

Adsorption of Fe(III) on smithsonite surfaces and implications for flotation



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ABSTRACT

Recently, there has been interest in the effects of ionic species on the flotation performance of minerals in liquid phase. Metal ions that are commonly present in flotation pulp can cause non-selective activation of target minerals and gangue particles during flotation. We have investigated the influence of Fe(III) adsorption on smithsonite surfaces using X-ray diffraction, X-ray photoelectron spectroscopy (XPS), zeta potential measurements, scanning electron microscopy, and micro-flotation tests. The results indicate that Fe(III) can adsorb onto the smithsonite surface, resulting in a series of changes to the surface properties. XPS results show that Fe(III) adsorption can change the atomic composition of the smithsonite surface, along with its chemical state. The zeta potential of the smithsonite became more positive and its pH shifted from 7.6 to 9.2 in the presence of 1×10^{-4} mol/L Fe(III). Micro-flotation tests revealed that iron ions have a strong inhibitory effect on smithsonite. The recovery of smithsonite decreased sharply as the Fe(III) concentration increased. When the concentration reached 5×10^{-4} mol/L, the recovery was only 21.35%. The Fe(III)-driven smithsonite inhibition mechanism may be attributed to strong oxidation and hydrolysis.

1. Introduction

As natural sulfide ore resource exploitation has continued, sphalerite supplies have gradually become depleted. In order to meet future zinc market demand, several studies have been devoted to the mineral processing of smithsonite, which is a typical zinc oxide mineral [1]. Froth flotation is the most widely used and effective method of recovering smithsonite. However, the mineral is characterized by higher hydrophilicity than its sulfide counterparts, and has physicochemical and surface chemistries similar to those of other materials. These properties render it more difficult to separate from gangues via flotation [2–5]. In addition, the flotation pulp contains metal ions. In most cases, these ions are referred to as “unavoidable ions” such as Cu(II), Pb(II), Zn(II), and Fe(III). They are released into the liquid phase along with broken fluid inclusions and minerals dissolved during mineral liberation processing [6–8]. In practical mineral flotation, the dissolved metal ions inevitably lead to changes in solution chemistry and affect mineral flotation [9,10].

Because of its importance to environmental and mineral engineering, the study of metal ion adsorption on mineral surfaces has garnered extensive continuing interest [11–14]. A few groups have studied the effects of ionic species on the flotation performance of smithsonite in the liquid phase. Shi et al. [15] investigated the solubilities of calcite and smithsonite mixtures and established correlations

between solution chemistry and flotation behaviour. The results showed that the surface conversion of calcite into zinc species plays an important role in the separation of smithsonite from calcite. Ou et al. [16] reported that smithsonite was observably depressed by Fe^{2+} and Fe^{3+} under alkaline conditions, in the presence of sodium oleate. They attributed this inhibition to the adsorption of Fe^{2+} and Fe^{3+} hydroxides on the smithsonite surface, which hindered the transport of collectors towards the surface. Moreover, Zhang et al. [17] investigated the effect of sodium silicate on the separation of smithsonite and calcite in the presence of Ca^{2+} via flotation testing. The results indicated that Ca^{2+} can activate the flotation performance of quartz remarkably within the 9.5–11.0 pH range. Meanwhile, the inhibition of sodium silicate on smithsonite was strengthened. When the reagent addition order was changed by preferentially setting sodium silicate into the flotation pulp, adsorption of Ca^{2+} onto the mineral surface was effectively hindered. Jiang et al. [18] investigated the effects of Cu^{2+} and Pb^{2+} on the floatability of smithsonite after sulfidation. They indicated that Cu^{2+} and Pb^{2+} could strengthen the smithsonite sulfidation process via a mechanism similar to that of the Cu^{2+} -sphalerite system.

Despite these studies on the effects of ions on smithsonite flotation, there are still some knowledge limitations with respect to interaction mechanisms that involve metal ions, reagents, and the smithsonite surface. It is generally known that adsorption of ions onto the mineral surface occurs before flotation. Thus, when the effect of ions on mineral

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flotation is investigated, it is necessary to consider that surface properties, such as the surface potential, atomic concentrations, and contact angle may have changed before reagent addition.

In this study, smithsonite was selected as a model adsorbent, and iron ions were chosen as the metal-ion adsorbates because of their presence in practical flotation pulps. X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), zeta potential measurements, micro-flotation tests, and surface adsorption measurements were performed to investigate interactions between the iron ions and smithsonite. A detailed study of the changes in surface properties after ion adsorption and the resulting influence on smithsonite flotation was performed.

2. Experimental

2.1. Materials and reagents

Hand-picked, high-purity smithsonite was obtained from the Lanping mine in China. The samples were washed with distilled water and dried. The ore samples were then manually processed using an agate mortar, sieved, and classified to obtain the desired particle size fractions. Particles 45–75 μm in size were used for metal ion adsorption studies and micro-flotation tests. Meanwhile, finer particles ($< 45 \mu\text{m}$) were ground to less than 5 μm for use in zeta potential measurements. XRD (D/Max 2200, Rigaku, Japan) pattern and chemical analysis data are shown in Fig. 1 and Table 1, respectively. The results indicate the purity of the smithsonite particles to be $> 95\%$ with only minor impurities present in the sample.

Analytical grade dodecylamine (DDA), hydrochloric acid, sodium hydroxide, and sodium sulfide were purchased from Sinopharm Chemical Reagent Co. Ltd., Shanghai, China. Finely powdered, analytical grade iron chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) was supplied by Nanjing union silicon chemical Co. Ltd for use as the source of Fe(III).

2.2. Methods

2.2.1. Adsorption of Fe(III) on the smithsonite surface

Metal ions were adsorbed onto the smithsonite surface in a vial containing 100 mL of the Fe (III) solution that was under magnetic stirring for 20 min. The Fe(III) concentration, as determined from the ion concentration in the practical flotation pulp of the oxide zinc ore, was approximately 1×10^{-4} mol/L. After solution preparation, the Fe (III) ion concentration was determined accurately via inductively coupled spectroscopy mass spectrometry (ICP-MS, XSERIES 2, Thermo Fisher Scientific, Germany). Typically, 5 g of the smithsonite sample was added to a vial that already contained 3×10^{-4} mol KNO_3 as the

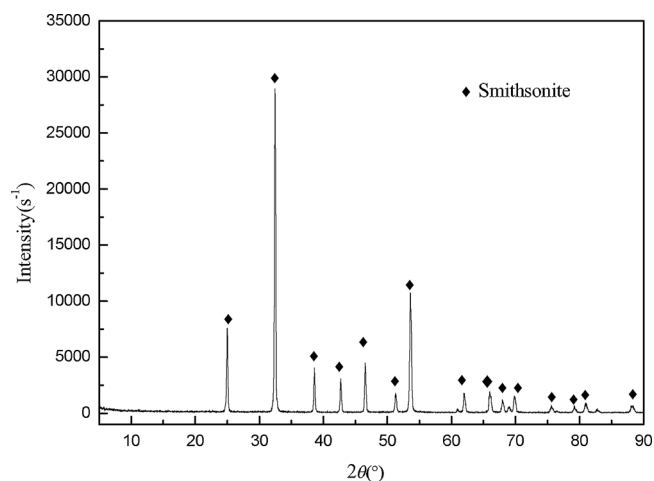


Fig. 1. XRD pattern of a pure smithsonite sample.

Table 1
Chemical analysis of a pure smithsonite sample (wt%).

Composition	Zn	Al ₂ O ₃	SiO ₂	TFe	Mn	CaO	MgO	Others	Total
Content	50.56	0.01	0.02	0.04	0.01	0.74	0.05	48.57	100.00

background electrolyte. When adsorption was complete, a centrifuge was used to separate the solute from the liquid phase. The separated liquid was stored in a closed vial and analysed via ICP-FE to determine its Fe(III) concentration. The separated solid was collected, air-dried, and used for XRD, SEM, and XPS analyses.

2.2.2. Scanning electron microscopy

Before and after the treatment with iron ions (1×10^{-4} mol/L), the surface morphology of the smithsonite was analysed via SEM (Jeol JSM-6360) at 20 kV. The sample preparation methods were the same as for the adsorption tests in Section 2.2.1.

2.2.3. X-ray photoelectron spectroscopy

XPS measurements were conducted for three types of smithsonite samples. Two of these samples were smithsonite samples treated with or without iron ions (1×10^{-4} mol/L). The third sample was produced as follows: after 20 min of Fe(III) adsorption, Na_2S (5×10^{-3} mol/L) was added into the solution to sulfidize the smithsonite for 5 min, and then the solute was separated from the liquid phase by centrifugation. Thereafter, the smithsonite samples were rinsed repeatedly before XPS measurements. Therefore, it is reasonable to assume that any loosely adsorbed ions were washed away from the mineral surface. The smithsonite sample XPS measurements were performed using a Kratos Amicus (England), with a conical anode Mg K α -ray source. The operating parameters were as follows: operating voltage = 12 kV, power = 180 W, and optimal energy resolution < 0.5 eV. The analysis chamber vacuum was 1×10^{-6} Pa, and the pass energy was 65 eV.

2.2.4. Zeta potential measurements

The zeta potential of smithsonite was measured using a Brookhaven Zetaplus zeta potential analyser (USA). Fifty mg of the mineral sample was added to 30 mL of an aqueous solution with the required iron ion concentration and pH. Then, the suspension was stirred for 20 min after adding 3×10^{-4} mol of KNO_3 as the background electrolyte. The pH values were adjusted by adding dilute HCl or NaOH solutions. The agitated suspension was sampled to record the zeta potential. The results presented are the average of three independent measurements with a typical variation of ± 2 mV.

2.2.5. Flotation method

Micro-flotation experiments were conducted in a mechanical agitation flotation machine. In each test, 2 g of the mineral sample was used in a 30 mL Fe(III) solution of the desired concentration. After 20 min of equilibration, a freshly prepared $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ solution of the required concentration was added for 5 min to sulfidize the smithsonite surface, and then the collector of DDA (3×10^{-4} mol/L) were added with the conditioning time of 2 min. The concentration of S^{2-} in the flotation pulp was 5×10^{-3} mol/L. Before the flotation tests, the pH of the suspension was adjusted by adding dilute HCl or NaOH solutions, after which flotation was performed for a period of 6 min. The products and tailings were weighed separately after filtration and drying, and finally, the recovery was calculated.

3. Results and discussion

3.1. Adsorption phenomena

Fig. 2 provides macroscopic evidence that the surface colour of the smithsonite sample changes from white to yellow after treatment with

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