Recovery of Copper, Nickel and Zinc from Sulfate Solutions by Solvent Extraction Using LIX 984N

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Received 22 February 2011; Accepted 26 April 2011

Abstract: Recovery of valuable metals such as copper, nickel and zinc from synthetic sulfate solutions using LIX 984N as the extractant dissolved in kerosene was investigated. The metals get extracted at different equilibrium pH of the aqueous phase and thus the pH of the aqueous solutions forms the basis of their separation. Copper was extracted at an equilibrium pH of 3.5, while nickel and zinc get extracted at an equilibrium pH of 7.3 and 8.8 respectively. The extraction isotherms of copper, nickel and zinc were obtained by contacting the respective metal solutions and 0.05 M LIX 984N at different A:O phase ratio at their respective equilibrium pH of maximum extraction. A two-stage counter-current extraction simulation was done to achieve quantitative extraction of the metals. Furthermore, a two-stage counter-current stripping simulation yielded quantitative recovery of the individual metals, from their loaded organic phases. On the basis of the results, a flow sheet of the process has been developed.

Keywords: Solvent extraction, Hydrometallurgy, Metal recovery, LIX 984N

Introduction

Chalcopyrites, pentaldite, spent catalyst, batteries, brass ash, zinc ash are some of the major sources for the production of copper, nickel and zinc. Apart from sulfides, significant resources for these metals are available as oxide/silicate deposits. These raw materials are subjected to various hydrometallurgical treatments which include atmospheric/pressure/bio-leaching of sulfate/chloride solutions. The leach liquor, thus obtained contains copper, nickel, zinc along with some impurities such as iron, chromium, aluminium, silicon etc. Liquid-liquid extraction has been applied to many solutions to obtain either metals or their salts in highly pure form, as these metals are industrially and economically more important and therefore there is a great need to separate and recover them. Solvent extraction has been
carried out extensively for the separation of cobalt and nickel with alkyl phosphorus reagents like Cyanex 272 and D2EHPA. Similarly Chelating reagents like LIX 87QN, LIX 54, LIX 973, LIX 34, LIX 984.

LIX 984N and LIX 841 have been used for the extraction, separation and recovery of copper, nickel and zinc from ammonium carbonate / ammonium sulfate solutions. Extraction of copper, nickel and zinc from their sulfate solutions, using LIX 841 was found to depend on the equilibrium pH of the aqueous phase. It was found that LIX 984N was very selective for copper extraction. In this paper, we report the solvent extraction separation of copper, nickel and zinc from synthetic solution using LIX 984 N as the extractant dissolved in kerosene, with an aim to separate these metals efficiently by single stage extraction. On the basis of this, a flow sheet of the process has been developed.

**Experimental**

Copper, nickel and zinc solutions were prepared by dissolving their corresponding sulfate salts in double distilled water. The chelating extractant, LIX 984N, a 1:1 mixture of LIX 84 and LIX 860N (mixture of 5-nonylsalicylal doxime and 2-hydroxy 5-nonylacetophenone oxime) was used as received from Cognis – Ireland. Deodourised kerosene (bp, 160-200 °C) was used as a diluent. All other chemicals and reagents used were of analytical grade.

Extractions were carried out by equilibrating a suitable aliquot of the solution containing metal ions at the desired pH with an equal volume of LIX 984N diluted in kerosene for 20 minutes, in glass-stopper bottles using a mechanical shaker. After phase disengagement, the aqueous phase was separated and metal concentrations were analysed by using chemito AA 203 atomic absorption spectrophotometer. The pH of the metal solutions was adjusted by adding dilute sulfuric acid or sodium hydroxide and using equiptronic pH meter model EQ-611. Experimental data were obtained in triplicate and those achieving a 98% mass balance were only accepted. The metal concentration in the organic phase was determined by stripping the loaded organic phase with acid. All experiments were carried out at room temperature.

**Results and Discussion**

**Effect of equilibrium pH**

Extraction studies were carried out with individual metal solutions containing 0.0025 M copper, nickel and zinc sulfate solutions. The extraction of these metals with 0.005 M LIX 984N as a function of pH was studied. The extraction of these metals depends mainly on the equilibrium pH of the aqueous phase as represented in Figure 1. Copper shows quantitative extraction at equilibrium pH 3.5, while nickel and zinc get extracted quantitatively at pH 7.3 and 8.8, respectively. Based on the data pertaining to the extraction behaviour of these metals, a synthetic solution containing 0.150 g/L copper, 0.140 g/L nickel and 0.163 g/L zinc was prepared and used.

**Recovery of copper**

The extraction isotherm for copper was obtained by contacting the metal solution and 0.05 M LIX 984N at different A:O phase ratios at an equilibrium pH of 3.5. The McCabe-Thiele plot (Figure 2) shows that quantitative copper extraction is achievable in two stages at A:O phase ratio of 5.5:1. This was confirmed by a two-stage counter-current extraction simulation experiment carried out at an A:O phase ratio of 5.5:1. Aqueous and organic phases were separated after second cycle onwards and analysed for metal values. The raffinate
was found to contain 0.001 g/L copper, 0.140 g/L nickel and 0.163 g/L zinc. The loaded organic phase contained 0.820 g/L copper. A two-stage stripping was carried out at A:O ratio of 1 with 190 g/L of sulfuric acid to strip copper from the loaded organic phase and the stripping efficiency was > 99%.

**Figure 1.** Influence of Equilibrium pH on the extraction of metals. Aqueous phase: 0.0025 M each of Cu(II), Ni(II) and Zn(II). Organic phase: 0.005 M LIX 984N. Contact time: 20 min. Temperature: Room temperature.

**Figure 2.** Extraction isotherm of copper. Aqueous phase: Cu(II): 0.150 g/L; Ni(II): 0.140 g/L; Zn(II): 0.163 g/L. Organic phase: 0.05 M LIX 984N. Equilibrium pH: 3.5 at room temperature.

**Recovery of nickel**

The raffinate after copper extraction was used for the separation of nickel. Extraction data for nickel was obtained by performing extraction at different A:O phase ratios at an equilibrium pH of 7.3. Figure 3. Shows that quantitative nickel extraction is achievable in two stages at A:O phase ratio of 4:1. This was confirmed by a two-stage counter-current extraction simulation carried out at an A:O phase ratio of 4:1. The raffinate was found to contain 0.001 g/L of nickel and 0.163 g/L of zinc and the loaded organic phase contained 0.500 g/L of nickel. A two-stage stripping was carried out to strip nickel from the loaded organic phase, using 190 g/L of sulfuric acid at A:O phase ratio 1 and the stripping efficiency was > 99%.
Recovery of Zinc

The raffinate after nickel extraction was used for the separation of Zinc. Extraction isotherm of zinc was obtained by performing extractions at different A:O phase ratios at an equilibrium pH of 8.8. A two-stage counter-current extraction simulation was carried out at an A:O phase ratio of 2:1 as obtained from McCabe-Thiele plot (Figure 4). The loaded organic phase was found to contain 0.310 g/L of zinc. A two-stage stripping was carried out to strip zinc from the loaded organic phase, using 150 g/L of sulfuric acid, at A:O ratio of 1:1 and the stripping efficiency was > 99%.

Figure 3. Extraction isotherm of nickel. Aqueous phase: Copper raffinate. Organic phase: 0.05 M LIX 984N. Equilibrium pH: 7.3. Contact time: 20 min at ambient temperature

Figure 4. Extraction Isotherm of Zinc. Aqueous phase: Nickel raffinate. Organic phase: 0.05 M LIX 984N. Equilibrium pH=8.8. Contact time: 20 min

Flow sheet for the recovery of copper, nickel and zinc

Based on the extraction behaviour of these metals, a flow sheet for the separation of copper, nickel and zinc has been developed using LIX 984N and is shown in the Figure 5.

Figure 5. Flow sheet for the recovery of copper, nickel and zinc from a leach liquor containing 0.150 g/L copper, 0.140 g/L nickel and 0.163 g/L zinc
Conclusion
LIX 984N was effectively used for the extractive separation of copper, nickel and zinc. Counter-current simulation extraction of these metals showed almost quantitative loading in two stages. The extraction of the metals depends on the equilibrium pH of the aqueous phase. Therefore, it was possible to separate the metals from one another based on their extraction behaviour at different equilibrium pH. Thus the recovery of these metals by counter-current stripping simulation was quantitatively accomplished.

Acknowledgment
We thank Dr. A. V. Reddy (Head, Analytical Chemistry Division, BARC) and Dr. Meera Venkatesh for helping us in this work. Thanks are also due to Mrs. Rohini and Mr. Shivkumar for their technical assistance. We are grateful to Dr. Werner Schwab (Cognis dealer, Germany) and Mr. Manish Kapadia (Cognis dealer, Mumbai) for supplying the LIX 984N sample for the present study.

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