



Extraction of vanadium from converter slag by two-step sulfuric acid leaching process



Junyi Xiang^a, Qingyun Huang^b, Xuewei Lv^{a,*}, Chenguang Bai^a

^a School of Materials Science and Engineering, Chongqing University, Chongqing 400044, China

^b School of Metallurgical and Materials Engineering, Chongqing University of Science and Technology, Chongqing 401331, China

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ABSTRACT

Because of its potential to achieve a high recovery rate of vanadium, the mechanical activation pre-treatment of vanadium-bearing converter slag followed by calcification roasting at low temperature and a two-step leaching process may be a viable alternative to the present roasting and one-step leaching process for vanadium extraction. The object of this study is to enhance the leaching efficiency of vanadium by mechanical activation and two-step leaching methods. The effects of solution acidity, liquid/solid ratio, particle size of the roasted slag, stirring speed, leaching temperature and time were investigated. The first stage leaching liquor was recycled in order to improve vanadium content in the leaching solution and reduce leaching solution consumption and wastewater discharge. Experimental results showed that approximately 81.8% vanadium was dissolved in the first stage of the leaching process with a solution pH of 2.5, and approximately 8.1% vanadium was dissolved in the second stage of the leaching process with a solution pH of 0. In the first stage, the vanadium leaching efficiency was significantly hindered by long recycle times, though this property also increased the content of vanadium in the leaching solution and greatly decreased the water consumption. In addition, mineralography and microstructures of the solid samples were studied by X-ray powder diffraction (XRD) and scanning electron microscopy (SEM) to help understand the mechanism.

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1. Introduction

To date, the most mature and commercial vanadium extraction technology for vanadium-bearing converter slag is the sodium salt roasting–water leaching process (Gupta and Krishnamurthy, 1992; Mahdavian et al., 2006). Unfortunately, this technology leads to a series of problems, such as fusion agglomeration of the slag at high roasting temperatures, emission of corrosive gases (HCl, Cl₂, SO₂ and SO₃), discharge of huge quantities of sodium-bearing wastewater, and utilization difficulty of the tailings (Li et al., 2016). Furthermore, the content of calcium in the converter slag must be strictly limited due to the formation of water insoluble calcium vanadates, which significantly reduce the recovery rate of vanadium (Aarabi-Karagani et al., 2010).

Calcification roasting–acid leaching appears to be a clean production method for extraction of vanadium from vanadium-bearing converter slag with less gas emission, less harmful wastewater and

little noxious solid waste poll. This process was first proposed by the Russia Tula factory in the 1970s, but it was not applied commercially due to the low recovery rate of vanadium (Zhang et al., 2015). According to the industry practices of Xichang Steel & Vanadium Corporation, Pangang Group, Sichuan Province, China, ~60 min of leaching time was needed for a maximum fraction (about 86%) of vanadium to dissolve (Lu et al., 2014). The production efficiency and cost are seriously hindered by such a long leaching time. The leaching of vanadium from converter slag can be divided into two distinct stages: stage one, where the leaching ratio is sharply increased over a short time, and stage two, where the leaching ratio is gradually increased over a long time (Zhao et al., 2014).

In the past several decades, a considerable number of investigations have been performed to obtain optimum conditions for improving the extraction efficiency of vanadium in calcification roasting–acid leaching processes. It was found that a decrease in the FeO content in a slag to 8% or below is shown to negatively affect the oxidation of vanadium in slag phases (Sadykhov et al., 2013). The key factor is the conversion degree of acid soluble

* Corresponding author.

E-mail address: lxuewei@163.com (X. Lv).

calcium vanadates which significantly influences the leaching efficiency. The ratio of vanadium extraction increases rapidly as the roasting temperature rises from 400 °C to 900 °C, then gradually decreased with further rising temperature (Zhang et al., 2015). It was found that the extraction of vanadium is strongly dependent on acid concentration, particle size, and liquid/solid (L/S) ratio (Zhao et al., 2014), while less dependent on temperature and stirring rate (Mirazimi et al., 2015). Most of the vanadium spinel phases form very small particles dispersed throughout the converter slag (Zhang et al., 2015) and are mostly inter-grown with olivine, which makes them difficult to liberate as valuable single minerals such that they can subsequently react with calcium compounds.

Mechanical activation can not only grind samples into extremely fine particles but also induce phase transformations, structural defects, strains at the grain level, and changes in the surface characteristics that enhance the reaction rate in the subsequent process (Balaz, 2008; Welham and Llewellyn, 1998). This approach has been reported to enhance the leaching of ilmenite concentrated ore (Sasikumar et al., 2004) and stone coal (Liu et al., 2014). It was found that the activation energies of the dissolution of both Ti and Fe in ilmenite concentrated ore decreased monotonically with activation time. The dissolution fraction of vanadium in stone coal increased by as much as 10 percentage point to 81.76% with 30 min of activation. The finer the vanadium slag, the higher the probability that single spinel minerals will be completely liberated from the slag, which results in more reactive sites exposed to the reagent.

An added drawback of the calcification roasting–acid leaching process is the fact that this process generates considerable quantities of acidic wastewater containing hazardous elements, such as V^{5+} , Cr^{6+} , and P (Fang et al., 2009), and little information is available on the effective treatment of such wastewater. Multistage leaching processes is an efficient way to improve the leaching rate and make full use of leaching agent. It has been reported to enhance the leaching of ilmenite, titanium slag (Balderson and Macdonald, 1999) and stone coal (Zhu et al., 2012). Furthermore, in our previous study, mechanical activation was found to decrease the roasting temperature, shorten the roasting time in the oxidation roasting step, and promote the subsequent leaching step (Xiang et al., 2017a, 2017b).

Hence, this work presents a study on the extraction of vanadium by a two-steps leaching process, starting from a mechanically activated converter slag in order to improve the vanadium leaching efficiency and productivity. The effects of acid concentration, leaching temperature, liquid/solid (L/S) ratio, leaching time, and stirring speed are evaluated. The recycling of the leaching liquor for the purpose of reducing water consumption, wastewater discharge, and emission of hazardous materials is also evaluated. With this study, the authors seek to improve the extraction efficiency and cleanliness of vanadium production from converter slag.

2. Experimental

2.1. Materials

The vanadium slag was acquired from the Panzhihua Iron and Steel Group Corporation, Sichuan Province, southwestern China. Vanadium slag (100 g) was first mixed with calcium carbonate (12.3 g) and was mechanically activated in a planetary ball mill (Retsch PM 100, Germany) for 40 min at a rotation rate of 400 rpm and a ball-to-slag weight ratio of 5:1. The mechanically activated slag was then roasted in a muffle furnace at 800 °C for 1 h. Calcium carbonate powder used in the roasting experiments had the calcite crystal structure and a purity of 99%. All the other reagents used for roasting, leaching and chemical analysis were of analytical grade

and of purity greater than 99.9%.

2.2. Procedure

The leaching experiments were performed at atmospheric pressure in a 500 mL three-necked flask equipped with a reflux condenser and a pH meter. The reaction mixture was heated by a thermostatic water bath and agitated by a magnetic stirrer.

For the first-stage leaching experiments, the leach solution was acidified by sulfuric acid to maintain a pH of 2.5 throughout the whole leaching process. Experiments were carried out at four different size ranges of the roasted slag (–48 μm, 48–75 μm, 75–125 μm and 125–150 μm), four different initial L/S ratios (5:1, 10:1, 15:1 and 20:1) and four different stirring speeds (0, 100, 200 and 300 rpm). Leaching temperature and holding time were also optimized in 30–90 °C range and under 60 min, respectively.

After the reactions, the slurry was filtered to separate the remaining unreacted solids from the leaching solution. The leaching solution was recycled for several times and then used for vanadium recovery. The unreacted solids were then used for the second-stage leaching experiment.

For the second-stage leaching experiment, five different sulfuric acid solutions with constant pH values of 2.5, 1.5, 0.8, 0 and –0.34 were investigated. Leaching parameters such as temperature (30, 50, 70 and 90 °C), initial L/S mass ratio (5, 10, 15 and 20) and holding time (0–60min) were also investigated.

After the second leaching experiments, the slurry was filtered out, and the leaching residue was washed with dilute water and finally dried at 120 °C for 24 h.

2.3. Characterization

The vanadium concentration in the solid samples and solution was determined by the ferrous ammonium sulfate titration method, while the concentration of other elements were checked by X-ray fluorescence spectrometry (XRF, Shimadzu XRF-1800). The dissolution rates of vanadium were determined by pipetting approximately 4 mL of the suspension from the flask at regular time intervals followed by centrifuging and chemically analyzing the supernatant solution. The percentage of vanadium dissolved was calculated by the following formula:

$$\mu(\%) = 100 L_{TV}/S_{TV} \quad (1)$$

where $\mu(\%)$ is the fraction of vanadium dissolved, L_{TV} is the total mass of vanadium in the leaching liquor, S_{TV} is the total mass of vanadium in the solid sample. It should be noted that, in the first stage, S_{TV} corresponds to the total mass of vanadium in the roasted slag, while in the second stage, it corresponds to the total mass of vanadium in the first stage residue.

The mineralogical compositions of the samples were assessed by XRD analysis using Rigaku D/max 2500 PC X-ray diffraction with Cu K α radiation ($\lambda = 0.154$ nm, 40 kV, 150 mA) at a scan rate of 0.3°/s. Optical images of the samples were acquired from a Zeiss optical microscopes. SEM/EDS analysis of the samples was carried out using a scanning electron microscope (VEGA 3 LMH; TESCAN) equipped with energy dispersive X-ray spectrometry (EDS, Oxford).

3. Results and analysis

3.1. Characterization of the vanadium slag

The XRD patterns of the vanadium slag and calcification-roasted slag are delineated in Fig. 1. For the vanadium slag, the major crystalline phases are ulvospinel (Fe_2TiO_4), spinel ((Mn, Fe) (V,

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