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Microwave roasting and leaching of an oxide-sulphide zinc ore

Kun Yang^{a,b,c}, Shiwei Li^{a,b,c,*}, Libo Zhang^{a,b,c}, Jinhui Peng^{a,b,c}, Weiheng Chen^{a,b,c}, Feng Xie^{a,b,c}, Aiyuan Ma^{a,b,c}

^a Yunnan Provincial Key Laboratory of Intensification Metallurgy, Kunming, Yunnan 650093, China

^b National Local Joint Laboratory of Engineering Application of Microwave Energy and Equipment Technology, Kunming, Yunnan 650093, China

^c Faculty of Metallurgical and Energy Engineering, Kunming University of Science and Technology, Kunming, Yunnan 650093, China

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ABSTRACT

In this paper, a novel combination of phase transformation and leaching is proposed to recover zinc from an oxide-sulphide zinc ore. Accordingly, various analytical technologies, such as X-ray diffraction, chemical analysis, Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy, are employed. The phase transformation of a raw ore and the effects of microwave heating and Na₂O₂ addition on zinc leaching are investigated. The results show that the addition of a strong oxide Na₂O₂ can directly transform the refractory phases, particularly zinc sulphide, into zinc sulphate. As a result, SO₂ emissions are avoided and an environmentally friendly zinc recovery process is realised. Both microwave heating and the Na₂O₂ level have significant effects on phase transformation and zinc leaching. The zinc leaching rate can reach 82.06% under the following conditions of microwave heating: adding Na₂O₂ at a mass ratio of 25%, leaching in an ammonium chloride solution with a total ammonium concentration of 7.5 mol/L and an ammonia/ammonium chloride molar ratio of 1:1, adding NaClO as an aid-leaching reagent and leaching for 1 h. By contrast, the zinc leaching degree for the ore samples roasted in a traditional electric resistant furnace is only 30.62%. Moreover, at the same oxidation degree, the total energy consumption by the proposed process is 0.78 kWh/(kg·ore), which is lower than that by traditional heating (same power). Thus, the combination technology of microwave-heated phase transformation and leaching is effective in treating oxide-sulphide zinc ore.

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1. Introduction

With the development of the zinc industry, traditional zinc resources, such as zinc sulphide ores, are becoming scarce and are unable to meet the need (Peng, 2005). Thus, non-conventional zinc resources, particularly oxide-sulphide zinc ores, which have large reserves, should be exploited and utilised. However, oxide-sulphide zinc ore presents several problems, including a high alkaline gangue content, polymetal intergrowth and the occurrence of multiple phases of a single metal. In recent years, the efficient utilisation of oxide-sulphide zinc ores has become a research hotspot (Yang et al., 2006).

The conventional zinc recovery process is characterised by the roasting-leaching-electrowinning processual sequence. This process involves roasting sulphides into oxides or sulphates (Chen and Dutrizac, 2004). This process is also suitable for oxide-sulphide zinc ores; however, owing to the limited raw materials and environmental hazards from SO₂ emissions, the widespread application of this process is restricted (Uçar, 2009). Novel hydrometallurgical routes always pose difficulties, particularly in leaching ZnS, ZnSiO₄ or ZnFeO₄, and acid leaching (Kelebek et al., 2004; Mizoguchi and Habashi, 1981; Perez

and Dutrizac, 1991; Souza et al., 2007) and ammonia leaching (Majima et al., 1981; Sarveswara et al., 1992; Wang et al., 2008) are normally employed in these settings. Therefore, identifying a cost-effective, environment-friendly zinc recovery process remains a major challenge.

As an emerging green technology, microwave technology is widely used in communication technologies, including radars, cellular phones, televisions and satellites (Thostenson and Chou, 1999). Unlike heat generated from traditional electrical resistance heating, microwave energy can be dissipated in the volume of the material and transformed into inner energy. Therefore, microwave energy allows for non-contact heating, efficient energy transfer, rapid heating, selective heating, volumetric heating, quick initiation and termination of heating, initiation of heating from the interior of the material body and a high level of safety and automation (Al-Harabsheh and Kingman, 2004; Haque, 1999). Therefore, the application of microwave energy to recover zinc from oxide-sulphide zinc ore has considerable potential.

In the present study, a new process for recovering zinc from the oxide-sulphide zinc ore is designed. The phase transformation process is discussed. Furthermore, the factors that can influence phase transformation and zinc leaching, including the heating technology and Na₂O₂ level, are investigated in detail. The objective of this study is to develop new metallurgical technology that uses environmentally friendly methods and reagents to enhance the zinc leaching efficiency for oxide-sulphide zinc ore.

* Corresponding author at: Kunming University of Science and Technology, Kunming, Yunnan 650093, China.

E-mail address: lswei11@163.com (S. Li).

Table 1
Main chemical composition of oxide–sulphide zinc ore (mass fraction, %).

Zn _T	Fe	Pb	S	SiO ₂	Al ₂ O ₃	MgO	CaO
35.23	9.91	4.84	13.54	7.51	1.02	0.21	1.47

Zn_T–total zinc.

2. Materials and methods

2.1. Materials and equipment

The oxide–sulphide zinc ore used in this study was obtained from Lanping County of China, and its main chemical composition is shown in Table 1. The zinc phase distribution in the ore is presented in Table 2.

As shown in Tables 1 and 2, the oxide–sulphide zinc ore is a refined zinc ore with a zinc content of 35.23%. The ratio of zinc oxide and zinc sulphide content is nearly 1.0 (ZnSO₄ and (Zn,Mn)Fe₂O₄ are both zinc oxides). X-ray diffraction (XRD) was also performed to identify the component phases of the ore further, and the results are provided in Fig. 1.

The crystalline zinc phases can be classified into two main categories, zinc oxide (ZnCO₃, Zn₂SiO₄ and ZnSO₄) and zinc sulphide (ZnS). Other crystalline components, such as PbS, Fe₂S and SiO₂, also exist. A key concept of zinc recovery from the oxide–sulphide zinc ore is to realise the simultaneous and selective leaching of multiple zinc phases.

A high-temperature microwave reactor used in this experiment is fabricated by the Key Laboratory of Unconventional Metallurgy of the Ministry of Education, Kunming University of Science and Technology. The reactor uses 2450-MHz magnetron tubes as microwave generators. The microwave reactor mainly consists of a rectangular cavity, four magnetron tubes, and matching waveguide transmission tubes. The power of the microwave reactor can be controlled from 0 kW to 6 kW through a regulator. The leaching process was conducted using a CJJ-93/HJ-6 six-connected magnetic stirrer with a speed limit of 2000 rpm and power of approximately 25 W per motor.

2.2. Analytical techniques

XRD analysis was conducted to characterise the component phases. Fourier transform infrared spectroscopy (FT-IR) analysis was performed to determine the functional group and track the course of the chemical reactions. The FT-IR spectra were acquired with a Thermo Fisher Scientific Nicolet iS10 FT-IR spectrometer, which has a DTGS detector, a Ge/KBr beamsplitter, plane mirror interferometer driven by an electromagnetic force and long-life middle/far infrared source. The spectrometer has the following specifications: an ASTM linearity lower than 0.1%, a spectral resolution above 0.4 cm⁻¹ and a sensitivity higher than 45,000:1. X-ray photoelectron spectroscopy (XPS) analysis can provide information about the surface of a sample, i.e., the species present and their chemical states. The XPS spectra were recorded using an ULVAC PHI-5000 VersaProbe II spectrometer with an Al K-alpha X-ray source (hν = 1486.6 eV) at a normal take-off angle and band-pass energy of 50 eV, corresponding to an energy resolution of 1.2 eV. The vacuum pressure in the chamber was sooty to 10⁻⁷ Torr. The X-rays were non-monochromatic, and the instrument was calibrated against the C1s peak binding energy (284.8 eV), which is in accordance with the studies of Buckley et al. (1989a, b) and Kosova et al. (2007). The charge

compensation was adjusted for each sample to obtain the maximum signal possible for Zn2p_{3/2} at Eb = 464 eV.

2.3. Experimental procedure

The synthetic leaching of the oxide–sulphide zinc ore could be divided into two processes, phase transformation and leaching. The phase transformation process was as follows: Na₂O₂ was mixed with the zinc ore samples at certain mass ratios, and each mixture was roasted in the microwave reactor to prompt refractory phase smelting. The leaching process involved grinding of the smelted phases to 50 μm particles, adding an oxidant to the leaching solution and mechanical agitation of the leach. The entire process is shown in Fig. 2.

3. Results and discussion

3.1. Heating curves of the oxide–sulphide zinc ore

The heating curves of the oxide–sulphide zinc ore samples with different Na₂O₂ levels are shown in Fig. 3.

As shown in Fig. 3, the microwave-absorbing capability of the oxide–sulphide zinc ore is weak or nil. Its initial heating rate is significantly low. At approximately 300 °C, its heating rate shows a gradually rising trend. Once Na₂O₂ is added, its microwave-absorbing capability significantly improves, and a higher heating rate can be achieved by adding more Na₂O₂. The oxide–sulphide zinc ore can be heated to 600 °C within 5 min when the added Na₂O₂ level exceeds 15%. Therefore, using Na₂O₂ as an additive can reduce the heating time, reducing the reaction energy.

In the present work, the selection of Na₂O₂ as the flux was based on its good microwave-absorbing properties, and the addition of Na₂O₂ was imperative to ensuring sufficient oxidation of the oxide–sulphide zinc ore and reduction of the reaction time. According to experiments, the addition of 25% Na₂O₂ is appropriate. The dielectric constants, heating rates of Na₂O₂ and main component phases of the complex ore samples heated to temperatures ranging from 200 °C to 800 °C in air are shown in Table 3. The dielectric constant measures the ability of a material to absorb or store microwave energy, i.e., the ability to be polarised.

As shown in Table 3, the component phases of the oxide–sulphide zinc ore were materials that do not, or at least weakly, absorb microwaves, whereas the microwave-absorbing property of Na₂O₂ was far superior to any of the zinc phases. As a result, Na₂O₂ could promote the microwave heating rate of the mixture, and it showed strong oxidising properties.

3.2. Phase transformation process

The applicable phase transformation process for the oxide–sulphide zinc ore was determined by first investigating the components that change during the heating process. In this study, the specific heating temperatures (i.e., 200 °C, 300 °C, 400 °C, 500 °C, 600 °C and 700 °C) were selected for phase-changing analysis, and the steps were as follows: Na₂O₂ was mixed with the oxide–sulphide zinc ore mixture at a mass ratio of 25%. Each temperature was achieved at a heating rate of 10 °C/min, and a holding time of 5 min was observed in a traditional electrical resistance furnace. Portions

Table 2
Zinc phase distribution of oxide–sulphide zinc ore.

Zinc phase	Zinc sulphate	Zinc oxides	Zinc sulphide	Franklinite et al	Zn _T
Mass fraction/%	0.20	15.90	17.88	1.25	35.23
Distribution/%	0.57	45.13	58.75	3.55	100

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