

Vanadium extraction from titano-magnetite by hydrofluoric acid



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ABSTRACT

Vanadium extraction from titano-magnetite by using hydrofluoric acid was put forward. The process of low intensity magnetic separation, hydrofluoric acid leaching, co-precipitation with NaOH, roasting with Na₂CO₃, circulating water leaching and precipitation of vanadium has been investigated. The results show that over 85% vanadium could be directly leached under the condition of sulfuric acid concentration of 5 mol/L, CaF₂ dosage of 5%, leaching temperature of 95 °C, leaching time of 3 h and liquid to solid ratio of 3 mL/g. With the pH value of 4.5, temperature of 40 °C and time of 30 min, the co-precipitation rate of vanadium in the acid leaching solution was over 99%. The water leaching rate of vanadium of the co-precipitation reached 95% under the condition of roasting temperature of 800 °C, Na₂CO₃ dosage of 6%, roasting time of 1 h, leaching temperature of 90 °C, leaching time of 40 min, liquid to solid ratio of 2 mL/g and leaching stage of 4. Finally, the total recovery of vanadium could reach 81% and the purity of the product V₂O₅ was 99.65% meeting the standard specification with precipitation of vanadium and calcination.

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1. Introduction

Vanadium is an important transition metal, which is widely used in high-quality ferrous, non-ferrous alloys and chemical industry due to its unique characteristic (Moskalyk and Alfantazi, 2003; Xiao et al., 2010). Vanadium is mainly existed in the form of combination with carnotite, mica, vanadinite, mottamite, patronite, by-product and so on (Aarabi-Karagani et al., 2010; Mirazimi et al., 2015; Gladyshev et al., 2015). There are two important vanadium sources in China, where one is stone coal ore and the other is vanadium titano-magnetite (Zeng et al., 2015; Zhu et al., 2012). Vanadium titano-magnetite is widely distributed in many provinces in China and the proven reserve is 9.83 billion tons. Furthermore, the total reserve of V₂O₅ reaches 25.96 million in the vanadium titano-magnetite (Qiu et al., 2013).

The extraction of vanadium from vanadium titano-magnetite basically includes two methods, namely direct extraction and indirect extraction from vanadium slag after the ironmaking and steelmaking (Guo et al., 2014). The direct extraction of vanadium from vanadium titano-magnetite was mainly applied by roasting with NaCl or Na₂SO₄ at above 1000 °C and water leaching. However, the dosage of NaCl was excessive high resulted in air pollution during the process (Li et al., 2011a, 2011b). The indirect extraction of vanadium from the vanadium slag was the main recovery technology of vanadium, where the process of roasting with Na₂CO₃, water leaching, purification and precipitation was operated (Li et al., 2011a, 2011b). However, the total

recovery of vanadium was less than 60% in the process. Due to the problem, the process of calcified roasting (direct roasting or no roasting), acid leaching, solvent extraction and precipitation was widely investigated (Zhao et al., 2014). High sulfuric acid concentration is usually used to obtain high recovery of vanadium. However, the problems of low leaching efficiency, high acid consumption and more acid wastewater have been occurred in the process (Zhang et al., 2013). Furthermore, it is very difficult to directly extract vanadium from the solutions due to highly acid concentration of solution, low concentration of vanadium and high content of impurity ions (Okudan et al., 2015; Ye et al., 2012).

Therefore, the aim of this paper is to research a novel technology of vanadium extraction from titano-magnetite. The acid consumption was decreased without environmental pollution in the process. Furthermore, the high total recovery of vanadium and high purity of the product V₂O₅ have been obtained.

2. Experimental

2.1. Materials

The sample of vanadium titano-magnetite ore was collected from Shanxi province, China. After crushing and grinding, the particle size of the sample was –0.074 mm accounting for 90%. The ore sample and vanadium concentrate after low intensity magnetic separation were analyzed by using inductively coupled plasma-atomic emission spectrometry (ICP-AES) and X-ray diffraction patterns (XRD), and the results are given in Table 1 and Fig. 1.

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Table 1

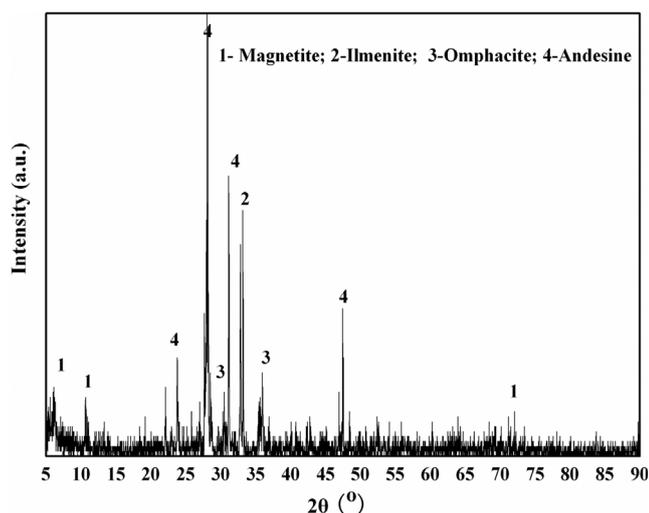
The main chemical composition of vanadium titano-magnetite and vanadium concentrate (wt.%).

Chemical composition	TFe	V ₂ O ₅	TiO ₂	Al ₂ O ₃	SiO ₂	CaO	MgO
Vanadium titano-magnetite	10.1	0.22	3.57	11.58	43.75	4.84	4.67
Vanadium concentrate	55.8	1.17	0.25	2.34	7.21	1.68	1.29

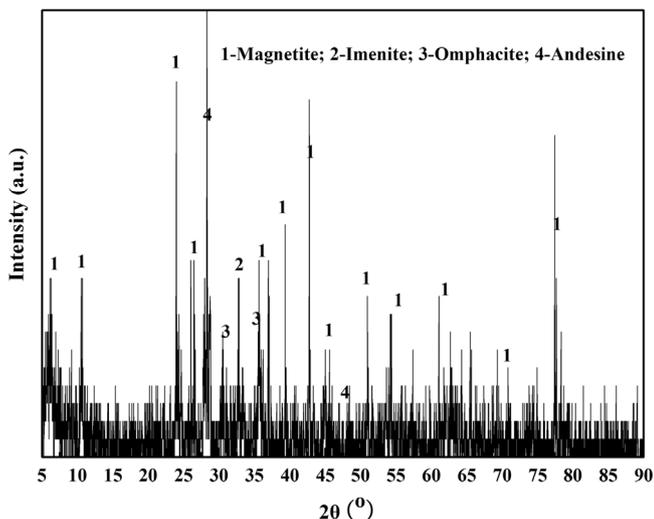
The analytical purity chemical reagents including sulfuric acid, calcium fluoride, sodium hydroxide, sodium carbonate and ammonium chloride from Dengke Chemical Reagent Technology Co., Ltd. were used. The water used in this study was distilled water.

2.2. Methods

The raw vanadium titano-magnetite was crushed and grinded to -0.074 mm accounting for 90%, and then the sample was separated with low intensity magnetic separation of 50 KA/m by using dry magnetic separator to obtain the vanadium concentrate. The vanadium



(a) Vanadium titano-magnetite



(b) Vanadium concentrate

Fig. 1. XRD pattern of vanadium titano-magnetite and vanadium concentrate.

concentrate was leached in hydrofluoric acid solution at certain temperature, liquid to solid ratio and leaching time. The acid leaching solution was obtained after filtration, and then was precipitated with NaOH and NaClO₃ by adjusting solution pH value of 4.5 after removal of aluminum and calcium (Ye et al., 2012). The solid containing vanadium and iron was obtained after filtration, and then was roasted with Na₂CO₃ in a furnace at a desired temperature. The roasted sample was obtained after cooling to room temperature. Then the roasted sample was circularly leached with water and filtered to obtain water leaching solution with high concentration of vanadium. Polymer ammonium vanadate was crystallized from the high concentrated vanadium solution by using NH₄Cl. Finally, the V₂O₅ product of high quality was obtained by calcinations of polymer ammonium vanadate at 520 °C. The flow sheet of vanadium extraction from vanadium titano-magnetite is shown in Fig. 2. In order to guarantee more accurate result, the experiment was repeated with three times, and the average value of three parallel experiments was selected as final data.

2.3. Detection measurement

The contents of chemical composition were analyzed by using a Plasma1000 inductively coupled plasma atomic emission spectrometry (Light Ace HK Ltd., China).

X-ray diffraction patterns were obtained by using a Rigaku D/MAX-III X-ray diffraction meter with Cu K α radiation (Panalytical B.V., Netherlands).

The leaching rate of vanadium was calculated by the following equation.

$$\alpha = \frac{m-n}{m} \times 100\% \quad (1)$$

Where: α is the leaching rate of vanadium (%), m is the vanadium quantity of leaching sample (g) and n is the vanadium quantity of leaching residue (g).

3. Results and discussion

3.1. Acid leaching with CaF₂

The vanadium is mainly fettered in crystal structure of magnetite in the form of V(III) in the vanadium concentrate. The V(III) is not easily leached by low concentrated sulfuric acid. The hydrofluoric acid is a strong corrosive acid, so the agent of CaF₂ was added in sulfuric acid to strengthen the leaching process. The effect of sulfuric acid concentration and CaF₂ on recovery of vanadium is shown in Fig. 3.

It can be seen from Fig. 3 that nearly 85% of vanadium recovery could be obtained with 8 mol/L sulfuric acid, but only 5 mol/L sulfuric acid was sufficient with adding 5% CaF₂. The recovery of vanadium was increased with increasing sulfuric acid concentration and CaF₂. The proper acid leaching condition was selected with sulfuric acid concentration of 5 mol/L, CaF₂ dosage of 5%, leaching temperature of 95 °C, leaching time of 3 h and liquid to solid ratio of 3 mL/g. Under the condition, the recovery of vanadium was 85.64%. The effect of CaF₂ on recovery of vanadium was investigated. The result on XRD pattern of acid leaching residue is shown in Fig. 4.

It is indicated from Fig. 4 that the diffraction peak of magnetite, ilmenite, omphacite and andesine were decreased after acid leaching without using CaF₂. Furthermore, the phase of quartz was generated, in which the dissolution reaction of magnetite, ilmenite, omphacite and andesine could take place with sulfuric acid but not quartz. However, the diffraction peak of magnetite and quartz were obviously decreased by acid leaching with 5% CaF₂, where the magnetite was further dissolved and the dissolution reaction of quartz with hydrofluoric acid has taken place. Hydrofluoric acid could obviously damage the crystal structure of minerals containing vanadium. The Gibbs free energy of

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