

Leaching of a sphalerite concentrate with $\text{H}_2\text{SO}_4\text{--HNO}_3$ solutions in the presence of C_2Cl_4

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Abstract

The enhanced leaching of sphalerite concentrates in $\text{H}_2\text{SO}_4\text{--HNO}_3$ solutions and the extraction of sulfur with tetrachloroethylene were studied. Variables of the process were investigated including leaching temperature, reaction time, liquid/solid ratio, and tetrachloroethylene concentration. The number of cycles that tetrachloroethylene could be recycled did not have a significant effect on zinc extraction. The results indicated that 99.6% zinc extraction was obtained after three hours of leaching at 85 °C and 0.1 MPa O_2 , when 20 g of sphalerite concentrate were leached in a 200 ml solution containing 2.0 mol/L H_2SO_4 and 0.2 mol/L HNO_3 , in the presence of 10 ml C_2Cl_4 . Leaching rates were significantly improved under these conditions. © 2005 Elsevier B.V. All rights reserved.

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1. Introduction

Zinc is an important metal required for various applications in metallurgical, chemical, and textile industries. It is mainly recovered from primary sulfide concentrates. As the policy on discharge of sulfur dioxide becomes more and more strict, the technology of direct leaching of sulfide ores becomes more attractive. The striking advantage of direct leaching is that sulfur in the ore is converted to elemental sulfur, which remains in the leach residues, thus thoroughly eliminating discharge of sulfur dioxide into the atmosphere. However, the elemental sulfur formed during leaching

usually covers the ore particles, which presents a major obstacle for effective leaching of the metals in sulfide ores (Jin zuomei et al., 1984; Dutrizac, 1990; Dutrizac and Chen, 1990; Lin et al., 1991; Bobeck and Su, 1985; Crundwell, 1987; Cheng et al., 1994; Lochmann and Pedlik, 1995).

Crundwell (1987) leached sphalerite concentrate in a solution of ferric iron and sulfuric acid. Surface reaction control model (Eq. (1)) was applied to explain

$$K't = 1 - (1 - \alpha)^{1/3} \quad (1)$$

the experimental data but the linear dependence deviated at 30 min of leaching (at about 70% conversion) and became parabolic (K' is the rate constant, t is time, α is conversion). The deviation was explained by product-layer diffusion.

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Bobeck and Su (1985) leached sphalerite in ferric chloride solution. The results indicated that during the initial stages of leaching, the process was controlled predominantly by the rate of the reaction of ferric ion with the sulfide at the sulfur–sulfide interface. Transport of ions across the product sulfur layer was found to be important during the later stages of reaction.

Cheng et al. (1994) leached zinc sulfide concentrate in a solution of ferric sulfate, sulfuric acid and sodium chloride. The shrinking core model was used to describe the dissolution reaction, limited by electrochemical processes in the beginning of the leaching, but later on, by surface reaction and pore diffusion. The experimental data followed Eq. (1) until 60–70% conversions, after which sulfur formation was stated to control the reaction.

Lochmann and Pedlik (1995) leached sphalerite concentrates in acidic ferric sulfate solution. A product layer was found to limit the extraction, and to reveal the character of the product, the passivated concentrate was washed to dissolve anglerite, jarosite or elemental sulfur. As the washed concentrate was re-leached, an improved extraction was noticed just in the case where elemental sulfur was removed. It was concluded that a layer of elemental sulfur controlled the dissolution rate.

The use of nitric and mixed nitric–sulfuric acids as a hydrometallurgical process leach medium has been known for some time (Prater et al., 1973; Bjorling, 1976; Ouellet et al., 1975; Queneau and Prater, 1974). In these processes, the oxidant is supplied by the breakdown of nitrate ion in acid solution. These processes are characterized by easy regeneration of lixivants using available technology and partial conversion of sulfide sulfur to elemental sulfur.

To enhance leaching, increasing the intensity of stirring, controlling the temperature of leaching, adding catalytic agents or using surface active reagents have been resorted to (Venkataswamy and Khangaonkar, 1981; Bobeck and Su, 1985; Dutrizac, 1989; Balaz and Ebert, 1991; Lochmann and Pedlik, 1995; Maurice

and Hawk, 1998, 1999; Doyle et al., 1978; Parker, 1981; Kawulka et al., 1975), but the results are by no means ideal. Venkataswamy and Khangaonkar (1981) reported the leaching of sphalerite by ferric chloride and the effect of an organic solvent for sulfur in order to improve the efficiency of the process. Leaching experiments were conducted at boiling temperatures (106 ± 1 °C). The results indicated that for a 6 h leach a Zn recovery of over 95% could be obtained (instead of about 68% without the aid of solvent).

Forward and Veltman first successfully carried out oxidation pressure leaching of zinc sulfide concentrates in sulfuric acid solutions for Sherrit in 1959. Originally, the leach temperature was restricted to below the melting point of sulfur, because molten sulfur would encapsulate unreacted concentrate and severely limit extraction. Subsequently, research of Kawulka et al. (1975) discovered that certain surface-active reagents inhibited the molten sulfur from occluding the unreacted sulfide particles and allowed leaching to be conducted at a temperature significantly above the melting point of sulfur, thus eliminating the need for separation and recycle of unleached sulfide particles. Typical additives were lignin sulphonate and quebracho; zinc extractions increased to 96–98%.

Surfactants were used in commercial zinc sulfide pressure leaching to prevent occlusion of unleached particles by liquid sulfur (Doyle et al., 1978; Parker, 1981), which permitted the use of higher leach temperatures, with corresponding enhanced reaction rates.

According to the above established technology, the authors carried out oxidation pressure leaching of sphalerite concentrates with $\text{H}_2\text{SO}_4\text{--HNO}_3$ solutions in the presence of C_2Cl_4 at 85 °C and 0.1 MPa O_2 . The objective of this work was to reveal that C_2Cl_4 extracted the elemental sulfur formed during the leach in the temperature below the melting point of sulfur, which greatly enhanced reaction rates. High quality sulfur was recovered by cooling the organic phase containing sulfur.

2. Experimental

2.1. Materials and reagents

In the present experiment, sphalerite concentrate having the chemical composition shown in Table 1 was first dried and then ground in a ball mill to below 74 μm . X-ray diffraction studies indicated the presence of sphalerite as the major zinc-containing phase along with pyrite and chalcopyrite in minor quantities. The percent contents of these minerals in the concentrate were: sphalerite (74.7%), pyrite (5.7%), and chalcopyrite (2.3%). Sulphuric acid, nitric acid and tetrachloroethylene were all analytical grade.

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