RESEARCH ARTICLE | APRIL 11 2024

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AIP Conf. Proc. 3071, 020001 (2024) https://doi.org/10.1063/5.0207659





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The Effect of H₂SO₄ Concentration and Time on The Sphalerite Leaching Process

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Abstract. Galena is frequently found in association with other sulphide minerals such as sphalerite and pyrite. The pyrometallurgical methods, is commonly used for zinc recovery and requires high temperatures, resulting in heavy metal pollution and SO₂, making it unsuitable for the environment. In this study, the use of H₂SO₄ without an oxidant for direct leaching of sphalerite concentrate was investigated. The sphalerite concentrate (200 mesh) was roasted for 3 hours at 700°C to reduce the sulphur content in the sample. Following that, leaching was performed using a sulfuric acid solution with a concentration range of 1-6mol/L for 1-5 hours. The zinc levels in the leached solution were determined using XRF (X-RayFluorescence) and SEM (Scanning Electron Microscopy). Before the leaching process, the particles of zinc sulphide concentrate have a clear surface and are roughly the same shape and size. The micrographs of the leaching residues show a progressive increase in the roughness of the solid as well as an increase in the amount of elemental sulphur covering theparticle surfaces after the leaching process. The presence of sulfuric acid is clearly important in the leaching process, as zinc extraction rate increased with increasing sulfuric acid concentration, but only up to 3 mol/L. The highest zinc extraction, 99.17%, was obtained at 2 mol/L H₂SO₄ after 3 hours.

INTRODUCTION

Zinc is a nonferrous metal that is widely used, ranking third after aluminium and copper ^{1,2}. More than 70% of metallic zinc is currently produced from zinc sulphide concentrate via a series of roasting-leachingelectrowinning processes ³. However, zinc production has declined in recent years due to the difficulty of locating high-grade ore deposits, as well as technical and environmental issues associated with metal recovery from sulphide ores⁴. Slags, zinc dross, and filter cakes are by-products of zinc production and can be developed further as alternative sources of zinc oxide, despite their low content and complex composition ⁵.

Galena is frequently found in association with other sulphide minerals such as sphalerite (ZnS) and pyrite (FeS₂). Zinc exists primarily in the earth's crust as a sulphide, and sphalerite is most important ore. Flotation has been extensively used to separate galena and sphalerite. Zinc is concentrated using froth flotation methods due to the low solubility of zinc sulphides in acid media⁶. Flotation is the most common and commercial technique for processing nonferrous oxide minerals, as well as sulfidation with alkali metal sulphides with the addition of cationic collectors for zinc recovery. Because of the presence of adsorbed sulphide ions, the hydrophilicity of the mineral surface decreases after sulfidation⁷. However, it is less effective for zinc recovery by conventional flotation when the zinc is amorphous, such as zinc in lead slag smelting ¹.

The zinc sulphide concentrate is roasted in the first stage to produce acid-soluble zinc oxide^{8,9}. At this stage, sulphur dioxide (SO₂) is produced by the reaction of sulphur in the concentrate and air at temperatures above 900°C. As a result, in a recovery unit, SO₂ is produced, which is then converted to sulfuric acid. The calcine produced by high-temperature roasting of the sulphide concentrate is mostly zinc oxide, with about 4% zinc sulphate and a trace of zinc ferrite spinel mineral ^{10–13}.

One of the pyrometallurgical methods, the Waelz and Ausmelt method, is commonly used for zinc recovery and requires high temperatures (1150–1250°C) in the process, resulting in heavy metal pollution, SO₂, and waste. The method appears to be economically and technically unfeasible, particularly for low-metal materials. Meanwhile, sulfuric acid leaching is the most widely used hydrometallurgical method because it is

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effective in extracting zinc oxide materials, but it has drawbacks for materials containing silicates and other impurities. A large amount of silicate will dissolve and gel, preventing the zinc sulphate solution from separating¹⁴.

There has been a lot of interest in using an alternative oxidant to speed up the atmospheric leaching process and make it work in even milder conditions¹³. Several reagents, including ferric ion in chloride and sulphate media¹⁵, hydrogen peroxide, manganese dioxide, nitric acid, and others, have been used to leach sphalerite concentrate. Zinc dissolution from sphalerite in acidic ferric solution and acidic bacterial solution containing iron has received a lot of attention ^{16,17}. In this study, the use of sulfuric acid without an oxidant for direct leaching of sphalerite concentrate was investigated. This study aims to compare zinc recovery in sphalerite concentrate with previous study ¹³ using ozone oxidant. It can be used as a guideline for selecting reagents for leaching sphalerite concentrate on a laboratory or industrial scale.

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Materials and Methods

Sphalerite (ZnS) samples were obtained from PT. X. The zinc sulphide concentrate is dry milled in a ball mill and sieved to obtain a 200mesh size fraction. Sulfuric acid (98% purity Merck, Germany) was used as an analytical reagent in this study. In this study, sample preparation was done with a 200mesh sieve. The sphalerite concentrate was then roasted for 3 hours at 700°C to reduce the sulphur content in the sample. Following that, leaching was performedusing a sulfuric acid solution with a concentration range of 1-6mol/L for 1-5 hours. The zinc levels in the leached solution were determined using XRF (X-Ray Fluorescence) and SEM (Scanning Electron Microscopy).

Characterization

X-ray diffraction (XRD) Merk PanAnalytical, Type: E'xpert Pro Cu K α , equipment at 2 θ 5-90°. The XRD parameter was used Cu K α radiation ($\lambda = 1.5406$ Å), with a voltage of 30 kV and a current of 15 mA. XRD analysis confirmed that the main mineral in the concentrate was sphalerite (ZnS), with only minor amounts of other minerals such as quartz (SiO₂) and galena (PbS) present, as shown in Figure 1. The composition of the major elements in the concentrate was determined using X-ray Fluorescence (XRF) analysis, and the results are shown in Table 1.

TABLE 1. Composition of major elements of the concentrate				
Element	Concentration (weight %)			
Al	1.09			
S	30.52			
Zn	45.28			
Fe	8.82			
Cu	1.33			

RESULT AND DISCUSSION

The X-ray diffraction (XRD) pattern for the sphalerite concentrate is shown in Figure 1. The main compound identified was ZnS, as expected, but there were also some PbS and SiO₂ detected.



FIGURE 1. Diffractogram XRD of sphalerite concentrate

The sphalerite concentrate was then roasted for 3 hours at 700°C to reduce the sulphur content in the sample¹⁸. Temperature and time are the most important factors influencing the zinc sulphide roasting process when roasting in air and at a fixed particle size. Temperature has a greater influence on a chemically controlled process than on a diffusion-controlled reaction. Time is also an important parameter influencing the rate of oxidation and determining the capacity of the equipment. Approximately 85% of the world's zinc is produced hydrometallurgical, with the Roast-Leach-Electrowinning process dominating (RLE). The mineral sphalerite is roasted in the RLE process to make it easier to leach in sulfuric acid solution. During the roasting process, the ZnS in the concentrate is converted to zinc oxide, also known as ZnO calcine. This procedure is carried out in a 950°C furnace. The roasting reaction is an exothermic reaction that generates energy to keep the process going. This process also generates sulphur dioxide gas (SO₂), which is typically converted to sulfuric acid in a gas cleaning unit and acid plant. Chemical reactions that occurin the roasting process are indicated by reactions^{19,20}.

$$2ZnS + 3O_2 \rightarrow 2ZnO + 2SO_2 + heat$$

By varying the sulphuric acid concentration from 1 to 6 mol/L at $27\pm2^{\circ}$ C, a series experiment was carried out to evaluate the effect of acid concentration on zinc dissolution efficiency. The results, as shown in the figure 2, show that the presence of sulfuric acid is clearly important in the leaching process, as zinc extraction rate increased with increasing sulfuric acid concentration, but only up to 3 mol/L.



FIGURE 2. Effect of sulfuric acid concentration on zinc extraction

Zinc extractions gradually increase with leaching time and sulphuric acid concentration, indicating that sulphuricacid can dissolve a significant amount of zinc sulphide directly. In the absence of oxygen, the direct acid

dissolution of zinc sulphide produces H2S rather than elemental sulphur. Reactions represent the chemical reactions that occur during this process. The highest zinc extraction, 99.17%, was obtained at 2 mol/L H_2SO_4 after 3 hours.

$$2ZnS + 3O_2 \rightarrow 2ZnO + 2SO_2 + heat$$

The zinc extraction rate decreased as sulfuric acid concentrations increased. This is consistent with the results of ¹³, who used ozone as an oxidant in the leaching of sphalerite concentrate. The amount of dissolved ozone decreased as the acid concentration increased. Because sulfuric acid and ozone both play important roles in zinc dissolution, it is critical to achieve an optimal balance of acid concentration and ozone solubility to maximize leaching rate. In this study, adds information about the leaching of sphalerite concentrate without oxidants, though the time required is longer than with oxidants.

Figure 3 shows the morphology of the zinc sulphide concentrates before and after leaching using SEM. Before the leaching process, the particles of zinc sulphide concentrate have a clear surface and are roughly the same shape and size (a). The micrographs of the leaching residues show a progressive increase in the roughness of the solid as well as an increase in the amount of elemental sulphur covering the particle surfaces after the leaching process (b).



FIGURE 3. SEM images of ZnS particles (a) before leaching and (b) after leaching

CONCLUSION

The presence of sulfuric acid is clearly important in the leaching process, as zinc extraction rate increased with increasing sulfuric acid concentration, but only up to 3 mol/L. The highest zinc extraction, 99.17%, was obtained at 2mol/L H₂SO₄ after 3 hours.

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Submission date: 22-Apr-2024 03:18PM (UTC+0700) Submission ID: 2227749264 File name: 020001_1_5.0207659.pdf (1.02M) Word count: 2355 Character count: 12625 RESEARCH ARTICLE | APRIL 11 2024

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