



Change in phase, microstructure, and physical-chemistry properties of high chromium vanadium slag during microwave calcification-roasting process

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ABSTRACT

The effects of microwave roasting and conventional roasting on oxidation behavior of high chromium vanadium slag (HCVS) during calcification-roasted process were compared in terms of the phase transformation, bonding structure change, valence state, microstructure, particle size, and surface properties of HCVS before and after microwave roasting and conventional roasting, which were characterized by XRD, FT-IR, XPS, SEM, BET specific surface area, and contact angle measurement, respectively. The temperature characteristics of HCVS during microwave and conventional roasting process were also investigated. HCVS was heated in a microwave field, and the heating time was shortened to 16 min at a microwave power of 2 kW, and to 58 min for conventional roasting. When compared to conventional roasting, microwave roasting promoted the oxidation degree of HCVS to a greater extent. The relative proportion of V^{5+} increased to 84.3% after microwave roasting and 76.9% after conventional roasting. The micrographs of HCVS after microwave roasting exhibited a significant change in morphology that involved reduction in particle size and subsequently an increase in roughness and porosity of the solid. BET specific surface area after microwave roasting was significantly increased from 16.3 m²/g to 45.1 m²/g. Microwave roasting decreased the contact angle to liquid and enhanced the wetting properties of HCVS. The leaching ratio of vanadium and chromium after microwave roasting increased from the initial values of 12.7% and 0.4% to 95.9% and 4.2%, respectively, and exceeded 82.5% and 2.1%, respectively, after conventional roasting.

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1. Introduction

Vanadium is a strategic metal and its major products include V₂O₅, V₂O₃, ferrovanadium that are widely applied in the metallurgical, material, and chemical industries due to their physical and chemical properties [1–3]. Vanadium is conventionally regenerated from recovered materials, such as vanadium titanium magnetite, stone coal, spent catalyst, fly ashes from oil industry, and vanadium slag, among which vanadium slag is the most important source for vanadium production [1,4,5]. Vanadium slag is the direct raw materials for vanadium extraction and is produced from vanadium titanium magnetite and accounts for approximately 60% of the vanadium production globally [6–8]. The vanadium slag that contains 5–8 wt% of vanadium and 2–5 wt% of chromium and is generated during the iron and steel-making process using vanadium titanium magnetite [1,9–13]. Recently, the production of ordinary vanadium titanium magnetite decreased with the development

of the steel industry. High chromium vanadium titanium magnetite has attracted significant research attention. The vanadium slag produced from high chromium vanadium titanium magnetite exhibits high chromium content. The content of chromium in vanadium slag increased to 12.54 wt% and even exceeded 12.54 wt%, thereby leading to several environment problems [14]. Currently, high chromium vanadium slag (HCVS) is not used for industrial production and only few studies examined HCVS [14–16]. Therefore, it is important to investigate HCVS.

The conventional vanadium recovery process is characterized by salt (NaCl, Na₂CO₃, or Na₂SO₄) roasting at 750–850 °C under an oxidation environment in a rotary kiln or multiple hearth furnace [8,17,18]. However, given low vanadium extraction ratio in single roasting, multiple roasting processes should be applied, thereby leading to the consumption of a large amount of energy [19]. Furthermore, the widespread application of the process is restricted given the limited raw materials and environmental hazards from SO₂, and Cl₂ emissions [2,8]. In order to avoid the emission of toxic gases, calcium roasting and sub-molten salt are reported to extract vanadium [19–21]. Although sub-molten salt is used to extract vanadium, it consumes large amounts of NaOH, thereby

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leading to increased production costs and environmental problems [2]. Calcification-roasting with lower costs of additive, absence of pollution gas emissions, and absence of sodium in tailings is a relatively clean and economic method [21].

With increases in the awareness of environmental protection, different intensification processes are proposed for the recovery of vanadium from vanadium slag. Among the processes, microwave roasting technology attracts increasing attention due to its distinct performance such as volumetric heating, selective heating, uniform heating, and non-thermal effect [22–24]. When compared to conventional roasting, microwave roasting can be dissipated in the volume of the material and transformed into inner energy [25]. The advantages of microwave roasting include decreasing the roasting temperature, accelerating the oxidation and decomposition of minerals, decreasing the holding time, saving energy, and obtaining a homogeneous microstructure [18,24,26,27]. Additionally, microwave roasting reduces environmental pollution and harmful waste discharge and alleviates pressure on the environment [28]. Therefore, a major challenge involves identifying a cost-effective and environment-friendly vanadium and chromium recovery process.

In the present study, the effects of microwave roasting and conventional roasting on oxidation behavior of HCVS with CaO during the calcification-roasting process were investigated. The heating curves of HCVS during microwave roasting and conventional roasting processing were examined. The crystal structure, bonding structure change, valence state, microstructure, particle size, and surface properties of HCVS were characterized by XRD, FI-TR, XPS, SEM, BET specific surface area, and contact angle measurement, respectively. In order to evaluate the effect of microwave roasting and conventional roasting on the leaching ratio of vanadium and chromium, leaching experiments were performed before and after microwave roasting and conventional roasting.

2. Experiments

2.1. Materials

HCVS produced from Jianlong Iron & Steel co., Ltd. (Heilongjiang Province, China) was used in the study. The chemical compositions of HCVS were characterized by X-ray fluorescence (XRF, Shimadzu XRF-550). The results are presented in Table 1. As shown in Table 1, the HCVS corresponds to a vanadium slag with a vanadium content of 11.6% and a chromium content of 11.3%. Additionally, X-ray diffraction (XRD) was also performed to further identify the component phases of HCVS. The results are shown in Fig. 1. The main phase of the HCVS corresponded to spinel-(Mn, Fe)(V, Cr)₂O₄, fayalite-Fe₂SiO₄, and augite-(Fe, Mn)₂SiO₄.

2.2. Experimental procedure

2.2.1. Microwave roasting

Prior to the microwave roasting, HCVS was dried in an oven for 24 h and subsequently HCVS was mixed with CaO at a m(CaO)/m(V₂O₅) ratio of 0.95. Microwave roasting experiments were conducted in a 2.45-GHz microwave oven (Tangshan Nano Source Microwave Thermal Instrument Manufacturing Co., Ltd) with an automatic temperature control system. The temperature of the test sample in the oven was measured continuously using a stainless steel-sheathed, K type thermocouple that was closed to the sample. The prepared samples were

Table 1

The chemical compositions of HCVS (mass fraction, %).

Component	TFe	FeO	SiO ₂	V ₂ O ₅	CaO	Cr ₂ O ₃	MnO	TiO ₂	Al ₂ O ₃
Mass fraction/%	34.7	22.8	20.5	11.6	1.4	11.3	8.7	6.7	3.5

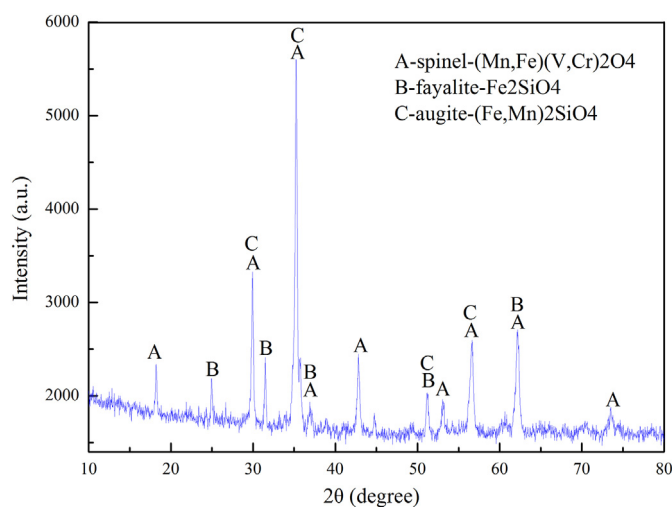


Fig. 1. XRD pattern of HCVS.

placed in a 200-mL microwave-transparent crucible and heated to 850 °C for 1.5 h. In order to observe the temperature rising curves of HCVS in the microwave field, microwave roasting experiments were performed at different power settings corresponding to 1 kW, 2 kW, and 3 kW. After microwave roasting, the samples were removed and cooled to room temperature in air. Subsequently, HCVS were milled and ground with a grinder (GJE100–1, Jiangxi Tongyong chemical experiment system type equipment Co., Ltd) for 3 min.

2.2.2. Conventional roasting

The conventional roasting experiments were conducted with a muffle furnace (XL-100, Hebi City Billion Yan Instruments Co. LTD). Then, the sample was treated by conventional roasting using roasting temperature, m(CaO)/m(V₂O₅) ratio, and roasting time as microwave roasting. The roasting power were performed with 2 kW. Then, the prepared samples were placed in a ceramic crucible for conventional roasting. After conventional roasting, HCVS were milled and grinded with a grinder as microwave roasting for 3 min.

2.2.3. Leaching experiments

The HCVS used in conventional roasting was also dried for 24 h and subsequently was mixed with CaO at a m(CaO)/m(V₂O₅) ratio of 0.95. The leaching experiment was performed using sulfuric acid (20% v/v) in a commercial magnetic stirrer water bath pot (DF-101, Gongyi Electric Equipment Co. LTD) at the atmosphere. The leaching temperature was 95 °C, and the leaching time was 2 h. A solid/liquid (S/L) ratio of 10 g/L was used in all the leaching experiments. The stirring speed was maintained at 500 min⁻¹ to ensure that all particles were suspended in the leaching solution. When the sulfuric solution was heated to the scheduled temperature, the sample was added into a 250-mL three-necked flask. After leaching, the solution was filtered via vacuum filtration. In order to thoroughly separate the water-soluble vanadates and chromates from the undissolved particles, the residual solids were washed with hot water several times during the filtration.

2.3. Analytical techniques

In the study, X-ray diffraction (XRD, X' Pert Pro MPD/PW3040, PANalytical B.V. Corporation, Netherlands) analysis via Cu K α radiation with a 2 θ angle ranging from 10° to 80° was conducted to characterize the component phases of HCVS. Fourier transform infrared spectroscopy (FT-IR, Nicolet-380, Thermo Corporation, USA) analysis was performed to determine the functional group and track the course of the chemical reactions with a spectrum ranging from 400 cm⁻¹ to 4000 cm⁻¹. Additionally, X-ray photoelectron spectroscopy (XPS)

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