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Hydrometallurgical leaching process intensified by an electric field for converter vanadium slag

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article info abstract

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Low-valent vanadium in the insufficient oxidation roasting process of sodium salt reduces vanadium leaching yield. Therefore, the electro-oxidation leaching method was investigated to strengthen the oxidation process of low-valent vanadium. The mechanism of the electro-oxidation hydrometallurgical leaching process was examined along with the electrochemical performance of the reaction system through X-ray diffraction, scanning electron microscopy, ultraviolet, and ultraviolet–visible diffuse reflectance spectroscopy. Results showed that low-valent vanadium can be oxidized effectively under an electric field in the leaching process. The leaching yield of vanadium reached 93.67% under the optimum conditions, including a current density of 1000 A/m², a liquid-to-solid ratio of 4:1 ml/g, a temperature of 80 °C, and a leaching time of 40 min. This percentage is 3.73% higher than that attained under the same conditions without an electric field. Meanwhile, the vanadium content in slag decreased from 7.15% to 0.98%.

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1. Introduction

Vanadium is currently defined as a strategic metal that is widely used in many fields, such as the steel, chemical, and aerospace industries, because of its excellent properties [\(Zhou et al., 2009](#page--1-0)). In China, the vanadium slag derived by conversion from hot metal containing vanadium is one of the most important sources of vanadium ([Li and Xie,](#page--1-0) [2012\)](#page--1-0). Well-known methods that have recently been used to extract vanadium from vanadium slag include blank roasting–acidic leaching, calcium salt roasting–acidic leaching, sodium salt roasting–water leaching, and acidic leaching–solvent extraction ([Li et al., 2009\)](#page--1-0). Sodium salt roasting–water leaching technology is comparatively mature and has been applied extensively in many vanadium enterprises. However, the resultant low-valent vanadium from insufficient sodium salt oxidation roasting and the vanadium component with a silicon-wrapped structure affect the improvement of vanadium extraction yield, thereby limiting the comprehensive utilization of vanadium resources.

Therefore, several oxidation methods have been investigated to treat low-valent vanadium, including oxidative roasting and oxygen pressure acid leaching [\(Deng et al., 2010; Liu et al., 2011, 2014; Mu et al., 2011;](#page--1-0) [Zhou et al., 2012\)](#page--1-0). However, these extraction technologies have some disadvantages, such as the high cost and their tendency to pollute the environment with HCl, Cl_2 , SO_2 in roasting process. Thus, out-field

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<http://dx.doi.org/10.1016/j.hydromet.2015.04.005> 0304-386X/© 2015 Elsevier B.V. All rights reserved. intensification and multi-field coupling technologies represent the development trend of hydrometallurgical processing. Scientists have recently focused on the development of electrochemical leaching technology to handle copper sulfide, lead, zinc sulfide minerals, bismuthinite, silver sulfide, pyrite, and molybdenite because this technology is highly flexible, easily operated, and has a low capability for pollution [\(Antonijevic and Pacovic, 1992; Cao et al., 2009; Cruz et al., 2005;](#page--1-0) [Souza et al., 2007\)](#page--1-0). However, few studies have been conducted on low vanadium leaching under electric fields.

The present work focuses on the electro-oxidation hydrometallurgical leaching process for converter vanadium slag. The mechanism of the electro-oxidation hydrometallurgical process is discussed along with the factors associated with vanadium extraction.

2. Experimental

2.1. Materials

The converter vanadium slag used in this study for sodium salt roasting was obtained from Panzhihua, China. Its main chemical composition was analyzed by X-ray fluorescence, as shown in [Table 1](#page-1-0). 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. 1 and 2.](#page-1-0) As indicated in [Fig. 1](#page-1-0), vanadium slag contains much water-soluble vanadate and parts of lowvalent vanadium compounds, such as VO_2 , $Na_2V_4O_9$, and NaV_2O_5 .

Table 1 Composition of vanadium slag (wt.%).

| | | Na | | ັບ | COLLA | Mn | | cα | AI | Mg | | |
|-------|-------|--|-------------|------|--------------|------|------|------|------|------|------|------|
| 28.04 | 25.62 | 1212 14.14 the contract of the contract of | 715 ,,,, | 6.98 | 6.86 | 5.92 | 2.19 | 1.67 | 1.55 | 1.38 | 0.12 | 0.05 |

Fig. 2 suggests that the vanadium slag particles are irregular in shape and distribute unevenly.

2.2. Methods

The leaching experiments were conducted in a 300 ml beaker that was heated in a water base whose temperature constantly remained within $+1.0$ °C. This beaker was connected to a mechanical agitator. The samples were crushed and ground to -0.074 mm particles. When the distilled water was heated to the desired temperature, 10 g slag was added and stirred at the appropriate agitation speed for a required time. A stainless steel electrode with an area of 2 cm \times 4 cm was selected for the electro-oxidation process. A direct current (DC)-stabilized power source with current monitoring was controlled within the range of 0–1 A. The experimental setup is depicted in [Fig. 3.](#page--1-0)

Following a scheduled duration, the leach liquor was separated from the residue by vacuum filtration. The experiments were conducted under different leaching temperatures, leaching times, liquid-to-solid ratios, and current densities. Titration with ammonium ferrous sulfate was used to determine the concentration of vanadium in the solution. Then, the vanadium extraction yield was calculated using the following formula:

$$
\eta_V = \frac{V \cdot C_V}{m \cdot \omega_V} \times 100\%,\tag{1}
$$

where η_v is the vanadium extraction yield, %; V is the volume of leaching liquid, l; C_v is the vanadium concentration in the leaching liquid, g/l ; *m* is the weight of the vanadium slag, g; and ω_{v} is the content of vanadium in the vanadium slag, %.

The theoretical power consumption of a unit mass of vanadium is Q_T = 0.526 A·h/g (V₄O $^{2-}_9$ \rightarrow 4VO $^{-}_3$) according to Faraday's law. The current efficiency was calculated using the following formula:

$$
\eta_i = \frac{M \cdot Q_T}{I \cdot t} \times 100\%,\tag{2}
$$

Fig. 1. XRD pattern of vanadium slag. The state of vanadium slag. Fig. 2. SEM image of vanadium slag.

where η_i is the current efficiency, %; M is the actual mass of the oxidized vanadium, g; Q_T is the theoretical power consumption of a unit mass of vanadium, $A \cdot h/g$; *I* is the size of the current, *A*; and *t* is the time, h.

3. Results and discussion

3.1. Effect of anodic current density

The effect of current density on vanadium extraction was investigated from 0 to 1250 A/m2 at 80 °C for 40 min with a liquid-to-solid ratio of 4:1 ml/g. As depicted in [Fig. 4,](#page--1-0) the leaching yield of vanadium increased with the increase in current density. However, current efficiency decreased at high current density. Therefore, the optimum current density for energy saving was set at 1000 A/m^2 .

3.2. Effect of leaching temperature

The effect of leaching temperature on vanadium extraction was investigated from 50 °C to 100 °C. The anodic current density, liquidto-solid ratio, and leaching time remained constant at 1000 A/m^2 , 4:1 ml/g, and 40 min, respectively. [Fig. 5](#page--1-0) indicates that the leaching yield of vanadium increased gradually with the increase in leaching temperature because a high temperature can accelerate molecular motion and collision between the vanadium-bearing particles, thus ensuring full contact among reactants. Thus, the leaching temperature should be as high as possible in practice. Nonetheless, the optimum temperature is set at 80 °C in this study in consideration of the cost and energy savings.

3.3. Effect of liquid-to-solid ratio

The effect of liquid-to-solid ratio on vanadium extraction was examined from 1:1 ml/g to 8:1 ml/g at an anodic current density of 1000 A/m², a temperature of 80 °C, and a leaching time of 40 min. [Fig. 6](#page--1-0) shows that the leaching yield of vanadium increased when the liquid-to-solid ratio increased to 8:1 ml/g. However, a high ratio

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