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Flotation recovery of vanadium from low-grade stone coal

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Abstract: Pre-concentration of vanadium from low-grade stone coal by the method of desliming–flotation was investigated. The mineral composition and microstructure of stone coal were studied systematically by means of X-ray fluorescence spectrometry (XRF), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results show that selective separation of vanadium-bearing minerals can be achieved by flotation in acidic solution using melamine (EA). The final vanadium concentrate with V_2O_5 grade of 1.88% and recovery rate of 76.58% is obtained by desliming–flotation process and 72.51% of the raw ore is rejected as tailings. The pre-concentration of vanadium from low-grade stone coal can increase V_2O_5 grade and decrease the content of acid consuming minerals, which would enable economical utilization of metallurgical vanadium extraction technology.
Key words: vanadium; stone coal; flotation; pre-concentration

1 Introduction

Vanadium is an essential microelement and widely distributed in nature. As an important strategic material, it has extensive applications in various fields, including steel industry and alloy materials [1,2]. In China, vanadium mainly exists in vanadic titanomagnetite and stone coal. Stone coal is a unique vanadium resource with huge reserve [3,4]. It was reported that the gross reserve of V_2O_5 in stone coal was 118 million tons, which was 2.7 times that in vanadic titanomagnetite and exceeded the total reserve in other countries [5]. The grade of V_2O_5 in stone coal is generally 0.13%−1.2%.

As a conventional extracting process of vanadium from stone coal, salt roasting−water leaching is being eliminated due to the consequent problems, such as serious environment pollution and low recovery in production [6]. In recent decades, many novel technologies have been proposed and developed to improve the extracting process, including blank roasting-acid leaching, direct acid leaching and oxygen pressure acid leaching [7]. Unfortunately, these researches only focused on the recovery rate of vanadium and pollution problem, instead of the production cost. In 2012, the market price of V_2O_5 decreased to about $10000 per ton [8], close to the production cost. As a result, only relatively high-grade ore (>0.5% V_2O_5) has economic value and is worth smelting, which only accounts for 40% of the total ore [9,10]. Considering the low economic benefit and heavy demand for vanadium resources, it is becoming an urgent task to develop novel process for the effective utilization of low-grade stone coals [11].

Beneficiation of low-grade ores before metal extracting process is an effective method to raise the grade of metal and further reduce production cost. ZHAO et al [12] used gravity separation to pre-concentrate vanadium from stone coal. Even though only 9.7% vanadium was lost by this method, the yield of tailing was low and only accounted for 28.9% of the feed ore. Among various kinds of beneficiation methods, flotation has been widely used in the treatment of sulfide and oxide ores due to its high throughput and selectivity [13,14]. In stone coal, vanadium mainly exists in illite, muscovite, roscoelite, and kaolin in the form of isomorphism. It also appears in tantalite and garnet in the form of absorption [15]. Besides, quartz, calcite and carbonaceous are found to be the main gangues in stone coal [16]. Although many studies have been reported on the flotation process of these minerals in the past decades, there is less information on pre-concentration of

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vanadium in low-grade stone coal by the method.

This work focused on the desliming process and flotation technology of low-grade stone coal in Shanxi province on the basis of its mineralogy. Recovery of slime concentrate was carried out in order to avoid its adverse effect on flotation. Then, flotation condition tests were conducted to study the effects of grinding fineness, pulp pH value and kinds and dosages of reagents on the recovery and grade of V_2O_5. Closed cycle tests were subsequently carried out based on the optimum parameters of condition tests.

2 Experimental

2.1 Material and characterization

The stone coal in our experiments was obtained from Shanxi Province, China. Chemical composition was characterized by X-ray fluorescence spectrometry (XRF) using a Philips spectrometer. Phase composition and microstructure of the stone coal were investigated by powder X-ray diffraction (XRD, D8-ADVANCE, Bruker Co. Ltd., Germany), chemical phase analysis and scanning electron microscopy with an energy dispersive spectrometer (SEM−EDS, JSM−6490LV, JEOL Co. Japan).

Moreover, particle size distribution of the stone coal (<3 mm) was measured using a wet sieve analysis. The sample was subjected to successive sieving starting from large to small sieves with 1.5, 0.6, 0.154, 0.074, 0.05, and 0.038 mm. The stone coal was gently sieved into a series of fractions (<0.038, 0.038−0.05, 0.05−0.074, 0.074−0.154, 0.154−0.60, 0.6−1.5 and >1.5 mm). Each fraction was filtered, dried, weighed, and analyzed by XRF. The analytical grade reagents used in this study and their abbreviations are listed in Table 1.

Table 1 Reagents used in flotation test

Reagent	Function
Sulfuric acid	pH regulator
Oleic acid	Ca-minerals collector
Sodium silicate	Dispersant
Sodium fluosilicate(SFF)	Depressant
Melamine(EA)	V-minerals collector
Dodecyl amine(DDA)	V-minerals collector
Octadecylamine(DC)	V-minerals collector
Terpenic oil	Frother

2.2 Flotation

After being crushed to less than 2 mm and deslimed, the ore (500 g) was ground using a XMQ ball mill. Flotation was carried out in a 1.5 L flotation cell at an agitation speed of 2045 r/min, in which pH regulator, depressant and collector were added at a pulp density of 30% (mass fraction). Two-stage flotation experiments were carried out using combination of reverse and direct flotation. Calcite was primarily floated to avoid negative effect on the subsequent flotation of vanadium-bearing minerals, and then the tailing from calcite flotation was further subjected to direct flotation. The detailed process flowsheet is given in Fig. 1. Flotation products such as slime, concentrate and tailing were filtered, dried, weighed, and analyzed by XRF.

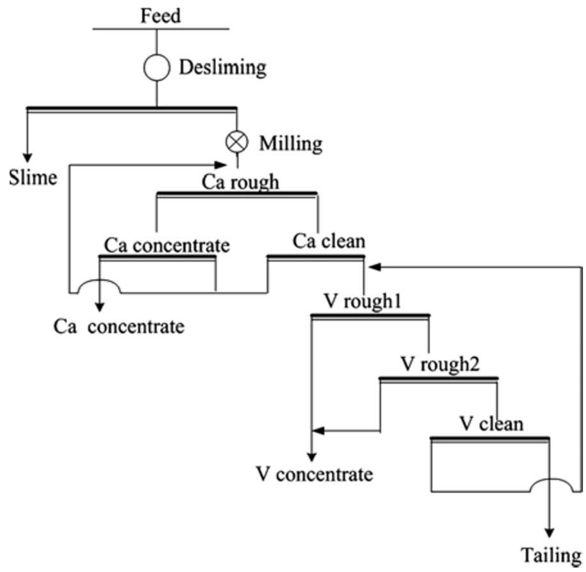

Fig. 1 Process flowsheet of stone coal

3 Results and discussion

3.1 Mineralogical characteristics

The mineralogical assembly consisted of quartz as the ore mineral and calcite, barite, dolomite, feldspar, muscovite, montmorillonite as the accessory minerals based on the XRD results. The chemical composition of stone coal is listed in Table 2, which shows relatively low vanadium (V_2O_5) content of 0.68% and high calcium oxide content of 2.98%. Vanadium phase analysis was carried out according to the sequential extraction procedures. The results are shown in Table 3. 68.80% of vanadium was found to exist in muscovite, while the others were in iron−aluminium oxides.

Table 2 Chemical composition of stone coal

$w(V_2O_5)$/%	w(Fe)/%	w(CaO)/%	w(S)/%	$w(SiO_2)$/%
0.68	1.14	2.98	0.56	75.72

$w(P_2O_5)$/%	w(BaO)/%	$w(Al_2O_3)$/%	Ignition loss/%
0.46	2.63	3.77	4.92

Microstructure and phase structure of the stone coal (<74 μm) were analyzed by SEM (Fig. 2) and EDS (Table 4). Vanadium in the stone coal existed in the form of muscovite and iron−aluminium oxides, which was