

Electro-oxidation of sphalerite in weak alkaline sodium chloride solution



Zhan-fang Cao ^{*}, Ming-ming Wang, Hong Zhong ^{*}, Zhao-hui Qiu, Pei Qiu, Ya-jun Yue, Guang-yi Liu, Shuai Wang

College of Chemistry and Chemical Engineering, Central South University, Changsha, Hunan 410083, PR China

ARTICLE INFO

Article history:

Received 5 May 2015

Received in revised form 10 July 2015

Accepted 10 August 2015

Available online 12 August 2015

Keywords:

Sphalerite
Electro-oxidation
Weak alkaline
Mechanism

ABSTRACT

The technology of Zn extraction from sphalerite concentrate by using electro-oxidation in weak alkaline sodium chloride solution has been investigated in this paper. The effects of several parameters on Zn recovery during the electro-oxidation have been investigated, and the optimized conditions are listed as follows: pH in the range of 8.5–8.8, NaCl concentration = 3 mol/L, reaction time = 1 h, solid–liquid ratio \geq 0.03 g/ml, anodic current density = 1342 A/m², stirring speed = 800 rpm. Under these experimental conditions, sphalerite concentrate can be oxidized efficiently. Furthermore, the electro-oxidation mechanism of sphalerite in weak alkaline NaCl solution has been studied. It was found that the elemental sulfur and the SO₄²⁻ were the major existing forms of S from sphalerite and Zn would precipitate in the form of Na₂Zn₃(CO₃)₄ in the case where Na₂CO₃ served as electrolyte pH regulator. To prevent SO₄²⁻ from accumulating in the electrolyte and precipitate Zn as Zn(OH)₂ instead of Na₂Zn₃(CO₃)₄, Na₂CO₃ was replaced by Ca(OH)₂ to stabilize the pH of electrolyte, and the effect of the addition of Ca(OH)₂ on Zn recovery was investigated.

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1. Introduction

The commonly used hydrometallurgical technologies to treat sphalerite are roasting–acid leaching–electrowinning and oxygen pressure acid leaching processes (Mei et al., 2001; Mulaba-Bafubandi and Waanders, 2005). However, SO₂ generated and discharged at the roasting stage in the former process will inevitably pollute the air (Peng et al., 2005; Santos et al., 2010). As to the latter process, the elevated temperature and high pressure required in the leaching step make the process not cost-effective (Zhang et al., 2008) and difficult to be performed.

Considerable attention has been paid to some other methods of dealing with sphalerite in recent decades. The utilization of ferric sulfate and ferric chloride to leach sphalerite has been reported by some researchers, and the relevant investigations on the kinetics of dissolution of sphalerite have been carried out (Bobeck and Su, 1985; Lochmann and Pedlik, 1995; Palencia and Dutrizac, 1991; Santos et al., 2010). The leaching of sphalerite with ammonia as lixiviant under high pressure oxygen condition has been studied deeply (Sarveswara Rao and Ray, 1998; Sarveswara Rao et al., 1993; Wang et al., 2011). Microbial leaching of sphalerite has been practiced and investigated for a long time (Ake and Stig, 1997; Wang et al., 2006). Furthermore, some researchers combined bioleaching and chemical leaching as an integrated process to treat zinc sulfide (De Souza et al., 2007). The studies on nonoxidative dissolution and oxidative leaching of sphalerite by hydrochloric acid and hypochlorous acid respectively have been reported in

some papers (Cho, 1987; Majima et al., 1981). Some investigators succeeded to achieve a high leaching efficiency of Zn from sphalerite in alkaline solution with the help of chemical conversion of lead carbonate (Zhang et al., 2008).

Besides, the electro-oxidation leaching has also been employed to treat sphalerite, which has been reported by a few researchers (Qiu, 1999). In the conventional electro-oxidation process in which acid chloride solution is usually adopted as its electrolyte (Arslan and Duby, 2003), the sulfide minerals are leached at the anode and the metal ions are reduced at the cathode simultaneously. The leaching of the sulfide minerals at the anode is often attributed to the oxidation of mineral particles by current, Fe³⁺/Cu²⁺ in the electrolyte and the oxidized chloride species generated at the anode and to the nonoxidative dissolution of mineral particles in acid electrolyte (Gordy, 1973; Kruesi, 1972; Qiu, 1999; Zhang et al., 1998). As the traditional electro-oxidation leaching is implemented in the strong acid chloride solution, the emission of Cl₂ which is widely recognized as the air pollutant will be inevitable in the process and severely restrict the development of electro-oxidation technology. Therefore, the electro-oxidation of sphalerite in weak alkaline sodium chloride solution, which has not been reported yet, was investigated in this paper.

It has been confirmed in our previous work that the electro-oxidation leaching of molybdenite in weak alkaline sodium chloride solution is feasible (Cao et al., 2010). Additionally, as pointed out by some other researchers, it is theoretically much easier for sphalerite than for molybdenite to be oxidized in the electro-oxidation process (Qiu, 1999). Hence, the possibility of the electro-oxidation leaching of sphalerite in weak alkaline sodium chloride solution has been investigated in this paper. The experimental tests on how the process parameters-

^{*} Corresponding authors.

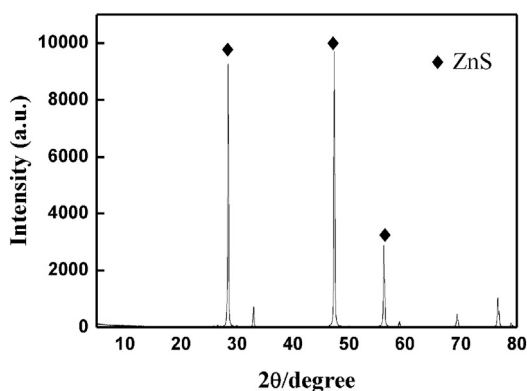


Fig. 1. X-ray diffraction result of the sphalerite concentrate.

electrolyte pH (regulated by Na_2CO_3), NaCl concentration, reaction time, liquid–solid ratio, anodic current density and stirring speed-affected the electro-oxidation of sphalerite were carried out. Moreover, the reaction mechanism in the process has been investigated and discussed. The addition of $\text{Ca}(\text{OH})_2$ rather than Na_2CO_3 to the electrolyte to stabilize weak alkaline environment has been studied.

2. Experimental

2.1. Concentrate sample and reagents

The XRF (EDX1800B X-ray Fluorescence meter produced by Skyray Instrument Co., Ltd., Kunshan, China) result of sphalerite concentrate used in all electro-oxidation tests is listed as follows (%): Zn 60.3, S 28.09, Fe_2O_3 4.42, SiO_2 2.88, Al_2O_3 1.39, Cu 0.09, Cd 0.14, Pb 0.38. XRD (Rigaku 2500 X-ray Diffraction meter produced by Rigaku Corporation, Japan, scanning range of 5° – 80° , scanning speed at $8^\circ/\text{min}$, JADE 5.0 analysis software, these conditions are applied to all XRD analyses in this paper) result of the concentrate sample (Fig. 1) shows ZnS was the major phase, while the particle size was in the range of $10\ \mu\text{m}$ to $100\ \mu\text{m}$ with 83% less than $40\ \mu\text{m}$ (MS-2000 Laser particle size analyzer, Britain). In all electro-oxidation tests, except for water which was tap water, all reagents involved in the tests were chemical grade. The reagents employed to analyze Zn^{2+} content in solution and precipitate SO_4^{2-} were analytical grade.

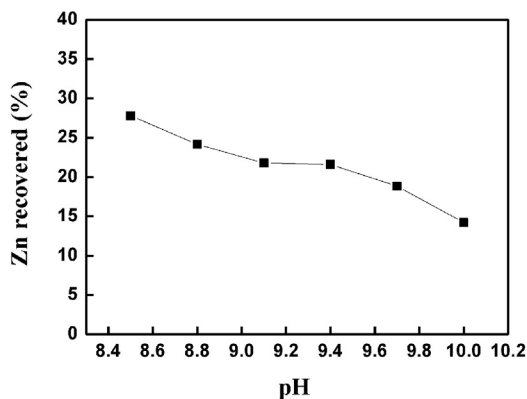


Fig. 2. Effect of pH on Zn recovery. Experimental conditions: NaCl concentration = 2 mol/L, reaction time = 1 h, S/L ratio = 0.03 g/ml, anodic current density = $1342\ \text{A}/\text{m}^2$, stirring speed = 800 rpm.

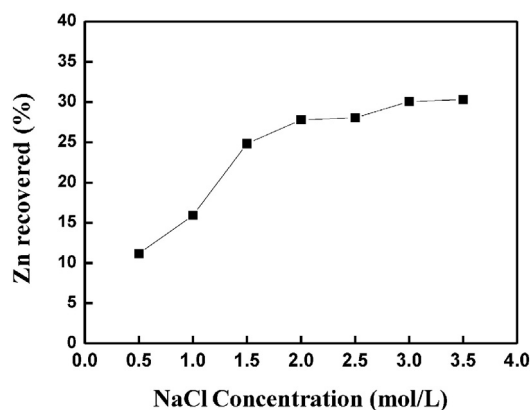


Fig. 3. Effect of NaCl concentration on Zn recovery. Experimental conditions: pH = 8.5–8.8, reaction time = 1 h, S/L ratio = 0.03 g/ml, anodic current density = $1342\ \text{A}/\text{m}^2$, stirring speed = 800 rpm.

2.2. Electro-oxidation experiments

2.2.1. Instruments and determination of Zn^{2+}

A self-made septum-free electrolytic bath made of plexiglas ($7.5\ \text{cm} \times 5.5\ \text{cm} \times 5.5\ \text{cm}$) was used in the electro-oxidation tests. DSA electrode (dimensionally stable electrode, RuO_2 , IrO_2 , SnO_2 coated mesh electrode) and graphite rod were adopted as anode and cathode respectively. The pH meter (pHS-3C pH meter produced by INESA Scientific Instrument Co., Ltd., Shanghai, China) was used to monitor electrolyte pH, the magnetic stirrer (DF-101S magnetic stirrer produced by Greatwall Scientific Industrial And Trade Co., Ltd, Zhengzhou, China) to sustain the suspension of mineral particles and the S22 Regulated Power Supply System (Chenhua Instruments Co., Ltd., Shanghai, China) to provide applied potential between anode and cathode in electro-oxidation tests.

In all electro-oxidation experiments, Zn from sphalerite precipitated in a certain form which depended on the specific alkaline environment. To analyze the mass of Zn recovered, the residue containing Zn precipitate was firstly dissolved in HCl solution. Then, the EDTA titration method (0.1 mol/L EDTA, xylenol orange used as indicator, sodium thio-sulfate used as masking agent, HAC–NaAc buffer responsible for stabilizing solution pH at 5.4–5.9) was adopted to analyze Zn^{2+} content.

2.2.2. Procedure

All electro-oxidation experiments were carried out at room temperature with current of 1.5 A and electrolyte volume of 100 ml. The other specific conditions are given in Section 3.

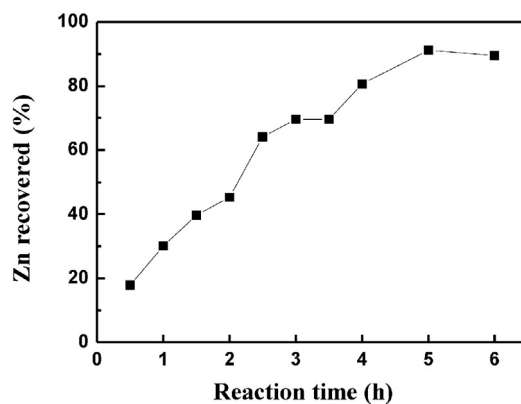


Fig. 4. Effect of reaction time on Zn recovery. Experimental conditions: pH = 8.5–8.8, NaCl concentration = 3 mol/L, S/L ratio = 0.03 g/ml, anodic current density = $1342\ \text{A}/\text{m}^2$, stirring speed = 800 rpm.

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