

Promoting sulfidation of smithsonite by zinc sulfide species increase with addition of ammonium chloride and its effect on flotation performance



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ABSTRACT

The sulfidation-flotation method is commonly used for the treatment of zinc oxide minerals. However, few studies have been conducted on the promoting sulfidation-flotation behavior and mechanism of smithsonite. In this paper, we investigate the promoting sulfidation of smithsonite by zinc sulfide species increase with addition of ammonium chloride (NH₄Cl) and its effect on flotation performance. The micro-flotation experiment results concluded that the addition of the ammonium chloride improved smithsonite flotation recovery by ~17%. Zinc ion adsorption experiments and Visual MINTEQ model analysis showed that more zinc sulfide species were produced in the presence of NH₄Cl. Zeta potential analysis indicated that additional sulfide ion species and isoamyl xanthate ions (IX⁻) from the pulp solution adhered to the smithsonite surface as NH₄Cl was added, and the SEM-EDS study and the contact angle results further confirmed the positive influence of NH₄Cl on the smithsonite sulfidation process. The TOF-SIMS results provided strong evidence for an increase in zinc sulfide species on the smithsonite surface and an increase in the sodium isoamyl xanthate (NaIX) molecular adsorption on the surface of sulfidized smithsonite with the addition of NH₄Cl. The thickness of zinc sulfide species formed after promoting sulfidation was ~29.20 nm, and the thickness of xanthogenate species formed was a few nanometers. The promotion in sulfidation may be due to the combined effect of the improvement of sphalerite precipitation adsorption and the ion-exchange reaction between the sulfide ions and the carbonate ions on the surface of the smithsonite.

1. Introduction

Zinc is an important base metal that is widely used in the production of alloys and in galvanization, which protects steel structures. It is also used as an additive in rubber and paints (Espiriari et al., 2006; Irannajad et al., 2009; Liu et al., 2016). At present, zinc is mainly concentrated from zinc sulfide minerals using conventional flotation techniques. However, with the depletion of zinc sulfide ores and the increasing restrictions on sulfur emissions, the effectiveness of beneficiating zinc from zinc oxide ores has assumed greater importance in recent years. This drives the urgent need to more effectively process these ores in order to produce a marketable product (Deng et al., 2017; Shi et al., 2012; Mehdilo et al., 2014).

Smithsonite (ZnCO₃), known as native zinc carbonate, is an important source of zinc. In China, smithsonite ores are relatively abundant and are mainly found in southwestern and northwestern China, such as in the Yunnan, Sichuan, Guangxi, and Gansu provinces (Ling et al., 2011; Tang et al., 2011). At present, the most common flotation technique commercially used for the treatment of zinc oxide ores is

sulfidized with Na₂S followed by treatment with a conventional cationic collector, an anionic collector, or a mixed anionic/cationic collector (Hosseini and Forssberg, 2007; Ejtemaei et al., 2011; Mehdilo et al., 2012; Sun et al., 2012; Mehdilo et al., 2013; Zhang et al., 2017). After sulfidation of the zinc oxide ore, the mineral's surface becomes less hydrophilic due to the presence of the chemisorbed sulfide ion, and it floats more efficiently. Under these conditions, collector consumption may also be significantly reduced (Gush, 2005; Pereira and Peres, 2005; Hosseini and Forssberg, 2006). However, the main disadvantage of processing with sulfidation-amine flotation lies in the severe influence of slime. In addition, the amount of sulfidation reagent and the pH of the pulp must be carefully controlled in the sulfidation-amine flotation. Thus, the main flotation characteristics of cationic collectors are lower selectivity and higher reagent consumption (Fa et al., 2005; Khaleghi et al., 2016).

Sulfidation-xanthates flotation is an effective method for treating smithsonite ores. In order to increase the recovery of smithsonite, advanced xanthenes, such as hexyl and amyl xanthates are usually employed as the collectors, and the bivalent copper ion is used as the

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activator. Previous research indicates that excess sodium sulfide acts as a depressant for the flotation of oxidized ore because the adsorption of divalent sulfide ions on the surface of the oxidized ore increases the negative charge, which prevents the adsorption of the collectors (Feng et al., 2016; Feng and Wen, 2017). Furthermore, in the sulfidation process, it is difficult to attain a low Na_2S concentration that adequately sulfurizes the mineral surface, while also allowing for sulfide collection. Hence, investigation into the promoting sulfidation of smithsonite and its effect on flotation performance with use of xanthate is very important for industrial applications of the sulfidation-flotation technique.

Many studies have been conducted on the behavior and mechanism of smithsonite sulfidation. The sulfidation mechanism of smithsonite includes (Newell and Bradshaw, 2007; Newell et al., 2007; Ejtemaei et al., 2014): (I) an ion-exchange reaction between sulfide carbonate ions on the smithsonite's surface, and (II) metal sulfide precipitation adsorption, i.e., sulfide ions react with the metal ion component, forming metal sulfide and adsorbing onto the smithsonite's surface. After sulfidation, a zinc sulfide species is formed on the smithsonite's surface, and the surface becomes less hydrophilic, resulting in more efficient flotation. Mehdi et al. (2013) concluded that the smithsonite surfaces were completely converted into ZnS at high Na_2S concentrations, and the mineral surfaces were coated with a dense sulfide film. Kashani and Rashchi (2008) demonstrated that increasing amounts of zinc sulfide formed on the smithsonite's surface with the addition of Na_2S , and that the number of zinc sulfide layers was limited. Wu et al. (2015) suggested that a zinc sulfide film was formed on the smithsonite surfaces after treatment with Na_2S . Although numerous studies on smithsonite flotation were carried out, most of these studies focused on the ion-exchange reaction mechanism, collector performance, and sulfidation conditions (Hosseini and Forssberg, 2006; Irannajad et al., 2009; Ejtemaei et al., 2011; Ejtemaei et al., 2014; Zhang et al., 2017). In addition, minimal information is available regarding the promoting sulfidation behavior of smithsonite and its effect on flotation performance with the use of xanthate based on the above-mentioned two sulfidation mechanisms.

In this work, NH_4Cl was employed to promote the sulfidation-xanthate flotation of smithsonite. Ammonium salts are strong electrolytes and will dissociate in water. The dissolved ammonium ions will dissolve smithsonite to form $[\text{Zn}(\text{NH}_3)_i]^{2+}$ ($i = 1, 2, 3, 4$) (Bingol et al., 2005; Ju et al., 2005; Yin et al., 2010). When these sulfurized compounds form ZnS, more active sites are available for collector binding than for sulfurized zinc oxides, which should improve the flotation performance. Motivated by these observations, micro-flotation experiments, surface adsorption tests, a Visual MINTEQ model, zeta-potential determinations, scanning electron microscope (SEM) analyses, and contact angle measurements were conducted to investigate the influence of NH_4Cl on the sulfidation-flotation of smithsonite with isoamyl xanthate. In addition, Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) was used to characterize the surfaces of the smithsonite and provided evidence for an increase in the formation of zinc sulfide species and adsorption of the isoamyl xanthate molecular. Our results provide a theoretical basis for the promoting sulfidation flotation of zinc oxide ores.

2. Materials and methods

2.1. Materials

The smithsonite samples in the present study were firstly obtained from a high grade of natural smithsonite ores in Yunnan Province, China. After Hand-picked carefully, the purity of the smithsonite particles to be $> 95\%$ was used in all the experiments. The ore samples were crushed, ground, and sieved using standard test sieves to obtain the desired particle size fractions (38–74 μm). X-ray diffraction (XRD) pattern of sample was shown in Fig. 1, and the smithsonite crystal was mainly observed. The diffraction peak was well consistent with the data

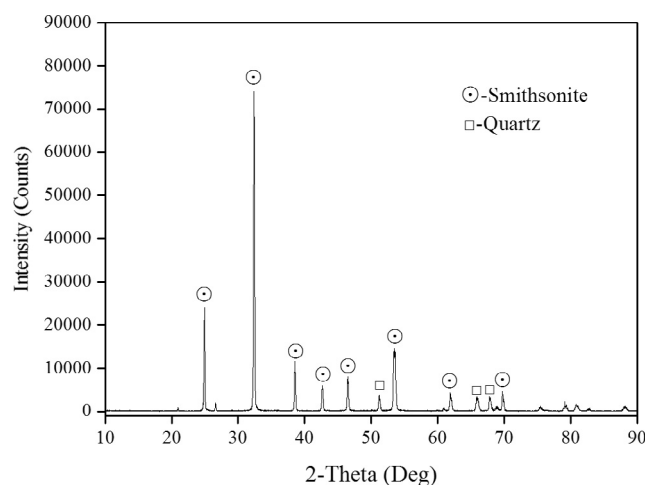


Fig. 1. X-ray diffraction pattern of the original smithsonite ore sample used in the experiments.

Table 1

Chemical composition of the original smithsonite.

Element	Zn _T	Fe	CaO	Al ₂ O ₃	Pb	Cd	SiO ₂
Content (%)	49.65	0.43	0.61	0.48	0.37	0.36	1.44

presented in the JCPDS card (No. 83-1765) (Feng and Wen, 2017). Chemical composition of the original smithsonite was detected by chemical titration analysis, which is reported in Table 1. The results showed that the test samples contained 49.69% Zn and low levels of mineral impurities. Analytical-grade $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ and $\text{CuSO}_4\cdot 5\text{H}_2\text{O}$ were employed as the sulfidizing agent and activator, respectively. Sodium isoamyl xanthate (NaIX) was used as the collector. Sodium hydroxide (NaOH) and ammonium chloride (NH_4Cl) with $> 98\%$ purity, were obtained from different chemical companies in China. The used in all the experiments pure deionized water, with a resistivity of 18 MU, was obtained from a Milli-Q50 system (Billerica, MA, USA).

2.2. Micro-flotation experiments

Micro-flotation experiments were conducted at room temperature in a mechanical agitation flotation machine. NaOH solution was added to the pulp to adjust the pH, and the pulp was conditioned for 5 min. A specific dosage of NH_4Cl solution was prepared, the stock solutions were poured into the pulp suspension, and the pulp was conditioned for 5 min. A specific dosage of $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ was added to the pulp, and the pulp was conditioned for 8 min. Subsequently, 1.5×10^{-3} mol/L $\text{CuSO}_4\cdot 5\text{H}_2\text{O}$ stock solution was added and allowed to activate for 3 min. Finally, the sodium isoamyl xanthate was added to the pulp and collection was conducted allowed to proceed for 5 min. At the end of the experiment, the froth product was filtered, dried, and weighed. The amount of smithsonite recovery was used to evaluate the floatability of the samples, and the flotation recovery of smithsonite was calculated by following equation:

$$R = \frac{m_1}{m_0} \times 100\% \quad (1)$$

where R is the flotation recovery of smithsonite; m_1 is the weight of froth product (g), and m_0 is the weight of smithsonite raw ore (g).

2.3. Experiments for zinc-ion adsorption

$\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ and NH_4Cl were used to create a solution with S^{2-} and NH_4^+ ion concentrations of 1.5×10^{-3} mol/L. A fresh smithsonite

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