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Enrichment of valuable elements from vanadium slag using superconducting HGMS technology

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Abstract

Vanadium slags is a kind of vanadiferous solid waste from steelmaking process. It not only occupies land, pollutes environment, but also leads to waste of resources. Based on the difference of magnetic susceptibility of different particles caused by their chemical and physical properties from vanadium slag, a new technology, superconducting high gradient magnetic separation was investigated for separation and extraction of valuable substances from vanadium slag. The magnetic concentrate was obtained under optimal parameters, i.e., a particle size -200 mesh, a magnetic flux density of 0.8 T, a slurry concentration of 5 g/L, an amount of steel wools of 25 g and a slurry flow velocity of 2 L/min. The content of Fe_2O_3 in concentrate could be increased from 39.6% to 55.0% and V_2O_5 from 2.5% to 4.0%, respectively. The recovery rate is up to 42.9%, and the vanadium slag has been effectively reused.

Keywords: Vanadium slags, Superconducting HGMS, Ferric oxide, Vanadic oxide, Extraction and separation

1. INTRODUCTION

Because environmental pollution is becoming increasingly serious, it must be paid much more attention. Meanwhile, China is currently in rapid progress of industrialization, which has resulted in a mass of industrial solid waste. How to treat this waste has become an urgent issue. Vanadium slags are the primary solid waste produced by ironmaking from blast furnace. On the one hand, the vanadium slags can occupy large amounts of green space such as farm land. On the other hand, it can cause serious pollution to the water and soil. If these slags can be used appropriately, environmental problems can be effectively controlled and bring huge economic benefits.

As well known, vanadium is a kind of valuable rare metal, which is widely used in steel, automobile, aviation and biology fields[1,2]. In China, vanadium-titanium magnetite resources are abundant, but the grade of vanadium is quite low. For example, the content of V_2O_5 in vanadium-titanium magnetite produced by Chengde Iron & Steel Company located in Hebei Province of China is only 0.2%. And the other vanadium-titanium magnetite produced by Panzhihua Iron & Steel Company located in Sichuan, the grade of V_2O_5 is 0.25%. Vanadium in vanadium-titanium magnetite mainly exists in the form of compounds. These ores cannot be used directly to extract vanadium due to the content of vanadium is low, and the cost is too high. Hence, it need to pre-enrichment vanadium ore and carried out vanadium extraction.

The extraction rate of vanadium from vanadium slags using chemical methods is very high, but chemical methods bring serious secondary pollution problems[3,4]. The superconducting high gradient magnetic separation (HGMS) is a new kind of magnetic separation technology. It is physical separation method based on the difference in magnetic properties of different substance[5]. The high saturation magnetic matrix (steel wools) is filled in the uniform background magnetic field so that the magnetic field gradient increased substantially and thus substance with a different magnetism could be separated. The technology is developed from conventional ferromagnetic technique. Instead of copper coils, superconducting coils are used to make magnetic flux density rise to more than 10 T, which enables the technology to be applied in more areas. In addition, the technology is shown great energy conservation potential because the superconductor is nonresistant at operating temperature[6]. And the separation efficiency is satisfied especially for weak magnetic material. However, to the best of our knowledge, the superconducting HGMS technology has rarely been used for slag treatment. And the content of V_2O_5 in vanadium slag usually is about 1.5%[3], which is economically valuable to extract vanadium from vanadium slag. The purpose of our study is to separate and reuse Fe_2O_3 and V_2O_5 from vanadium slags using superconducting HGMS technology.

2. EXPERIMENTAL

2.1. The raw materials

The vanadium slags samples which after water leaching were provided by Chengde Iron & Steel Company. The samples were ground to 200 mesh for component testing. The samples were analyzed by XRD with the diffraction

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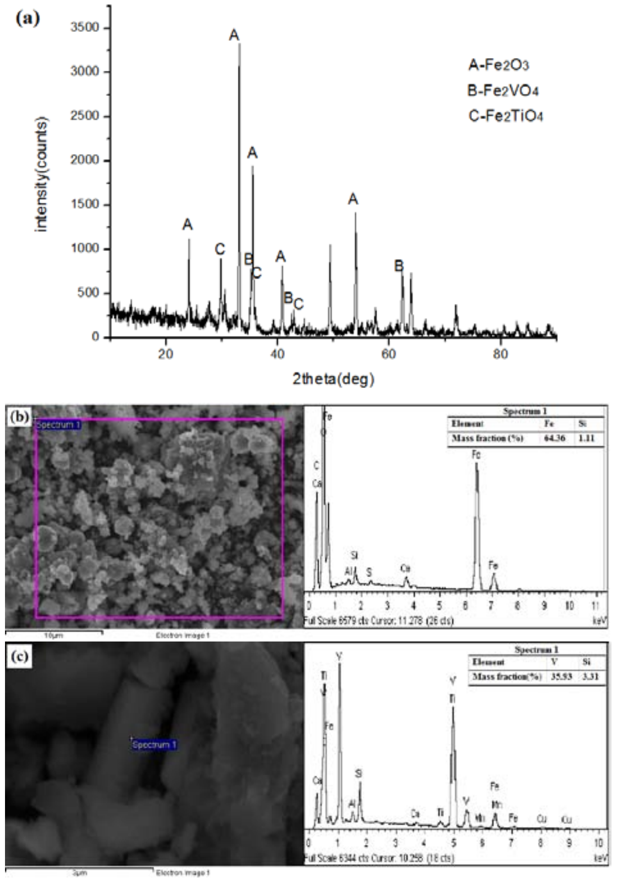
TABLE 1
CHEMICAL COMPOSITION OF VANADIUM WASTE SLAG.

Chemical composition	Fe_2O_3	Al_2O_3	V_2O_5	TiO_2	SiO_2	CaO	MgO	Cr_2O_3	MnO	Na_2O
Mass fraction （%）	39.56	3.32	2.50	9.05	25.49	2.52	2.75	3.59	4.10	6.47

TABLE 2
THE MAIN PARAMETERS OF MAGNET.

Item	**Specification**
Central magnetic field strength	5.5 T
Room temperature aperture	300 mm
Length of magnet (not including iron shield)	1110 mm
Height of magnet (not including iron and service tower)	780 mm
Working current	150 A
Magnets inductance	127 H



(a) XRD analysis of the raw vanadiferous slags
(b) Element content of Fe in iron oxide particles
(c) Element content of V in vanadium contained particles
Fig. 1. Compositions and microstructure of vanadium slags.

condition of 2 θ/θ coupling continuous scanning, step width of 0.02, Cu target, Voltage 40 kV, and Current 150 mA. The results are shown in Fig. 1. It is clearly that V, Fe and Ti are existed in the form of Fe_2O_3, Fe_2VO_4 and Fe_2TiO_4. The XRF analysis results of the samples are presented in Table 1. Fe_2O_3 and V_2O_5 are the targeted valuable substance of this study. According to Table 1, the percentage of Fe_2O_3 and V_2O_5 are 39.6% and 2.5%, respectively. Different substances have different magnetic

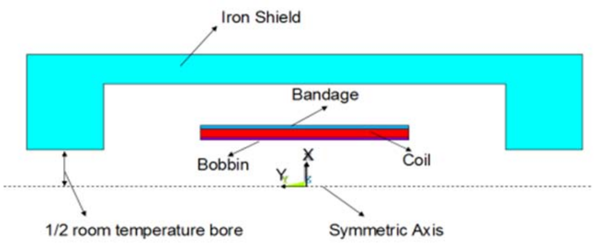


Fig. 2. The basic structure of magnet.

susceptibility, Fe_2O_3 and V_2O_5 can be separated and reused from vanadium slag according to the difference of magnetic susceptibility. In this study, Fe_2O_3 and V_2O_5 will be extracted and enriched by superconducting HGMS technology.

2.2. The steel wools

Magnetic particles were captured by the magnetic matrix i.e., steel wools. The steel wools were provided by Changsha Yigao Mining and Metallurgy Environmental Protection Materials, Ltd. The steel wools are made of 304 Stainless Steel which is a very common Stainless Steel species in China. The chemical composition is as follows (wt %): C≤0.08, Si≤1.0, Mn≤2.0, Cr: 18.0~20.0, Ni: 8.0~10.5, S≤0.03, P≤0.035, N≤0.1. The diameter of wool is 0.02 mm. Steel wools can raise the gradient of the magnetic field and increase the magnetic force acting on magnetic particles.

2.3. Magnet's primary structure

The magnet mainly consists of a superconducting coil, vacuum du watts, chiller, electric control instrument, and iron shield. The superconducting coil and iron shield are shown in Fig. 2. The main parameters of the magnet are exhibited in Table 2.

2.4. Ancillary equipment

Auxiliary equipment consists of peristaltic pump (WT600-1F/KZ25), analytical balance (AUY220), constant temperature oven (JXX1-277607), X-ray camera (D/MAX-RB), Scanning Electron Microscope (SUPRATM55), constant temperature magnetic stirrer (HJ-3) and so on.

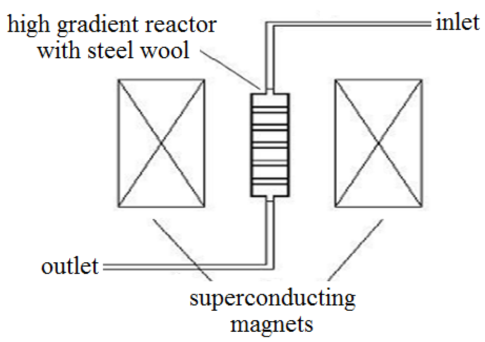


Fig. 3. Schematic diagram of superconducting HGMS.

2.5. Separation device

Low temperature cooling system provides the necessary environment for superconducting magnet. In order to ensure that the entire temperature consistency and stability of superconducting system, superconducting coils are immersed in liquid helium. Low temperature cooling system is designed to be after-condenser mode. Evaporation of helium is condensed again in condenser, then returned to the liquid helium pool so that the superconducting magnets have become a zero evaporation system [7].

The size of the separation device is 440 mm in length and 40 mm in diameter. Hoses are connected to both ends of the separation device, and it is fixed at the center of superconducting magnetic separation machine. The experimental schematic diagram of the separator is shown in Fig. 3. The prepared slurry (the dispersion medium is water) is fed into the high gradient reactor via the inlet. Magnetic particles are captured by the steel wools and nonmagnetic particles are to be exported with the slurry through the outlet.

The particles in magnetic field were under not just the stress of magnetic force, but a combined stress including fluid force, gravity, buoyancy, etc. Magnetic flux density, particle size, slurry concentration, slurry velocity and loading of steel wools all have significant effect on magnetic separation efficiency.

3. RESULTS AND DISCUSSION

3.1. Effect of the particle size

The effect of particle size on separation efficiency was investigated from 100-325 mesh. The experiments were carried out under the condition of a magnetic flux density of 1.0 T, a slurry concentration of 10 g/L, a slurry flow velocity of 1 L/min and a quality of the steel wools of 35 g. The results are shown in Fig. 4. It can be seen from Fig. 4 that the particle size of the samples has little effect on the separation efficiency. With the decrease of the particle size, the iron content is slightly increased while the vanadium decreases slightly. Excessive grinding will increase costs, so subsequent experiments selection -200 mesh is more appropriate.

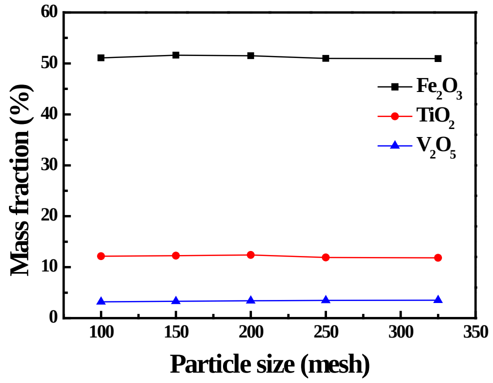


Fig. 4. The influence of particle size on separation efficiency.

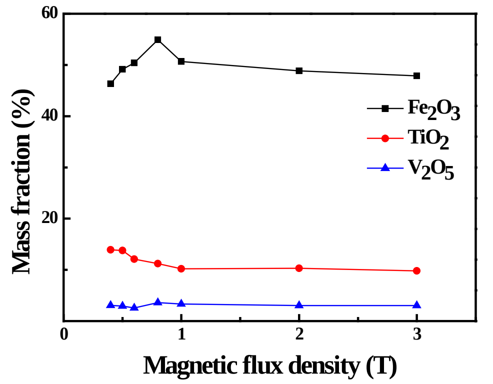


Fig. 5. The influence of magnetic flux density on separation efficiency.

3.2. Effect of the magnetic flux density

In the experiments, different magnetic flux densities from 0.2 to 3.0 T were attempted with a slurry concentration of 10 g/L, a slurry flow velocity of 1 L/min and a quality of the steel wools of 35 g. Then take the magnetic concentrate which was captured by steel wools to chemical analysis. The results are shown in Fig.5. According to Fig.5 and Table 1, the content of Fe_2O_3 in magnetic concentrate increased from 39.6% to 55.0%. As the magnetic flux density became stronger, the content of Fe_2O_3 did not continually increase during the processing. With the increase of the magnetic flux density, some weak particles with low iron content were absorbed by steel wools because of magnetic flocculation, causing a decrease of iron oxide content. When the magnetic flux density was 0.8 T, iron content reached up to the height, and V_2O_5 content also reached the maximum (4.0%). It was concluded that 0.8 T was the optimal magnetic flux density in the above conditions.

3.3. Effect of the slurry concentration

Based on the above test results, the optimum magnetic flux density was 0.8 T and fixed at the optimal value in the subsequent experiments. The effects of slurry concentration on separation efficiency were investigated under the following conditions to test: a slurry concentration from 1 to 7 g/L, a slurry flow velocity of 1 L/min and a quality of the

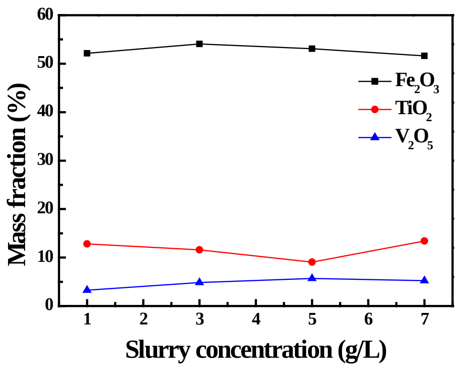


Fig. 6. The influence of slurry concentration on separation efficiency.

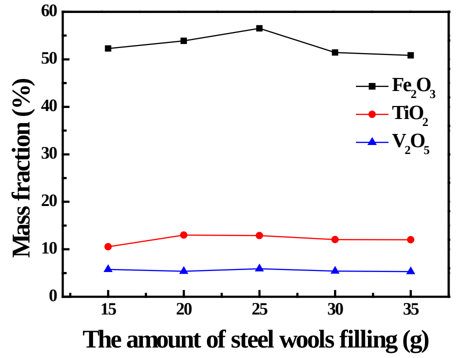


Fig. 7. The influence of amount of steel wools on separation efficiency.

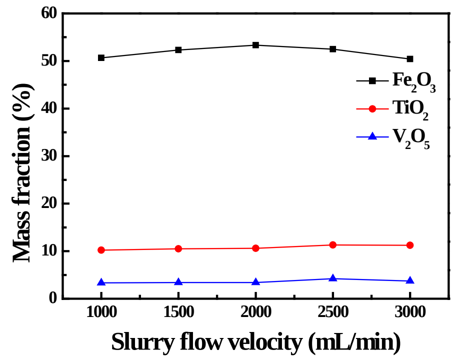


Fig. 8. The influence of slurry flow velocity on separation efficiency.

steel wools of 35 g. The results are shown in Fig.6. As seen from Fig.6, the slurry concentration has little effect on separation efficiency. When the concentration was about 5 g/L, the experimental results were the best. The slurry concentration is an important parameter because it affects production efficiency in practical production. The optimal value for slurry concentration was selected as 5 g/L.

3.4. Effect of the amount of steel wools filling

To investigate the effects of the amount of steel wools on separation efficiency, the amount of steel wools was changed from 15 to 35 g/L, with magnetic flux density of 0.8 T, slurry concentration of 5 g/L, slurry flow velocity of 1 L/min. The results are exhibited in Fig. 7. It can be seen from Fig.7 that when the mass of filling steel wools was 25 g, the content of Fe_2O_3 reached up to the maximum, and the content of V_2O_5 was almost the same changing trend under the different steel wools mass. The optimal value of steel wools mass was 25 g.

3.5. Effect of the slurry flow velocity

The experiments were performed with slurry flow velocity from 1 to 3 L/min, and other parameters were fixed at their optimal value. The results are exhibited in Fig. 8. It is clearly that the flow velocity has a weak effect on the separation efficiency and the optimal value was 2 L/min.

3.6. Comprehensive Condition Experiment

Based on extensive comparative tests, the optimum technical parameters are obtained: sample size of 200 mesh, magnetic flux density of 0.8 T, slurry concentration of 5 g/L, the amount of steel wools of 25 g and slurry flow velocity of 2 L/min. The recycling rate is up to 42.9%, the content of Fe_2O_3 and V_2O_5 are reached about 55.0% and 4.0%, respectively.

3.7. Discussion

In the magnetic field, particles are under a three-way competition: magnetic force, gravitational or inertial force, attractive or repulsive interparticle force. Magnetic force plays an important role in the separation processing. The magnetic force (F_M) is calculated by the following equation[8]:

$$\mathbf{F_M} = V\chi B \frac{dB}{dx} \quad (1)$$

where χ is magnetic susceptibility, V is particle volume, B is Magnetic flux density, dB/dx is magnetic flux density gradient.

According to the formula, F_M is mainly affected by the particle size, magnetic susceptibility, magnetic flux density and magnetic flux density gradient. The diameter of the particle is tiny, so the granularity of vanadium slag has little influence on magnetic processing. Magnetic flux density and magnetic susceptibility are the major influencing factor.

Another important force is drag force, it can be calculated by the following equation[9]:

$$\mathbf{F_D} = 6\pi\eta r_p(\mathbf{v}_f - \mathbf{v}_p) \quad (2)$$

where η is the rate of viscosity, r_p is the radius of particle, v_f is the slurry flow velocity and v_p is the particle velocity.

The magnetic particles can only be separated from non-magnetic particles when the condition $F_M > F_D$. The increase of slurry flow velocity would strengthen the hydrodynamic viscous resistance; in other words, F_D increased, the resultant force acting on the magnetic particles decreased. To get a good separation efficiency must control these two forces[10].

4. CONCLUSIONS

As a new kind of technology, superconducting HGMS was proved to be an effective method to recycle the valuable substances from vanadium slags. The results show that the valuable substances in vanadium slags (such as Fe_2O_3, V_2O_5) can be efficiently recycled under the appropriate parameters, i.e., a particle size -200 mesh, a magnetic flux density of 0.8 T, a slurry concentration of 5 g/L, an amount of steel wools of 25 g and a slurry flow velocity of 2 L/min. Laboratory scale, the recovery rate of 42.9% of concentrate was obtained. The content of Fe_2O_3 was increased from 39.6% to 55.0% and V_2O_5 from 2.5% to 4.0%. The vanadium slags had been effectively reused.

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