

Performance and selectivity of the new column flotoextraction method



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ABSTRACT

The main difference between the new Column Flotoextraction (CFE) method and the conventional SX and SPRAY methods is the substitution of organic phase bubble dispersion, instead of its drop dispersion into the aqueous phase. This substitution is intended to increase the contact area of phases and to enhance the buoyancy force of the organic phase. The main aims of this study are the introduction of the CFE method and the comparison of its performance and selectivity with SX and SPRAY methods. The results indicated ~10% and ~20% increase of cobalt (Co) extraction from dilute (100 mg Co/l) synthetic solution in A/O = 40 and ~1% and ~6% increase of Co extraction from concentrated (1000 mg Co/l) synthetic solution in A/O = 30 for the CFE method compared to SX and SPRAY methods, respectively, due to improved contact surface of the phases. In the same extraction time (1 min), the increase of Co extraction from dilute and concentrated solutions in the CFE method compared to the SX method were ~16% and ~4%, which demonstrate improved kinetics of the extraction process in CFE method. Improvement of separation factors in the CFE method relative to SX and SPRAY methods warrants the better performance of this innovative method.

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1. Introduction

Solvent extraction (SX) or liquid–liquid extraction is one of the most important hydrometallurgical methods for selective removal, purification and concentration of valuable ions from aqueous solutions using organic solvents. Successful performance of this method in mineral processing requires clear and concentrated pregnant solutions from ore leaching (Habashi, 1999). Reduced concentration of the solution demands increased agitation of the two phases and increased interfacial area between them in order to reach a suitable rate of mass transfer. Therefore, the power consumption would be high for a certain extent of extraction. In addition, formation of stable emulsions due to vigorous agitation would increase separation time and loss of organic phase due to entrainment. On the other hand, the increased volume of the organic phase for enhanced collision probability of trace metal ions with extractant agent and improved extraction kinetics would increase operating costs. Subsequently, this increase of consumption can be followed by environmental problems due to an increase of organic phase loss (Rydberg et al., 2004).

Despite these limitations, two points are important to note. Firstly, continuous depletion of mineral deposits coupled with low grades of the minerals increase the operating costs in the production of concentrated pregnant solutions for extraction contractors. Secondly, the current environmental concern in the world is the increasing volume of dilute solutions containing valuable ions and heavy metals with the development of various industries such as mining, and the high costs for the removal of their environmental problems. Improved performance of extraction equipment for dilute solutions not only expands the usable reserves, but may also enable the use of wastewaters as new sources of feed solution to the SX process. This technological development is valuable for effective recovery of valuable ions from dilute solutions or elimination of environmental problems of toxic and heavy metals in wastewaters.

Research efforts conducted to achieve this important aim have led to the invention of various extraction technologies such as Predispersed Solvent Extraction (PDSE) (Tarkan and Finch, 2005), froth flotoextraction (Sebba, 1987), solvent extraction with bottom gas injection without moving parts (Dibrov et al., 1998; Sohn and Doungeethaveeratana, 1998), liquid membranes, nondispersive solvent extraction, microemulsions and reverse micelles (Rydberg et al., 2004), Air-Assisted Solvent Extraction (AASX) (Doungeethaveeratana and Sohn, 1998; Tarkan and Finch, 2006), Compressed Air-Assisted Solvent Extraction (CASX)

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(Li et al., 2008), etc. Enabling the satisfactory disengagement of phases, reducing the process time and increasing the extraction in high A/O ratios are the main objectives of the approaches proposed in these studies.

Consistent with these technological developments, the new Dissolved Nitrogen Predispersed Solvent Extraction (DNPDSE) method was developed. The mixing operation of the two phases in this method was based on organic phase bubble dispersion, instead of its drop dispersion into the aqueous phase. This substitution was aimed at increasing the contact area of the two phases (to improve recovery of metal ions from dilute solutions) and to enhance the buoyancy force of the organic phase (to improve separation of the two phases). In this method, solvent-coated nitrogen bubbles smaller than 100 µm were produced during along and multi-step preparation process by applying a system under pressure similar to the Dissolved Air Flotation (DAF) method (Tavakoli Mohammadi et al., 2013a,b). The Column Flotoextraction (CFE) method is the modified DNPDSE method aiming to improve system performance (reducing the period of extraction process), better separating the two phases, facilitating the extraction process and improving the safety (removing the system under pressure of organic compounds) by making changes in the production system of solvent-coated bubbles. In this method, by applying the pumps and spargers similar to the downcomer of the Jameson Cell in the contactor structure, instead of the system under pressure in the DNPDSE contactor as well as production of solvent-coated air bubbles in a size range of 100–600 µm, the main inadequacies of the DNPDSE method have been overcome.

In this study, major and minor aims have been pursued. The main aim of this research is introducing the new method of CFE and comparing its performance and selectivity with SX and SPRAY methods. The term SX method in this paper refers to conventional bulk mixing to the organic and aqueous phases. In order to eliminate the effect of unknown causes and chemical compounds in the solutions and real wastewaters on the extraction results, synthetic aqueous solutions have been used. To select the major and associated elements in these solutions, environmental problems of mines in Iran were addressed. The environmental study of zinc (Zn) production plants showed that the solid residues of leaching, neutralization, hot purification, cold purification and electrowinning processes in these plants contain valuable metals such as Zn, cobalt (Co), and manganese (Mn). These residues have been accumulated and stored in tailings dams due to lack of appropriate extraction technologies, and have caused environmental problems over time. Taking into account the high economic value of these metals and the mentioned reasons, the need to devise methods for extracting metals from these residues with maximum efficiency, based on modern methods with minimum environmental impacts is of the utmost importance. The secondary aim of this study is to investigate the potential of the new CFE method in extraction of valuable elements from these residues by examining synthetic solutions containing similar elements. Due to the high economic value (even in low concentration), Co has been selected as the major element, with Zn and Mn as associated elements in the synthetic solution.

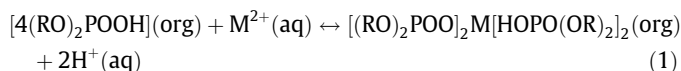
2. Materials and methods

2.1. Materials

Reagent-grade $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ (Fluka, Switzerland), $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (Fluka, Switzerland), $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ (Merck, Germany) and Na_2SO_4 (Merck, Germany) were used to prepare the synthetic aqueous solutions. To evaluate the surfactant effect of dilute silicone oil (Shin Etsu, Japan) on the SX method, a synthetic aqueous solution containing 500 mg Co/l (2381.4 mg $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ /l) (the intermediate

concentration of dilute and concentrated solutions) and 9600 mg Na_2SO_4 /l in the extraction stage as well as a synthetic aqueous solution containing 100 g H_2SO_4 /l were used in the stripping stage of the pregnant organic phase, respectively. Na_2SO_4 reagent was used to remove the effect of ionic strength on extraction results, and its concentration was considered twice the ionic strength due to the presence of selected elements in the synthetic solution. Characteristics of the synthetic aqueous solutions used to evaluate the extraction performance and selectivity of SX, SPRAY and CFE methods are presented in Tables 1 and 2, respectively.

According to Table 2, to provide the same conditions for extraction of the three elements and enable a correct selectivity comparison of the three methods, a similar concentration of elements in the synthetic solution was used. Dilute sulfuric acid (Merck, Germany) and ammonia (Merck, Germany) solutions were used for pH adjustment of the synthetic solution. The organic phase was a mixture of 10% v/v chelating type extractant D2EHPA (Cognis), 5% v/v modifier TBP and 85% v/v diluent kerosene. The extraction mechanisms of D2EHPA with Co, Mn and Zn cations (M^{2+}), which leads to the transfer of their complexes from the aqueous phase (at their extraction pH) to the organic phase, are as follows (Rydberg et al., 2004):



In the CFE method, 11.4 g/l dilute silicone oil was used in the organic phase to improve its foaming capacity to produce bubbles (Tarkan and Finch, 2005).

2.2. Methods

2.2.1. Foam stability measurement method

To convert the organic phase into bubbles in the CFE contactor, the best conditions should be provided for its foaming. For this purpose, the first step is to select the appropriate surfactant and determine its optimum concentration to get the highest quantity of foam from the organic phase (Tarkan and Finch, 2005). The experimental set-up of the foam meter is shown in Fig. 1.

The Baykerman method (Sebba, 1987) was used to measure the foam stability. The organic phase (5 ml) containing different amounts of silicone oil was transferred into the cylindrical part, and the air required to produce the solvent-coated bubbles was generated using a compressor with a constant flow rate of 200 ml/min. The air enabled foam production by passing through a sparger in the bottom part of the foam meter. The total height of foam and the organic phase (H) for each experiment was measured after a steady state was achieved after 2 min. The foaminess unit for the organic phase was calculated using Eqs. (2)–(4):

$$H_f = H - H_0 \quad (2)$$

$$V = \pi r^2 H_f \quad (3)$$

$$\Sigma = V/Q \quad (4)$$

Table 1

Specifications of synthetic aqueous solutions used in evaluation of extraction performance.

Specification	Solution type	
	Dilute	Concentrated
mg $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ /l	476.30	4762.70
mg Co/l	100.00	1000.00
mg Na_2SO_4 /l	1920.00	19200.00

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