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Contribution of ammonium ions to sulfidation-flotation of smithsonite

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ABSTRACT

Sulfidation-flotation is commonly performed to recover base metal oxide ores. Sulfidation could become the key process in recovering zinc from zinc oxide ores. We investigated the common zinc oxide mineral. An ammonium salt was used to enhance sulfidation-flotation of smithsonite. The effect of ammonium ions on sulfidation and flotation of smithsonite was investigated by microflotation, inductively coupled plasma analysis, scanning electron microscopy, and X-ray photoelectron spectroscopy. Addition of the ammonium salt facilitates flotation of smithsonite and improves the flotation recovery by ~25%. Solubility tests under different conditions indicate that the surface solubility of smithsonite in the presence of ammonium ions significantly decreases. Energy-dispersive X-ray spectroscopy based semi-quantitative analysis shows that the concentration of sulfur on the ammonium enhanced sulfurized smithsonite surface is 8.55%, whereas that on the sulfurized smithsonite surface is 2%. XPS analysis reveals that smithsonite is more easily sulfurized by sodium sulfide in the presence of ammonium ions. Therefore, ammonium ion addition can be used to enhance sulfidation of smithsonite and improve the flotation performance.

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1. Introduction

Sulfidation-flotation is commonly performed to recover base metals from oxide ores. The process involves sulfidation of the oxide by sulfur dioxide, sulfide ions, or metabisulfite ions, which allows known sulfide collectors to be used to recover target base metal oxides. Natural sulfide ores are primary zinc sources. Because such reserves are becoming depleted and the demand for zinc is continually increasing, exploitation and utilization of zinc oxide ore bodies has become inevitable. However, flotation of zinc oxide ores is more difficult than that of sulfide minerals [23]. Smithsonite ($ZnCO_3$) is a common zinc oxide mineral. Smithsonite occurs as a secondary mineral in the weathered zone or oxidation zone of zinc-bearing ore bodies. Smithsonite is a variable colored trigonal mineral that is rarely found in the crystalline form, but it is typically part of botryoidal masses. Smithsonite is hexagonal scalenohedral with point group $3\bar{2}m$. Carbonates with intermediate sized divalent cations normally crystallize in calcite structures [6,24,25].

Smithsonite flotation has been extensively investigated. A range of collectors to recover zinc oxide after sulfidation have been investigated [3–5,13,17,21]. These include:

- (I) Cationic collectors, such as amines ([4,11], Hosseini and Forssberg, 2006; [13,15,20]);
- (II) Sulfhydryl collectors, such as xanthate, after metal ion activation [8,10] Hossen and Forssberg, 2006; [22];
- (III) Mixed anionic/cationic collectors, such as xanthates with amines [9,12];
- (IV) Fatty acids [10,15,24];
- (V) Other sulfhydryl collectors such as mercaptans [11].

Although they have been extensively investigated, these processes are not widely performed in industrial applications. A major problem of sulfidation-flotation is the existence of slimes, which reduce the sulfidation efficiency. Although slimes are typically removed before sulfidation, they will reduce the overall base metal recovery [14,19]. Because xanthate collectors are less affected by slimes than amine collectors, the former were used in this study. The main aim of the study was to optimize surface sulfidation of smithsonite, which is an important factor for improving sulfidation-flotation of smithsonite.

In this study, we investigated surface sulfidation of smithsonite in the presence of ammonium ions. Ammonium salts are strong electrolytes and will dissociate in water. The dissolved ammonium ions will dissolve smithsonite and other oxidized ores to form $[M(NH_3)_n]^{2+}$ ($n = 1-4$) [1,16,26,27]. When these sulfurized compounds form MS, where M is a metal element and S is sulfur, more active sites are available for collector binding than for sulfurized

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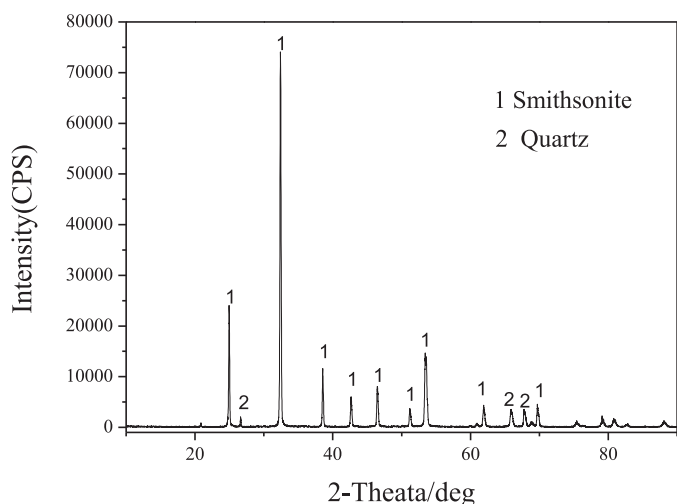


Fig. 1. X-ray diffraction pattern of the smithsonite sample.

Table 1
Chemical composition of smithsonite sample.

Composition	Zn	Fe	CaO	Al ₂ O ₃	Pb	Cd
Content (%)	49.46	0.4	0.59	0.50	0.67	0.46

metal oxides, which should improve the flotation performance. We investigated the contribution of ammonium ions to smithsonite sulfidation and flotation by microflotation experiments, inductively coupled plasma (ICP) analysis, scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDS), and X-ray photoelectron spectroscopy (XPS).

2. Materials and methods

2.1. Materials and reagents

The smithsonite mineral samples were obtained from Yunnan, China. The ore sample was crushed in an agate mortar, and the coarse fraction ($-74\ \mu\text{m} + 19\ \mu\text{m}$) was used in the microflotation experiments. Mineralogical analysis of the smithsonite mineral sample was conducted with a Rigaku D/max 2550VB + 18 kW powder diffractometer with a Cu/K α X-ray source at 40 kV and 40 mA. The results of the X-ray analyses are shown in Fig. 1, which indicate that the ore sample consists of smithsonite and quartz. The chemical composition of smithsonite is shown in Table 1.

Ammonium nitrate was selected as the ammonium ion source. Na₂S·9H₂O was added as the sulfurizing agent, CuSO₄·5H₂O was used as the activator, and commercial grade sodium isoamylxanthate (NaIX) was used as the collector. All of the reagents, except for NaIX, were of analytical grade. Deionized water was used throughout.

2.2. Microflotation experiments

The microflotation experiments were performed in a mechanically agitated 50 mL flotation cell. The mineral suspension was prepared by adding 5.0 g of pure smithsonite to 40 mL of water. Solutions with various ammonium nitrate concentrations were added to mineral suspension samples (reaction time 5 min). Freshly prepared Na₂S·9H₂O solution was then added to sulfidize the smithsonite surface (reaction time 5 min). The pulp pH was adjusted to 10.5 and CuSO₄·5H₂O solution was added into the pulp solution (reaction time 2 min). The pulp was conditioned with NaIX for 3 min and floated for 5 min. After the flotation tests, the

concentrate and tailings were filtered and dried before weighing. The flotation recovery was calculated based on the solid weight distribution between the two products. All of the microflotation experiments were performed in duplicate.

2.3. Solubility tests of smithsonite under different conditions by ICP analysis

Pure smithsonite with particle sizes of approximately 0.5–1 cm was cleaned with deionized water. Samples were dried and weighed and placed in two similar ball milling jars. The closed ball milling jars were installed on an impact ball milling instrument (MM400, Retsch, Germany). The vibration frequency and grinding time were set to 900 min⁻¹ and 8 min, respectively. Smithsonite samples (2 g) were then added to solutions to obtain a 5% pulp concentration. The solutions were mechanically stirred in a beaker for 120 min with samples periodically taken for kinetic monitoring. Four solubility tests were performed: smithsonite in deionized water, ammonium nitrate solution, sodium sulfide solution, and a mixture of ammonium nitrate solution and sodium sulfide solution. The ammonium nitrate and sodium sulfide concentrations were 1×10^{-3} M. Each test was performed in a neutral environment. After dissolution, the supernatant was analyzed by ICP after centrifuging (TL-4.7 W, SCI, China) for solid–liquid separation.

2.4. SEM-EDS analysis

Before and after sulfidation, the surface morphology and composition of the smithsonite grains were examined by SEM-EDS with a Jeol JSM-6360 instrument at 20 kV.

2.5. XPS analysis

The solid sample with ammonium and sulfur ions was examined by XPS, which was performed with a Kratos Amicus apparatus (England) with a conical anode Mg K α source. The operating parameters were operating voltage 12 kV, power 180 W, and optimal energy resolution <0.5 eV. The analysis chamber vacuum degree was 1×10^{-6} Pa and the pass energy was 75 eV.

3. Results and discussion

3.1. Microflotation studies

The flotation recovery of smithsonite as a function of the NaIX concentration in the absence and presence of different ammonium ions concentrations with 1×10^{-3} M Na₂S·9H₂O as a sulfurizing agent and 1×10^{-3} M CuSO₄·5H₂O as an activator is shown in Fig. 2. The flotation recovery of smithsonite with 1×10^{-3} M Na₂S·9H₂O as a sulfurizing agent and addition of 1×10^{-4} M NaIX is 17.39%, whereas it is 54.41% with addition of 5×10^{-3} M NaIX. Thus, with addition of ammonium ions, the flotation recovery of smithsonite significantly increases. Under the abovementioned experimental conditions, the flotation recovery of smithsonite increases with increasing ammonium ion concentration. The flotation recovery of smithsonite increases from 67.95% to 81.91% when the ammonium ion concentration is increased from 1×10^{-4} M to 1×10^{-3} M with 5×10^{-3} M NaIX as the collector. These results show that ammonium ion addition improves sulfidation of smithsonite and significantly facilitates flotation of smithsonite, which results in better flotation performance. These samples are referred to as “enhanced sulfidation”.

3.2. Smithsonite solubility tests under different conditions

The experimental results of the solubility test are shown in Table 2. The solubility of smithsonite in deionized water follows

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