SELECTIVE SOLVOMETALLURGICAL LEACHING OF LEAD AND ZINC FROM JAROSITE RESIDUES FROM THE ZINC INDUSTRY

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Abstract

The relatively new branch in extractive metallurgy called solvometallurgy was investigated for selective leaching of Pb and Zn from iron-rich jarosite residues, which are typically landfilled. After screening of several lixiviants, the ionic liquids [A336][Cl] and [C101][Cl], equilibrated with HCl, leached the most Pb and Zn of the ionic liquids investigated. When the ionic liquids were equilibrated with relatively lower HCl concentrations, the dissolution of Fe decreased drastically and, thereby, the selectivity for Pb and Zn improved. [A336][Cl] equilibrated with 0.5 M HCl leached 62 wt% Pb, 27 wt% Zn and 7 wt% Fe, while [C101][Cl] equilibrated with 0.5 M HCl leached 73 wt% Pb, 31 wt% Zn and 10 wt% Fe.

Introduction

The extractive metallurgy of industrial process residues is complex because the relevant metals are present in low concentrations, and often locked in complex matrices (sulphides, oxides, phosphates or silicates).[1] As a result, it is difficult to recover these metals. Most hydrometallurgical leaching processes suffer from substantial acid consumption and poor selectivity, resulting in leachates with high concentrations of impurities. However, by replacing the aqueous phase in hydrometallurgical processes by organic solvents with dissolved reagents, it is possible to attain high selectivity and reactivity because non-hydrated anions have a greater affinity to bind to metal ions. This approach to extractive metallurgy, based on the use of organic solvents instead of an aqueous phase, is called "solvometallurgy".[1] In this work, solvometallurgical leaching was carried out on industrial jarosite residue samples. The residue was produced as a by-product during purification and refining of zinc from its sulphide ore (ZnS, sphalerite). Due to its high rate of generation and relatively high Pb and Zn content, jarosite could be an important resource for these metals.[2]

Results and discussion

The jarosite sample was dried and milled to a fine powder of a size smaller than 1.65 µm (D90). Powder-XRD analysis revealed that Pb occurs in the form of anglesite ($PbSO_4$) and Zn in the form of sphalerite (ZnS). In order to determine the elemental composition, the two samples of the material were completely dissolved in acid using a microwave digester. The digested acidic solutions were analysed by both TXRF and ICP-OES. The analysis results of duplicates and the two techniques were comparable. The results are shown in Table 1. Solvometallurgical leaching of jarosite was investigated to leach Pb and Zn. A series of 14 lixiviants were screened to select the best candidate (Table 2). The lixiviants tested include organic acids, acidic extractants, basic extractants, neutral extractants, and HCl dissolved in alcohols and ionic liquids. The highest amounts of Pb and Zn were leached by the quaternary ammonium chloride ionic liquid Aliquat® 336 ([A336][Cl]) and the quaternary phosphonium chloride ionic liquid Cyphos® 101 IL ([C101][Cl]) after their equilibration with aqueous solutions of HCl.

Table 1: Elemental composition of the jarosite residue

	Fe	Pb	Ca	Zn
Concentration [g/kg]	175	40	25	24

Table 2: Concentrations of Pb, Zn and Fe in the leachates

No.	Lixiviant	Pb [mg/L]	Zn [mg/L]	Fe [mg/L]
1	26.5 M formic acid	0	110	2,101
2	17.5 M acetic acid	0	15	934
3	2.5 M Versatic acid	0	0.4	3
4	1.4 M di-(2-ethylhexyl)phosphoric acid (D2EHPA)	0	29	580
5	Cyanex 272 (equilibrated with water)	0	134	150
6	Cyanex 272 (unequilibrated)	0	37	90
7	[A336][NO3] (equilibrated with 5 M HNO3)	0	24	2
8	[C101][NO3] (equilibrated with 5 M HNO3)	0	157	207
9	Ethanol	0	67	639
10	1.2 M HCl in ethanol	11	688	3,450
11	5.4 M HCl in 1-octanol	117	660	11,000
12	TBP equilibrated with 12 M HCl	166	928	15,757
13	[A336][Cl] equilibrated with 12 M HCl	722	952	12,978
14	[C101][Cl] equilibrated with 12 M HCl	2,615	1,316	14,272

Leaching parameters: Stirring 2 hours, 60°C, 2,000 rpm, S/L ratio 1:10)

Next, the effect of different HCl concentrations in [A336][Cl] and [C101][Cl] were investigated. The leaching efficiency of Pb, Zn, and Fe increased with increasing HCl concentration in the ionic liquid (Figure 1).

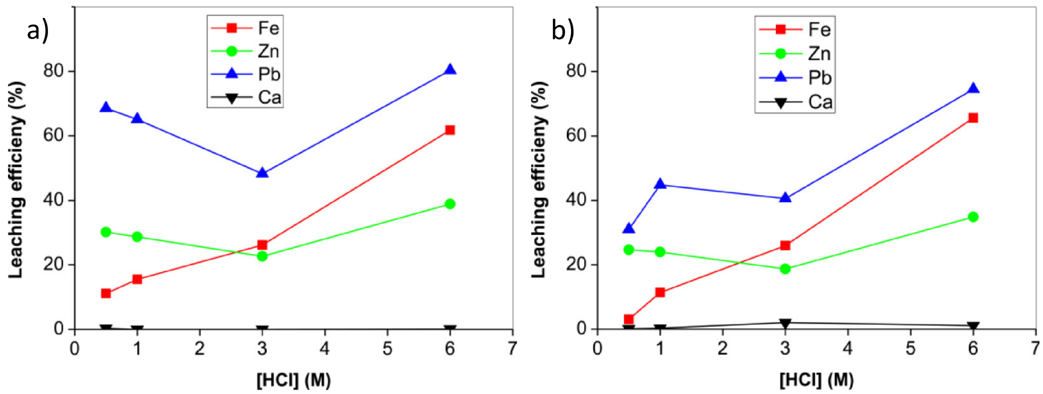

Figure 1: Leaching efficiency with a) [C101][Cl] and b) [A336][Cl], equilibrated with different HCl concentrations. Leaching parameters: contact time 2 hr, 60°C, 2000 rpm, S/L ratio 1:10

The leaching efficiencies of [C101][Cl] equilibrated with 0.5 M HCl were 68% Pb, 30% Zn and 11% Fe and they increased to 80%, 39% and 61%, respectively, with the ionic liquid equilibrated with 6 M HCl. Similarly, for [A336][Cl], the leaching efficiencies increased from 31% Pb, 25% Zn and 3% Fe to 74% Pb, 34% Zn and 66% Fe when the HCl concentration used for equilibration was increased from 0.5 to 6 M. Although equilibrating the ionic liquids with 6 M HCl leached more Pb and Zn, it also leached more Fe. The co-dissolution of Fe interferes in the downstream processes of the metal recovery. Therefore, ionic liquids equilibrated with 0.5 M HCl are preferred due to their better selectivity against Fe. Consequently, the ionic liquids equilibrated with 0.5 M HCl were chosen as the lixiviants for further studies on the leaching process, resulting in the following optimised parameters values: residence time 2 h, temperature 45°C, solid/liquid ratio 1:15 and stirring speed 1,500 rpm (Table 2).

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Table 3: Leaching efficiencies for jarosite at the optimised conditions

Lixiviant	Pb [%]	Zn [%]	Fe [%]
[A336][Cl] eq. with 0.5 M HCl	62	27	7
[C101][Cl] eq. with 0.5 M HCl	73	31	10

Conditions: contact time: 2 hrs, temperature: 45°C, Solid/liquid ratio: 1:15, and stirring speed: 1,500 rpm

Conclusion and future work

The presence of HCl in the lixiviant is critical for leaching Pb from jarosite. All the lixiviants without HCl did not leach any Pb. [A336][Cl] and [C101][Cl] equilibrated with HCl leached the highest amount of Pb and Zn. The selectivity is strongly influenced by the concentration of HCl used for equilibrating the ionic liquid. The selective leaching of Pb (62–73%) and Zn (27–31%) against Fe (7–10%) from the studied jarosite residue took place with [A336][Cl] and [C101][Cl] after equilibration with 0.5 M HCl. When the ionic liquids were equilibrated with higher concentrations of HCl, the Fe dissolution increased more than the valuable metal dissolutions and thus resulted in a poor selectivity. Presently, work is on-going to up-scale the leaching experiments on the jarosite residues from milligram to gram scale. Next, the recovery of the metals from the leachate will be investigated by non-aqueous solvent extraction or with non-aqueous ion exchange.

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