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Effect of iron in zinc silicate concentrate on leaching with sulphuric acid

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ABSTRACT

It is shown that the iron content in zinc silicate concentrates with either high (8–11%) or low (3%) iron does not significantly affect the kinetics or overall recovery of zinc extraction in sulphuric acid. Most of the iron was present as hematite and franklinite with little iron contained in willemite. A small reduction in zinc recovery from 98.5% to 97.5% was observed for silicate ores containing 12% iron. The activation energy determined from high-iron concentrate leaching, 78 ± 12 kJ/mol, is statistically similar to that from low-iron concentrate, 67 ± 10 kJ/mol, suggesting the same rate-controlling step. The leaching of high high-iron concentrates enables a higher mass recovery during flotation. A flowsheet is proposed comprising a magnetic separation step to produce a magnetic and a non-magnetic product so that iron dissolution from the magnetic concentrate acts as a source of soluble iron for impurities removal.

1. Introduction

The roasting of zinc sulphide concentrates produces zinc, iron and other metal oxides (known as calcine), which are readily leached in sulphuric acid solutions (Çopur et al., 2004), with zinc ferrites one of the major species in the leaching residues (Youcai and Stanforth, 2000). These ferrites can be very refractory to chemical attack and one method for their zinc recovery is to leach these residues with hot, concentrated sulphuric acid solutions, although this step will always dissolve a considerable amount of iron. This also requires a large quantity of acid during leaching and then a series of downstream iron and impurity metal removal steps (Youcai and Stanforth, 2000). Several processes to remove dissolved iron have been applied at zinc industries, such as the jarosite [$XFe_3(SO_4)_2(OH)_6$], goethite (FeOOH), hematite (Fe_2O_3) and paragoethite (ferrihydrite) processes; each of them having its own advantages and disadvantages (Pappu et al., 2006). Notwithstanding, a small iron concentration in the zinc process is beneficial. Raghavan et al. (1998) have proposed that there are two major steps to remove impurities from the zinc sulphate solution to those levels required for the electrolyte. The first stage takes place in the neutral leaching step where co-precipitation of several deleterious impurities such as antimony, arsenic and germanium occurs, along with that of iron hydroxide (Eq. (1). The second step comprises cementation with zinc dust.

$$2FeSO_4 + 3ZnO + MnO_2 + 2H_2SO_4 + H_2O \rightarrow 2Fe(OH)_3 + 3ZnSO_4 + MnSO_4 \quad (1)$$

In the case of silicate concentrates, Souza (2000) has devised an integrated process to treat zinc silicate concentrates in the same plant that processes zinc sulphide concentrates by the RLE process (the integrated process). Among the different options available, the author has suggested only one step of zinc silicate leaching where stepwise addition of sulphuric acid dissolves the silicate with a minimum silica gel formation. The neutralisation of the residual acidity with lime or limestone to pH 4.0 provides good settling and filtration properties of the leaching residue. This leaching approach, industrially applied at the Três Marias Zinc facility, presents high zinc recovery (>98%), treating 350,000 tonnes/year of zinc silicate concentrate (Brook-Hunt, 2005). Although there is no reference to the iron influence during leaching in the integrated process, the interest for the hydrometallurgical processing of high high-iron silicate concentrates appeared when the Vazante Mine in Brazil noticed the occurrence of high-iron silicate ores that would be submitted to the flotation step, which precedes the hydrometallurgical treatment.

The purpose of the present work is to examine the effect of the iron content in the concentrates on both zinc dissolution kinetics and recovery. A new treatment flowsheet is proposed using the iron content in the concentrate to eliminate impurities.

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2. Materials and methods

The chemical analysis of both the high- and low-iron flotation concentrates is presented in Table 1. The high-iron concentrate contains 34–39% Zn and 8–11% Fe, whereas the low-iron sample has more zinc (46%) and less iron (~3%). Prior to the leaching experiments, these concentrates were dry ground and wet sieved to yield a particle size distribution between 150 and 38 µm. Zinc and iron contents, surface area, total porous volume and average pore diameter of the sieved fractions are also presented in Table 1.

For the kinetics study, chemical leaching experiments were carried out batch-wise with 10 g/L solids in 500 mL solution in a closed water-jacketed borosilicate glass reactor (750 mL total volume) agitated by a magnetic stirrer. This enabled adequate dispersion of the mineral particles without evaporation loss of the solution. Leach solutions were prepared using reagent grade H_2SO_4 and distilled water. At selected time intervals, a known amount (3 mL) of slurry was withdrawn, filtered and analysed for zinc in solution (Atomic Absorption Spectrometry, Perkin Elmer AAnalyst 100) to determine zinc extraction. The volume change was taken into account for the zinc extraction determinations.

Batch leaching experiments under industrial conditions (150 g/L solids; 70 ± 2 °C; 7 h retention time; 35 g/L final acid concentration and 2.75 L total volume) were also carried out to determine zinc extraction from three different zinc flotation concentrates, assaying 5%, 9% and 12% iron. These experiments aimed to confirm those results achieved during the kinetic studies with low solid concentrations and pulp volume.

Surface area and pore volume were determined by nitrogen adsorption. Nitrogen isotherms were performed with a Nova 1000 High Speed Gas Sorption Analyzer (*Quantachrome*). Sample degassing was carried out at 80 °C, for 24 h, to avoid decomposition. Nitrogen adsorption was performed at −196 °C. Data were collected from a relative pressure (p/p_0) of 0.05 to 0.98. A large sample (~4.0 g) was used and the Nova 1000 parameters (equilibration tolerance, time to remain in tolerance and maximum equilibration time) were set at 0.05, 360 and 720, respectively, to improve the accuracy of low surface area measurements with nitrogen adsorption.

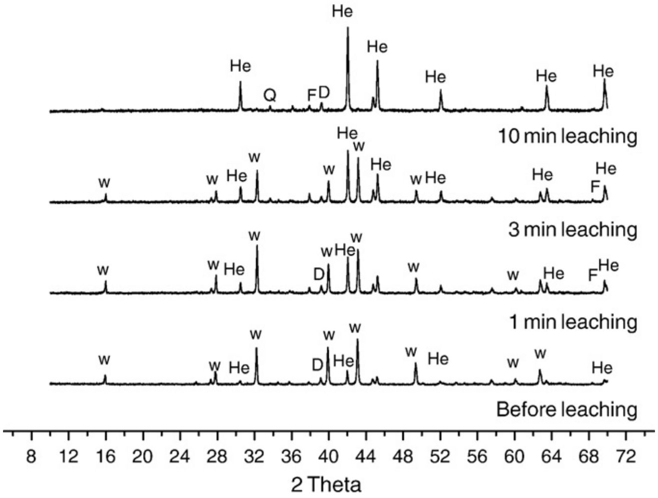

Fig. 1. XRD pattern of the high-iron zinc silicate concentrate. Q: quartz, He: hematite, w: willemite, F: franklinite, D: dolomite.

X-ray diffraction was carried out on a Shimadzu 600 diffractometer equipped with an iron tube and graphite monochromator. Willemite was identified as the main mineral phase; with quartz, hematite and hemimorphite also present as minor phases in the low-iron concentrate; while franklinite and dolomite, but not hemimorphite, were observed in the high-iron concentrate. Therefore zinc in this concentrate is associated with willemite and franklinite (a minor species) (Figs. 1 and 2). Quantitative mineralogy was carried out by transmitted light microscopy (Leica).

The analyses of both concentrates and leach residues were also carried out by SEM-EDS. The samples were coated with graphite by electro-deposition, using a Jeol JEE 4C instrument and observed in a JEOL JSM 5510 scanning electron microscope (SEM), equipped with a spectrometer for micro-analysis, based on an Energy Dispersive X-ray Spectroscopy System (EDS) and having an accelerating voltage of 0.5–30 kV. Electron microprobe analysis confirmed willemite as the main zinc mineral since the metal content of different grains is similar to that of a pure mineral (theoretical, Table 2 and Fig. 3). Iron is not present in the willemite structure, as shown in Fig. 3(b).

3. Results and discussion

3.1. Effect of leaching parameters

The effect of the agitation speed on zinc extraction for the high- and low-iron concentrates was assessed in the range 360–720 rpm. The stirring rate did not affect the zinc extraction regardless of the iron content in both concentrates. Therefore, the dissolution process did not seem to be controlled by mass transfer through the liquid boundary film, despite the possible change in solution viscosity caused by silica

Table 1
Chemical analysis (Zn and Fe) and surface parameters of different screened fractions of low- and high-iron zinc silicate concentrates

		Unit	150–105 µm	105–75 µm	75–53 µm	53–45 µm	45–38 µm
High-iron silicate concentrate	Zn	(%)	39.4	39.7	35.6	35.2	34.1
	Fe	(%)	8.0	9.8	11.7	10.3	11.1
	SiO_2	(%)	25.1	24.3	23.8	23.2	22.9
	Surface area	m^2/g	1.2	1.0	0.6	0.6	1.0
	Total porous volume	mm^3/g	3.6	3.3	2.5	2.5	2.5
	Pore average diameter	Nm	12.1	13.1	17.9	18.0	9.8
Low-iron silicate concentrate	Zn	(%)	46.9	46.0	46.9	46.6	47.3
	Fe	(%)	3.74	2.92	3.19	3.02	3.25
	SiO_2	(%)	29.4	29.5	29.6	30.1	25.9
	Surface area	m^2/g	0.8	0.5	0.5	0.5	0.7
	Total porous volume	mm^3/g	3.5	1.4	1.3	2.1	1.3
	Pore average diameter	Nm	17.6	11.6	10.8	15.8	7.4

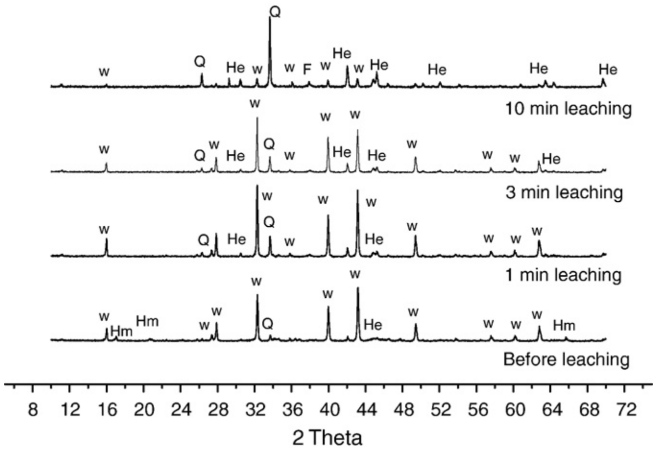

Fig. 2. XRD pattern of the low-iron zinc silicate concentrate. Q: quartz, He: hematite, w: willemite, F: franklinite, Hm: hemimorphite.