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Simultaneous extraction of vanadium and titanium from vanadium slag using ammonium sulfate roasting-leaching process



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ABSTRACT

Sodium and calcification roasting processes are traditional technologies to recover vanadium from vanadium slag. However, these processes are associated with many drawbacks, including high energy consumption, serious environment pollution, and the inability to simultaneously extract associated titanium resources. In this paper, a novel technology for simultaneous extraction of vanadium and titanium from vanadium slag was proposed, in which the vanadium slag was roasted with recyclable (NH₄)₂SO₄ (AS) at moderately high temperatures followed by dilute H₂SO₄ leaching. To enhance the extraction, an activation pretreatment of the vanadium slag through high-temperature water quenching was employed. The results demonstrated that the activation significantly accelerated the extraction, with the vanadium and titanium extraction increasing by 16% and 12%, respectively, compared with the raw vanadium slag. The extraction of vanadium and titanium were 91% and 77%, respectively, after roasting at an AS-to-vanadium slag mass ratio of 4:1 and 370 °C followed by leaching in a 6% H₂SO₄ solution. X-ray diffraction analysis indicated that the spinel phases in the vanadium slag, such as FeV2O4, Fe2TiO3, and Fe₂MnO₄, began to transform into (NH₄)₃V(SO₄)₃, (NH₄)₃Fe(SO₄)₃, (NH₄)₂Mn(SO₄)₂, and TiSO₄ at 320 °C and a nearly complete conversion could be achieved at 370 °C. The mass ratio of AS to vanadium slag significantly affected the extraction of both vanadium and titanium, which increased with the increasing mass ratio until an 8:1 ratio was achieved, after which, the extraction decreased. A stratification phenomenon of the vanadium slag and ammonium bisulfate at high AS/slag mass ratios was observed, which could be responsible for the decreasing extraction.

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1. Introduction

Vanadium is an important strategic metal and is widely used in the iron and steel industry, as well as in non-ferrous alloys due to its excellent hardness, tensile strength, and corrosion resistance [1]. Vanadium accounts for only 0.02% of the crustal weight, which although is a higher abundance than that of copper, zinc, and nickel, and ranks 22nd in the known elements, it does not naturally occur in its pure state [2]. Research has confirmed that vanadium initially presents in igneous rocks and gathers in a water-insoluble state of V(III) [3]. Because the ion radii of V(III), Mn(III), Fe(III), and Ti(III) are close, it is quite common for these ions to form a polymetallic symbiotic ore. Vanadium-titanium magnetite ore is a typical polymetallic symbiotic ore that is primarily found in Russia, China, and South Africa. In China, vanadium slag, a by-product of vanadium-

* Corresponding author. E-mail address: lic@scu.edu.cn (C. Li). titanium magnetite in the converter steelmaking process, is a typical vanadium metallurgical raw material, which contributes to more than 40% of V_2O_5 production in the country [4].

In general, it is almost impossible to separate vanadium from vanadium slag in an unenhanced procedure because there is little vanadium in vanadium slag that could dissolve in water, alkali, or acid directly [5,6]. Further, vanadium primarily occurs in the very stable spinel phase in vanadium slag. Theoretically, it is necessary to recover vanadium from vanadium slag by transforming the low-valence insoluble vanadium in the spinel phase into high-valent soluble vanadium [7–9]. The traditional commercial methods for vanadium extraction from vanadium slag are sodium roasting-water leaching and calcification roasting-acid leaching [10,11]. In the two methods, the vanadium slag is roasted with an additive, such as NaCl or CaO, above 850 °C to decompose the spinel phase structure and vanadium extraction of about 75–80% is obtained [12]. Recently, several studies have been conducted to enhance vanadium extraction. Li et al. researched the process of extracting



vanadium by calcification roasting and ammonium carbonate leaching and obtained 96% vanadium recovery [6]. Rashchi et al. revealed the effect of alkaline roasting and H₂SO₄ leaching on vanadium extraction from vanadium slag using surface response methodology [13]. Xie et al. investigated the oxidation behavior of the vanadium phase in vanadium slag using the sodium roasting process and achieved 90% vanadium extraction [14]. Although over 90% vanadium extraction could be obtained in these studies, a roasting temperature of at least 800 °C was required, which is energy intensive. The sodium-roasting and calcification-roasting processes also involve the emission of harmful gases and high additive consumption [15]. In addition, both the sodium and calcification roasting processes seldom allow for the simultaneous extraction of titanium, a high-value-added metal associated in vanadium slag, since the titanium cannot be dissolved during vanadium extraction. Therefore, it is necessary to develop a new extraction route.

The obvious advantages of the ammonium sulfate roastingleaching (ASRL) process are high efficiency, low energy consumption (roasting temperatures of only 300–400 °C), and environmental friendliness (ammonium sulfate is a recyclable extractant), especially in dealing with low-grade symbiotic minerals [16,17]. Saleh et al. researched the technology of extracting zinc by the ASRL process and obtained a 95% zinc extraction rate at 350 °C [18]. Highfield et al. investigated magnesium extraction from serpentine using the ammonium sulfate roasting process and determined that the silicate minerals could decomposed at 350 °C [19]. Li et al. used ammonium sulfate to decompose low-grade nickel laterite at 400 °C, where 90.8% nickel and 85.4% cobalt could be selectively extracted [20].

In this study, we proposed a novel ASRL process that can be used to simultaneously extract and recover vanadium and titanium from vanadium slag under mild conditions for comprehensively utilizing vanadium slag. In order to enhance the extraction, raw vanadium slag was melted and water quenched for activation, which could be easily realized in industry. The decomposition behavior and microstructure morphology of vanadium slag in the presence of ammonium sulfate at different roasting temperatures is investigated. Furthermore, the effect of the ammonium sulfate dosage on the extraction is discussed. The mineral composition of leaching residues is also characterized.

2. Experimental

2.1. Materials

Table 1

The vanadium slag used in this research was provided by Panzhihua Steel & Iron Co., Ltd, Sichuan province, China. The vanadium slag was crushed and milled to a thickness of 75 μ m by a ball mill after drying at 110 °C for 12 h. The chemical composition of the slag is shown in Table 1. The X-ray diffraction (XRD) patterns of vanadium slag are shown in Fig. 1. The XRD patterns indicate that FeV₂O₄ (Ref. code: 01-075-0317), Fe₂TiO₄ (Ref. code: 01-071-1141), FeMn₂O₄ (Ref. code: 01-075-0035), and Ca(Fe, Mg) (SiO₃)₂ (Ref. code: 00-024-0204) occur as the primary phase of the vanadium slag. (NH₄)₂SO₄ and H₂SO₄ were of analytical grade.

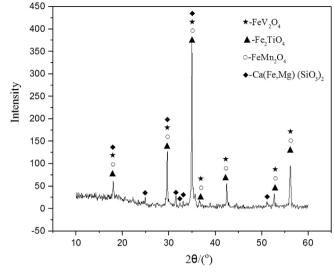


Fig. 1. XRD analysis of raw vanadium slag.

2.2. Procedure

In the water quenching process, the raw vanadium slag was placed in a high-purity SiO₂ crucible, which was positioned centrally and heated by MoSi₂ rods in an electric furnace (laboratory customization). When heated to 1500 °C in air atmosphere, the slag was dropped rapidly into a bucket located immediately below the hearth through a specially designed outlet and cooled by water in the bucket. After being crushed and milled, the water quenched vanadium slag was dried at 110 °C for 12 h. In each roasting test, the vanadium slag sample was evenly mixed with $(NH_4)_2SO_4$ (AS) at a predetermined mass ratio. The mixture was placed in a porcelain boat, heated at a rate of 10 °C/min until 370 °C, and then annealed for 90 min in an electric furnace (KSL-1200X, Heifei Kejing Materials Technology Co., Limited, Heifei, China). The roasted slag was then withdrawn and cooled in a desiccator to room temperature. The roasted samples were leached in 6 vol% H₂SO₄ at a liquid-tosolid mass ratio of 8:1 and 60 °C for 1 h. The obtained slurry was run through a vacuum filter. The leaching solution and residues were collected and analyzed to determine the leaching ratio of both vanadium and titanium. The simultaneous extraction of vanadium and titanium from vanadium slag using ammonium sulfate roasting-leaching process was shown in Fig. 2.

The ratio could be calculated by following equation (1):

$$\alpha = \{1 - (m_2 \times w_2) / (m_1 \times w_1)\} \times 100\%$$
(1)

where $m_1(g)$ is the mass of the vanadium slag used in the roasting procedure; $w_1(wt.\%)$ is the content of vanadium or titanium in the vanadium slag; $m_2(g)$ is the mass of the leaching residue obtained from the leaching procedure; and $w_2(wt.\%)$ is the content of vanadium or titanium in the leaching residue.

2.3. Characterization

The XRD (DX-2007, Fangyuan Instrument Factory, China), with

Chemical composition of the vanadium slag used in this study (wt./%).

Composition	V ₂ O ₅	TiO ₂	Fe ₂ O ₃	MnO	SiO ₂	MgO	CaO	Al_2O_3	Others
Wt./%	17.52	13.10	36.73	10.00	14.91	2.34	2.26	2.20	0.93

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