



Technical note

Reductive leaching of indium-bearing zinc residue in sulfuric acid using sphalerite concentrate as reductant



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ABSTRACT

Indium and zinc extraction from an indium-bearing zinc residue were investigated using sphalerite concentrate as a reductant in sulfuric acid medium. The effects of amount of sphalerite concentrate, sulfuric acid concentration, particle size, leaching time as well as temperature were discussed. The results showed that high indium and zinc extraction yield as well as high $\text{Fe}^{2+}/\text{Fe}^{3+}$ molar ratio could be obtained by reduction leaching of zinc residue with addition of sphalerite concentrate as a reductant. The optimal leaching condition was determined as 150 g/L H_2SO_4 , 0.95 times of theoretic amount of sphalerite concentrate for 4 h at 90 °C while using particles in the range of 74–58 μm . The leaching efficiencies were 94.8% of indium, 96.1% of zinc and 92.8% of iron, respectively, and a $\text{Fe}^{2+}/\text{Fe}^{3+}$ molar ratio of 7.5 in the leach solution was also obtained. The process above was a viable method that effectively extracted zinc and indium and converted Fe^{3+} into Fe^{2+} at the same time.

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1. Introduction

Indium is a valuable metal used in electrical industry extensively, which used mainly as indium tin oxide-films in liquid crystal displays (Hsieh et al., 2009; Biswas et al., 2015). The main source of indium is the waste generated from the zinc industry, from which it is recovered as a by-product (Alfantazi and Moskalyk, 2003). In the conventional hydrometallurgical process of roast-leaching-electrowinning, which employed to treat zinc concentrates, more than 98% of indium and 20–30% of zinc present in material are transferred into leach residue. This residue is an important resource for recovery of indium and zinc (James et al., 2000).

In the leach residue, a significant portion of the zinc and iron is in the form of zinc ferrite (ZnFe_2O_4), and most of the indium is present due to zinc ferrite isomorphism (Rao and Rao, 2005). The Waelz kiln process is the traditional way to treat the zinc residue for recovery zinc, indium and lead from the zinc residues. Unfortunately, this process has the drawbacks of high energy-consuming, air pollution and the resulting slag still has eco-compatibility problems (Menad et al., 2003; Huang et al., 2012; Mombelli et al., 2015). The hot acid leaching is another conventional method for recovery zinc and indium from the zinc residue (Nii and Hisamatsu, 1966a, 1966b). In the hot acid leaching process, zinc, iron, indium and other valuable metals are dissolved. While most of the iron ions in the obtained leach solution are ferric ion, in order to separate and recover zinc and indium, jarosite precipitation for removing of ferric ion is a necessary process. However, in the case of indium,

during the jarosite precipitation processes, most of indium in the leach solution was reported to the jarosite. The recovery rate of indium from the jarosite is lower (Ning and Chen, 1997; Yuan et al., 2008; Li et al., 2010).

Besides hot acid leaching process, the reductive leaching is an effective method to extract zinc and indium from zinc residue. It was reported that the reducing conditions in the leaching process resulted in enhanced dissolution rates of zinc from zinc ferrite (Elgersma et al., 1992; Nii and Hisamatsu, 1966a, 1966b; Wu et al., 2012). Various electrochemical studies also confirmed it (Lu and Muir, 1988; Bhat et al., 1987). In the reductive leaching process, zinc, indium, and iron are dissolved, and ferric iron is simultaneously reduced to ferrous iron (Li et al., 2006; Markus et al., 2004). Indium in the leach solution can be efficiently selectively extracted by direct solvent extraction (Li et al., 2015). Iron in the leach solution can be removed by hematite (Riveros and Dutrizac, 1997; Yang et al., 2014).

The reduction leaching processes is an effective method for minerals containing oxide of iron, nickel, cobalt and manganese (Abbruzzese, 1990; Kumar et al., 1993; Monade and Momada, 1999; Das and De Lange, 2011). Reductive leaching of metal oxide chalcopyrite, electric arc furnace dust, spent zinc-carbon batteries as well as stibnite flotation concentrate have also been reported in several studies using different acids and reductants (Dreisinger and Abed, 2002; Mahlangu et al., 2006; Furlani et al., 2009; Kim et al., 2009; Ghafarizadeh et al., 2011). However, the reduction leaching of zinc and indium from indium-bearing zinc residue using sphalerite concentrate as a reductant has barely been considered. In the present study, the reductive leaching of zinc and indium from the indium-bearing zinc residue using sphalerite concentrate as a reductant has been investigated. The aim is to provide

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an effective method of reductive leaching for extraction of zinc and indium and replace the traditional hot acid leaching process.

2. Materials and methods

2.1. Materials

The indium-bearing zinc residue and sphalerite concentrate used in this work were obtained from Yunnan Province of China. The compositions of the materials were listed in Table 1. X-ray diffraction of the indium-bearing zinc residue identified zinc ferrite (ZnFe_2O_4) as the main mineral components in this residue (Fig. 1 (a)), and sphalerite (ZnS) and christophite ($(\text{Zn,Fe})\text{S}$) as the main mineral components of sphalerite concentrate (Fig. 1(b)).

2.2. Methods

A five-necked, round-bottomed flask (1 L) was fitted with a mechanical stirrer, a sample collection, a pH/Eh meter and two condenser tubes. The flask was then immersed in a water bath and maintained at the selected temperature ± 1.0 °C. 570 mL of leaching solution was placed in the flask and heated to the desired temperature while being magnetically stirred (400 rpm). 50 g of indium-bearing zinc residue and the required amount of sphalerite concentrate was then added to the reactor. The addition of sphalerite (η) was evaluated by the formulas:

$$\eta = \frac{M_{\text{Real}}}{M_{\text{Theory}}} \quad (1)$$

$$M_{\text{Theory}} = \frac{M_{\text{ZR}} \times C_{\text{Fe}} \times 32}{C_{\text{S}} \times 56 \times 2} \quad (2)$$

where M_{Real} is the actual amount of sphalerite, M_{Theory} the theoretic amount of sphalerite, M_{ZR} the amount of zinc residue, C_{Fe} the wt% of Fe in zinc residue, C_{S} the wt% of S in sphalerite. At the end of each leaching experiment, the slurry was filtered, and the leach liquor and the solid residue were analyzed, and extractions of zinc, indium and iron were calculated from the solid chemical analysis.

Zinc was analyzed by complex titration with EDTA. Indium concentration was determined by ICP with mass spectrometric detection (ICP-MS). The concentrations of ferrous ion were analyzed by complex titration with potassium dichromate. The concentrations of ferric ion were determined by finding the difference between overall iron and ferrous ion concentrations. X-ray powder diffraction was carried out using Rigaku D/MAX 2500v diffractometer (Japan). The potential of solution was measured by a platinum electrode and an Ag/AgCl electrode used as the reference electrode.

3. Results and discussion

3.1. Effect of amount of sphalerite concentrate

Fig. 2 presented the effect of amount of sphalerite concentrate on extractions of zinc, iron and indium. It can be seen from Fig. 2 that the extractions of zinc, iron and indium increased and then decreased with the increase of amount of sphalerite concentrate. In the absence

Table 1
Chemical composition of indium-bearing zinc residue and sphalerite concentrate.

Components	Zn	Fe	In(g/t)	Ag(g/t)	S	SiO ₂	Cu	Pb
Indium-bearing zinc residue (%)	23.40	30.9	510	97.2	3.75	5.72	1.00	0.64
Sphalerite concentrate (%)	47.27	14.44	354.7	45.7	33.08	2.43	0.74	0.061

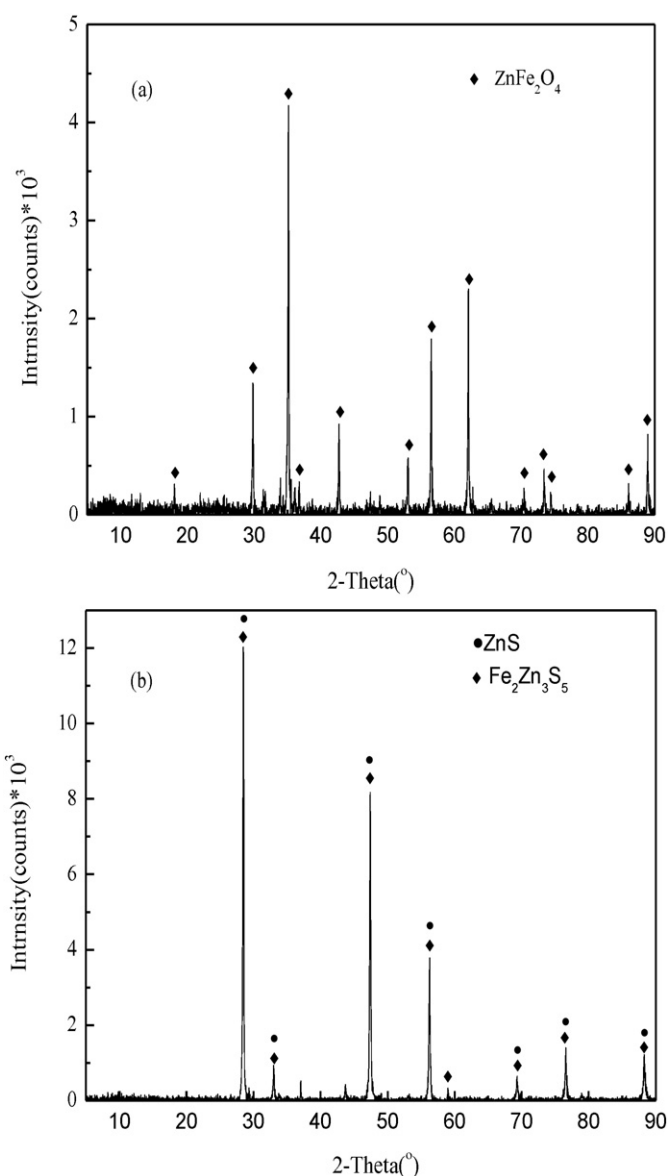
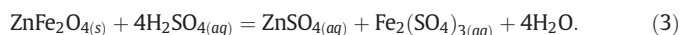
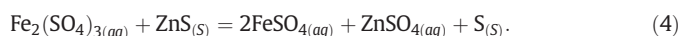


Fig. 1. XRD of indium-bearing zinc residue (a) and sphalerite concentrate (b).

of sphalerite concentrate, the extractions were 87.8% zinc, 78.8% iron and 81.6% indium after 5 h. With 0.95 times of theoretic amount of sphalerite concentrate, the extractions of zinc, iron and indium were increased significantly to 96.4%, 93.8% and 95.8%, respectively. The zinc ferrite was dissolved in sulfuric solution as follows:



By dissolving of zinc ferrite, Fe^{3+} is released. The high Fe^{3+} concentrations retarded the dissolution of ZnFe_2O_4 . In sulfuric acid solution, the Fe^{3+} was reduced to Fe^{2+} by sphalerite as follows:



By simultaneously dissolving ZnFe_2O_4 and converting Fe^{3+} to Fe^{2+} , the dissolution of zinc ferrite will not be retarded by the presence of Fe^{3+} , and the reaction of zinc ferrite dissolution (Eq. (3)) was shifted to the right. What is more, the activity of H^+ was increased and zinc ferrite dissolution was promoted by converting Fe^{3+} to Fe^{2+} (Dimitrios and George, 1991). The extractions of zinc, iron and indium had stopped the ongoing growth when the initial amount of sphalerite was over

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