



Vanadium recovery from stone coal through roasting and flotation

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Abstract: A new process for vanadium recovery from stone coal by roasting–flotation was investigated based on the mineralogy. The process comprised four key steps: decarburization, preferential grinding, desliming and flotation. In the decarburization stage, roasting at 550 °C effectively avoided the negative effect of the carbonaceous materials in raw ore and generation of free CaO from calcite decomposition during roasting. Through preferential grinding, the high acid-consuming minerals were enriched in the middle fractions, while mica was enriched in the fine and coarse fractions. Through flotation, the final concentrate can be obtained with V₂O₅ grade of 1.07% and recovery of 83.30%. Moreover, the vanadium leaching rate of the final concentrate increased 13.53% compared to that of the feed. The results reveal that the decarburization by roasting at 550 °C is feasible and has little negative impact on mica flotation, and vanadium recovery from stone coal is conducive to reducing handling quantity, acid consumption and production cost.

Key words: vanadium-bearing stone coal; roasting decarburization; mineralogy; preferential grinding; flotation

1 Introduction

In China, stone coal is a specific vanadium-bearing resource. The gross reserve of vanadium in terms of V₂O₅ in stone coal is 118 million tons, which accounts for over 87% of the domestic reserve of vanadium [1,2]. Hence, various vanadium extraction techniques from stone coal are investigated. Generally, these techniques involve long processes like roasting, acid leaching, ion purification, precipitation and calcination [3–6]. However, due to the low vanadium grade, complex chemical composition and various occurrences of vanadium of stone coal, vanadium extraction from stone coal is commonly confronted with the problems of enormous ore handling quantity, high acid consumption and high production cost [7].

For the sake of relieving these problems, pre-concentration of vanadium in stone coal before leaching is an effective method. ZHAO et al [8] took gravity separation to pre-concentrate vanadium from stone coal. Although the V₂O₅ loss was low in gravity

separation stage, the yield of final tailings was not high and the separation for the acid-consuming minerals, especially calcite, was not satisfactory. In order to obtain satisfactory separation results, WANG et al [9] chose flotation for beneficiation due to its high handling capacity and high selectivity, and obtained satisfactory flotation results as the stone coal was weathered. Under that condition, little carbon existed in the raw ore and the chemical composition and mineral texture were comparatively simple, which was easy to obtain relatively higher flotation efficiency and better separation results. However, most stone coal in China exists as primary ore, where the carbon content usually ranges from 8% to 12%, and the chemical composition and mineral texture are rather complex. The carbonaceous materials are disseminated among various minerals such as oxides, carbonates, silicates and sulfides, which closely coexist as fine-grained particles. Due to the coating of carbonaceous materials on the surfaces of mineral particles, the floatability differences among different minerals decrease significantly and flotation separation effect is not satisfactory. Even for the severely

metamorphic stone coal that carbon mainly exists as graphite, the negative effect of carbon on flotation of stone coal still resulted in a long process, huge reagent consumption and production cost, so technique of decarbonization by roasting before flotation was developed [10–13]. Usually the roasting temperature was set over 650 °C so as to remove the carbon quickly without considering the negative effect on flotation. Our previous research [14] found that, with the increase of roasting temperature, reactions like the oxidation of pyrite, combustion of carbon and calcite decomposition occurred successively in vanadium-bearing stone coal. At 600–700 °C, carbon and pyrite disappeared, and octahedral structure in mica was not damaged, while the remaining free CaO after sulfur-fixation of calcite made the pH value and concentration of Ca^{2+} increase in flotation pulp. Under that condition, it is not conducive to flotation because the fine particles coagulated in the non-selective state, and much fatty acid was consumed, as well as the quartz was activated.

Given that the negative effect of carbonaceous materials and free CaO on flotation should be balanced, reasonable roasting temperature is important for optimizing the flotation process. The focus of this study is to determine a simple and reasonable flotation process for typical primary vanadium-bearing stone coal at relatively low roasting temperature (550 °C). The flotation results are interpreted within the perspective of the effect of the mineralogy on the flotation process and vice versa.

2 Experimental

2.1 Materials and reagents

The vanadium-bearing stone coal used in this study was obtained from Hubei Province, China. The raw ore was firstly crushed to below 2 mm in size with two-stage jaw crusher and one-stage roll crusher. The crushed ore was blended and then split into 200 g samples for mineralogy and flotation tests. The analytical grade reagents used in this study and their abbreviations are listed in Table 1.

Table 1 Reagents used in flotation

Reagent	Function
Sulfuric acid	pH regulator
Fatty acid (YS)	Reverse flotation collector
Sodium fluosilicate	Depressant
Acidic water glass	Depressant
Dodecyl amine (DDA)	Direct flotation collector

2.2 Procedure

The crushed raw ore was firstly decarbonized in a SXZ-10-B muffle furnace at 550 °C for 90 min and then

wet-ground in a XMB-70 laboratory rod mill at 50% solids, until a particle size distribution of 84.62% passing 74 μm was achieved. The rod mill product was subjected to desliming by free settling and then the flotation tests was conducted in a 0.5 L flotation cell at an agitation speed of 1992 r/min, in which pH regulator, depressant and collector were added. Two-stage flotation experiments were carried out containing reverse and direct flotation. The detailed conditions and process flowsheet are given in Fig. 1. The flotation products contained slime, V concentrate, tailings 1 and 2. The slime and V concentrate are merged into final concentrate, and the rest are rejected as final tailing. The vanadium of preferential grinding product and final concentrate were leached by 5% CaF_2 and 15% (volume fraction) H_2SO_4 at 95 °C for 6 h.

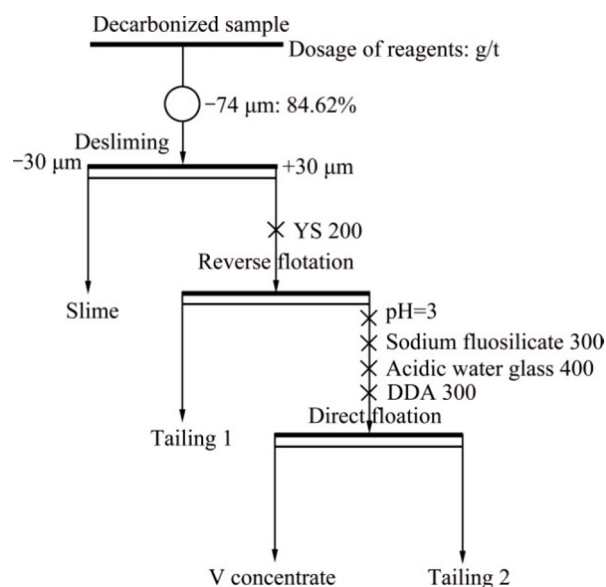


Fig. 1 Flotation conditions and process flowsheet of decarbonized sample

2.3 Test methods

The vanadium grade was measured by Test Methods of Vanadium in Coal Standard (GB/T 19226–2003). X-ray diffraction (XRD) analysis was conducted by D/Max-III A X-ray diffractometer with Cu K_α radiation, voltage 40 kV, current 30 mA and at the scanning rate of 15 ($^\circ$)/min from 5° to 70°. The phases were identified by comparison of the peak positions and d values with data published by the international centre for diffraction data (ICDD). The chemical analysis was performed with the Xios advanced X-ray fluorescence (XRF) analyzer. Detailed mineralogy of the raw ore and decarbonized samples was investigated using Leica DMLP polarization microscope and quantitative evaluation of minerals by scanning electron microscopy (QEMSCAN). Valence distribution was measured on ZDJ-4A automatic potentiometric titrimeter by ammonium ferrous sulfate titration method [15]. Vanadium chemical phase analysis

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