Effect of temperature on leaching efficiency

The effects of temperature on the vanadium leaching ratio of different leaching ways were examined at  $55\text{--}95^\circ\text{C}$ , and the results (Fig. 4)

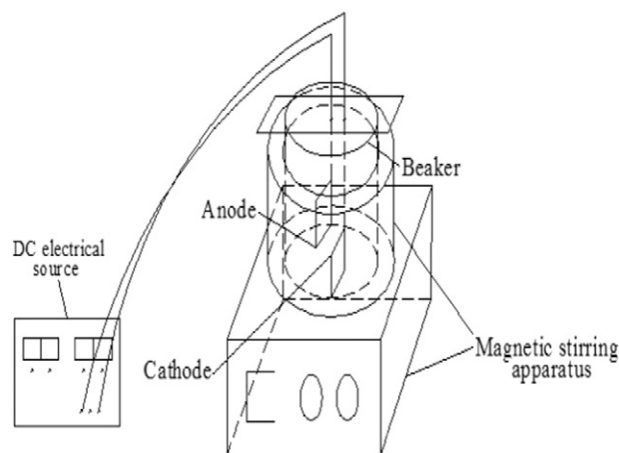


Fig. 3. Schematic diagram of experimental apparatus.

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