



Selective recovery of valuable metals from partial silicated sphalerite at elevated temperature with sulfuric acid solution



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ABSTRACT

The purpose of this work was to study the feasibility at laboratory-scale of a hydrometallurgical process for the selective recovery of valuable metals from partial silicated sphalerite in an oxygen pressure acid leaching system. The factors influencing dissolution efficiency of the ore were investigated and optimized. Under optimum conditions (i.e., temperature of 433 K, sulfuric acid concentration of 41.2 g/L, leaching time of 2.5 h, liquid/solid ratio of 6 mL/g, and pressure of 1.6 MPa) over 97% Zn was extracted into the leach liquor together with 0.3% SiO₂ and 2.9% Pb. The leaching slurry had good solid–liquid separation characteristics, and the filtration rate could be as high as 716 L/m² h. About 96% oxidation of sulfide sulfur to sulfate was achieved under these conditions. Analysis of the content of elemental sulfur in the leaching residues indicated that the fraction of sulfide sulfur determined as elemental sulfur was about 10% at 393 K, and that it decreased with temperature down to 0.5% at 453 K. Ultimate solid residues, which have been concentrated in silica and lead, can be oriented toward the lead smelter after alkali roasting–water washing pretreatment for metal recovery.

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1. Introduction

The rapid consumption of primary zinc resources and ever-increasing demand for zinc metal continue to stimulate worldwide investigations on alternate zinc resources for recovering zinc metal. One such alternate resource with respect to diminishing zinc reserves is complex zinc-bearing ores. They generally contain zinc metal in the form of multimetallic sulfides or partial silicated sphalerite, and have not been utilized widely owing to a lack of available cost effective processes [1,2].

The conventional method for producing zinc and other metals from complex zinc-bearing ores has been the differential flotation method, which produces individual metal concentrates that easily may be treated by pyro- and/or hydrometallurgical processes. Researchers from different countries have been working on beneficiation of these ores for several decades [3–8]. However, very little progress has been made due to some mineralogical characteristics and flotation chemistry differences among complex ores from different regions [8,9].

In the case of multimetallic sulfides, several methods to deal directly with these ores have been reported and compared in the literature [10–16]. Among these methods, pressure acid leaching

has a promising future, and its application in the zinc industry has attracted attention as an environmentally friendly process. A hydrometallurgical process based on pressure oxidation leaching of zinc sulphide concentrate was first developed commercially by Sherritt Gordon, Fort Saskatchewan, Canada [17]. The pressure leaching expansion was commissioned in 1983, and the operation was described in detail by Johnston and Doyle [18]. Since then, pressure acid leaching has been used by many researchers for effectively extracting zinc from zinc-bearing ores, such as sulfide ores [15,19–22] and oxidized ores containing high content of silica [23,24]. These studies have indicated that this process has had an extensive application in some zinc minerals processing operations. Nonetheless, little work has examined the application of pressure acid leaching process to treat effectively and economically the partial silicated sphalerite. Such ores have been found in large quantities in southwest China, and have presented a metallurgical challenge for many years [6,25,26]. As reported earlier, beneficiation experiments indicated that it also would be very difficult to produce desirable metallurgical samples from partial silicated sphalerite using traditional ore beneficiation methods, due to the fact that both sulfides and silicates co-exist at high silica levels and fine grained nature [7,25] and that the ores contain many kinds of impurities and substantial amounts of pyrite and gangue minerals (like quartz and calcite), which make the beneficiation of said ores difficult. The complex mineralogy of said ores, their high silica content, and their generally low solubility in most solvents have

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placed a limitation on effectiveness of standard leaching techniques, particularly because the silicate constituent present in the raw material readily enters solution as a silica gel with the zinc, leading to the difficulty of separating the valuable-metal containing solution from the solid leaching residue [27,28]. Thus, such ores were very often left untreated as a result of the metallurgical difficulties encountered in selective recovery of zinc and other metals from partial silicated sphalerite.

The high temperature leaching of zinc containing raw mineral materials under pressure has attracted considerable attention from the zinc industry during the recent years. By this process according to the existing established application of said pressure leaching technology to sulfide ores and oxidized ores containing high content of silica it is possible to treat directly the partial silicated sphalerite. For partial silicated sphalerite, pressure leaching technology as a preferred route mainly involves a sulfuric acid solution as the reaction medium and can destroy various minerals rapidly.

The aim of this study is to evaluate the selective leaching behavior of partial silicated sphalerite using a sulfuric acid solution under oxygen pressure, and thus to determine the conditions under which the best leaching efficiencies may be achieved that also produce good characteristics of solid-liquid separation.

2. Experimental

2.1. Materials

Partial silicated sphalerite samples were obtained from Yunnan Province of China. The ore, which was used for the leaching experiments, was ground to less than 104 μm in a ball grinder.

Sulfuric acid (AR) with a purity of 98% and a density of 1.84 g/mL was used as the leaching agent in all experiments. All the aqueous solutions were prepared by using distilled water. To enhance ore dissolution, reagent grade sodium lignosulfonate was added as a surfactant to prevent the inactivation of unreacted ore particle surfaces by precipitated sulfur [29].

2.2. Equipment and procedure

All experiments were conducted in a 500 mL vertical titanium-lined autoclave (Model 09041904-2/2 Reactor, Weihai Chemical Machinery Instrument Limited Company, Shandong Province, China). The autoclave was fitted with an internal water-cooling titanium spiral tube, and a sparging tube through which O_2 was bubbled into the leaching solution. Agitation was provided by a titanium propeller on a shaft connected to a variable-speed magnetic stirrer having a digital controller unit. The temperature of the reaction medium was monitored and controlled with an accuracy of $\pm 1^\circ\text{C}$ by a platinum resistance temperature sensor that was connected to a digital temperature controller.

For each run, 20 g of ore mixed with both a known weight of sodium lignosulfonate and 200 mL of leaching solution containing a pre-calculated concentration of H_2SO_4 were placed in the autoclave. The slurry was heated to the required temperature varying from 373 to 453 K under 1.2 MPa of oxygen pressure while being agitated at 550 rpm, unless otherwise stated. The leaching was maintained for a predetermined time. After reaching the specified time, heating was stopped, the oxygen pressure was shut off, and the samples were cooled to 343–353 K. The leaching sample was filtered after the final pH value of leaching slurry removed from the reactor was measured by pH meter (Model Rex PHSJ-5, Shanghai INESA Scientific Instrument Co., Ltd, Shanghai, China). The volume of the leaching solution was measured by the graduate cylinder. The filtrate cake was washed twice with 20 mL of dilute sulfuric acid (0.02 vol% H_2SO_4) to prevent possible

precipitation of soluble metals. After drying in a baking oven at 338 K for 24 h, the crushed and homogenized residue was chemically analyzed to determine the content of zinc, lead, sulfur and silica. Analysis methods of zinc, lead, sulfur and silica in the residue were briefly stated as follows [30]. The zinc and lead in the residue were analyzed by EDTA titration. The elemental sulfur was analyzed by extraction with carbon disulfide (as the solvent for sulfur) in a Soxhlet apparatus, and the silica was analyzed by silicon molybdenum blue spectrophotometry. Zinc extraction was calculated as follows:

$$\text{Zinc extraction (\%)} = \left[1 - \frac{\text{Zn present in the residue}}{\text{Zn initially present in the partial silicated sphalerite}} \right] \times 100.$$

The filtration rate was determined by the following formula: The filtration rate ($\text{L}/\text{m}^2 \text{ h}$) = $V/(S \times t)$, where V (L) is the volume of the leaching solution, S (m^2) is the filter area of the funnel, and t (h) is the filter time.

3. Results and discussion

3.1. Chemical and mineralogical characteristics of ore sample used

The chemical composition of the partial silicated sphalerite was analyzed by chemical methods as described in this book that wrote by the test institute of Beijing General Research Institute of Mining & Metallurgy [30]. Zinc, sulfur, silicon, iron, lead and calcium are the major constituents of the partial silicated sphalerite ground, which has the following chemical composition (wt.%): ZnO 24.52, SO_2 40.52, Free silica 16.8 (Total silica 21.3), Fe_2O_3 20.36, PbO 8.09, CaO 2.8, Al_2O_3 1.91, K_2O 1.19.

The phases detected by the X-ray diffraction (XRD) technique in the ground ore are mainly sphalerite, hemimorphite, quartz, galena, pyrite, troilite, muscovite, calcite and cerussite. These compounds show a relatively complex structure, which makes the ore beneficiation difficult. The occurrence of main heavy metal (zinc and lead) in the ore ground was investigated by means of chemical phase analysis as described by Zhang [31]. The results are shown in Table 1. It can be revealed from Table 1 and XRD analysis results that the major zinc-containing phases in the ore sample are sphalerite and hemimorphite, and that the main phase composition of lead is galena and cerussite. In addition, according to the chemical composition and XRD analysis of the ore sample, about 14.25% of the iron is presented mainly with the form of pyrite and troilite, and the ore sample contains about 16.8% free silica as quartz, the remaining silica exists mainly in hemimorphite. The gangue mineral in the ore sample is mainly quartz, followed by calcite. Both calcite and muscovite present in the ore sample would cause significant increase in acid consumption.

The backscattered electrons (BSE) image by scanning electron microscope (SEM) coupled with energy dispersive X-ray spectrometer (EDS) can be used to complement the XRD analysis and

Table 1
Phase composition of zinc and lead in the sample.

Constituent	Zinc		Lead	
	Content (wt.%)	Distribution ratio (%)	Content (wt.%)	Distribution ratio (%)
Carbonate	1.92	9.57	2.89	37.05
Silicate	6.86	34.2	0.76	9.74
Sulfide	10.92	54.43	3.58	45.9
Others	0.36	1.8	0.57	7.31
Total	20.06	100	7.8	100

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