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Separation of zinc from manganese, magnesium, calcium and cadmium using batch countercurrent extraction simulation followed by scrubbing and stripping

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ABSTRACT

A systematic study for the separation of zinc from solutions containing zinc, manganese, iron, cadmium and magnesium as sulfates was developed after a batch countercurrent extraction, followed by scrubbing and stripping. In the first step, batch countercurrent experiments were carried out on a synthetic solution containing 14.60 g/L Zn(II), 6.4 g/L Mn(II), 2 g/L Mg(II), 819 mg/L Ca(II) and 60 mg/L Fe(II). The results of this step showed that at a three-stage extraction and six cycles under the conditions being O/A ratio of 1:1 and temperature of 40 °C, 13.06 g/L equal to 89.50% of zinc was extracted by D2EHPA whereas only 83 mg/L of manganese was loaded by the extractant. As a result, the low extraction percentage of manganese is an attractive advantage of this process. In comparison to the literature, the high separation factor of zinc and manganese (i.e. 643.79) of this study was obtained without any pH adjustment or using any synergetic mixture. To remove the trace concentrations of cadmium, calcium, manganese and iron from the loaded organic solution, a series of scrubbing experiments was conducted. With regard to the scrubbing results, a solution with a composition containing 6 g/L Zn and 18 g/L H₂SO₄ could scrub trace concentrations of metals from the loaded organic solution. Finally, the McCabe–Thiele diagram of the stripping stage predicted that at an O/A phase ratio of 2:1, two countercurrent stages are sufficient to transfer zinc from the organic solution to the aqueous solution. The obtained aqueous solution contained 88.8 g/L of Zn and the impurities were under the limited concentrations of electrowinning.

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1. Introduction

Zinc solvent extraction (SX) has a long perspective. Many studies have been carried out on zinc solvent extraction. One of the primary researches was the solvent extraction of zinc with dithizone published in 1958 (Schweitzer and Honaker, 1958). Since this year and possibly before that, many researches and industrial works have been performed in the field of zinc solvent extraction. Thus, in 2015, providing something new in the field of zinc solvent extraction may be difficult. Most of the current zinc SX works have focused on developing a process to recover zinc from various pregnant solutions obtained from chemical processes. For instance, Fleitlikh et al. (2014), Lum et al. (2014), Baba and Adekola (2013), Innocenzi and Veglio (2012) and Baba and Adekola (2011) have developed processes for the recovery of zinc from various liquors via solvent extraction steps. Therefore, developing a solvent extraction process for the recovery and separation of zinc from various impurities coming from leaching of specific resources still has novelty.

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Separation of zinc from various impurities using a synergetic mixture of extractants has been investigated by many researchers. For instance, the synergetic effects of both trioctylphosphine oxide (TOPO) and tributyl phosphine (TBP) (Hu et al., 2013), β -diketone and TOPO (Chen et al., 2011), TBP and bis(2,4,4-trimethylpentyl)phosphinic acid (Baba and Adekola, 2013), lonquest 801 and D2EHPA (Zhu and Cheng, 2012), and LIX63, Versatic10 and TBP (Zhu et al., 2012) have been investigated. The mentioned methods cannot often be industrialized due to the technical issues of the extractant mixture, unavailability of extractants and their high cost. Therefore, the development of a simpler process (instead of the mentioned methods) can be valuable. Batch simulation of a countercurrent extraction was presented by

Bush and Densen (1948) and developed by Treybal (1963) and Baird (1993). This simulation is usually used as a laboratory procedure and can be carried out in various ways. In this approach, the loaded organic solvents and the raffinate solutions leave "the opposite ends of the cascade in ratios different from that in the feed and in amounts varying with time" (Treybal, 1963). Using this approach, the simulation of an actual countercurrent extraction in a laboratory scale is being made possible. In addition, various parameters and conditions of a countercurrent extraction such as maximum loading of the organic solution, the extraction conditions and the concentration of desired and undesired







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10 Table 1

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The compositions of the solution used in the solvent extraction stu	.udv.

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Element	Zn (g/L)	Mn (g/L)	Mg (g/L)	Ca (mg/L)	Co (mg/L)	Ni (mg/L)	Cd (mg/L)	Fe (mg/L)
Concentration	14.60	6.40	2.00	819	0	0	51.1	<60.0

elements in the organic solution and raffinate can be found in this series of experiments. A number of studies have applied such an approach to investigate a SX system. Lee et al. (2002) used this approach to investigate "solvent extraction separation of indium and gallium from sulphate solutions using D2EHPA". They applied a batch simulation of a threestage countercurrent extraction to reach their aim. The copper extraction using LIX 84I has also been investigated using the mentioned method by Jena et al. (2002). Banda et al. (2012) employed TOPO and Alamine 308 to separate and recover Mo and Co from the chloride leach solution obtained from the petroleum refining catalyst. They used a batch simulation of countercurrent extraction experiments to evaluate the conditions of their system. "Evaluation of a versatic 10 Acid/Nicksyn™ synergistic system for the recovery of nickel and cobalt from typical lateritic leach liquor" has been studied using a batch simulation of a countercurrent extraction (Preez and Kotze, 2013). In this research, the countercurrent extraction was performed in three stages. "Batch simulation of multistage countercurrent extraction of uranium in yellow cake from monazite processing with 5% TBP/kerosene" was recently investigated (Injarean et al., 2014). In this study, batch simulation experiments were conducted in a 5-stage countercurrent extraction. Selective extractions of Zn and Cd from Zn-Cd-Co-Ni sulfate solution using di-2-ethylhexyl phosphoric acid extractant have been investigated by batch countercurrent experiments (Owusu, 1998). In this study, simulation of a 3stage countercurrent operation with five cycles was used for the recovery of zinc. In addition, this solvent extraction study has investigated the potential selective extraction of both Zn and Cd from Zn-Cd-Co-Ni sulfate solution.

As previously discussed, there is a dearth of literature on zinc SX using the batch countercurrent extraction simulation. Therefore, in the present study, separation of zinc from manganese, magnesium, calcium and cadmium solutions using batch countercurrent extraction simulation followed by scrubbing and stripping was carried out to provide a suitable pregnant solution for zinc electrowinning.

This study was carried out to develop a process for the recovery of zinc from a simulated zinc pregnant solution with a composition similar to that of leach solution obtained from a complex zinc ore with high manganese.

2. Experiment and procedure

2.1. Materials

Synthetic leach liquor was prepared to simulate the sulfate leach liquor of Mehdi Abad, Iran zinc ore by dissolving analytical grade sulfates of iron, cadmium, calcium, nickel, magnesium, zinc, and manganese in distilled water with the pH adjusted to 4.2 by adding 1 M H₂SO₄. The compositions of the solution were shown in Table 1. The organic solutions were prepared on a volume basis (30% D2EHPA and 70% Irannol kerosene). No reagents were used to adjust the pH due to the spontaneous nature of the equilibrium state. Before the experiments, the oxidation potential was controlled by an Eh-meter. With respect to the EhpH diagram for a system containing Zn, Mn, Fe, Mg, Ca and Cd constructed by HSC 5.1 software (Fig. 1), in the water stability region, the manganese and iron species are in the forms of Mn(II) and Fe(II) in major areas. Furthermore, the potential of the synthetic solution was controlled to be in the mentioned area. Insignificant extraction of iron by D2EHPA (below 3%) also confirmed that the iron existed in the form of ferrous ions. In addition, the valences of species were confirmed by the analytical methods in the laboratory.

2.2. Experimental procedures

In order to design the structure of the batch countercurrent extraction experiments, the following steps were followed:

- Obtaining the number of extraction stages and the O/A phase ratio using construction of the extraction isotherm and McCabe–Thiele diagrams.
- (ii) Designing the batch countercurrent experiments regarding the number of stages obtained from the previous step.

All the experiments were carried out in duplicate and 95% confidence interval (CI) was applied to estimate the experimental error bar.



Fig. 1. Eh-pH diagram of the synthetic solution of this study constructed by HSC 5.1.

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