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NONDISPERSIVE LIQUID-LIQUID EXTRACTION OF Zn(II), Cu(II), Co(II), AND Cd(II) FROM DILUTE SOLUTION WITH DEHPA IN A HOLLOW-FIBER MEMBRANE MODULE

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ABSTRACT

The ability to concentrate dilute aqueous solutions containing metals is desirable both from the standpoint of waste volume reduction and increased ease of concentration measurement. The first challenge in designing a liquid-liquid extraction (LLE) system to concentrate metals is to find an organic system that can be used to simultaneously remove a number of metals. The goal of this work was to find a LLE system that extracts zinc, copper, cadmium and cobalt simultaneously. In the LLE extraction experiments, nearly 100% extraction of all four metals (Zn(II), Cu(II), Cd(II) and Co(II) at about 25 ppm initially) was achieved when all of them were extracted simultaneously using 0.3 mol/L DEHPA in n-heptane. The extraction order observed as the pH of the aqueous solution increased was Zn(II)>Co(II)>Cu(II)>Cd(II). Based on these results, a polysulfone Hollow Fiber Contained Liquid Membrane (HFCLM) system was designed and operated to study the nonequi-

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librium extraction of all four metals via nondispersive LLE. The aqueous and organic streams were operated at 100% recycle. Interference effects between the four metal ions are discussed.

INTRODUCTION

Metals like zinc, copper, cadmium and cobalt are commonly found in industrial wastewater. There is an increasing environmental concern about heavy metal contamination of groundwater and soil from industrial wastewater discharge. Therefore, an effective and efficient method of removal and recovery of these metals from dilute solutions is necessary. Current practices use precipitation or ion exchange and create a sludge that has to be disposed of in a landfill. Electrochemical metal recovery is a promising alternative; however, waste streams are often too dilute in metals and have low plating efficiencies (Raghuraman et al., 1994).

One way of recovering metals from wastewater, prior to discharge, would be to use liquid-liquid extraction. Liquid-liquid extraction uses a solvent, which is immiscible in water, to extract the metal from the wastewater. Extraction, however, still produces a waste solvent, contaminated with the metal that has to be disposed of.

There have been recent studies using liquid membranes to remove and recover metals. Liquid membranes may offer an attractive alternative to conventional solid or liquid ion-exchange processes where large volumes of solution require treatment (Pearson, 1983). Two types of membranes are Emulsion Liquid Membrane (ELM) and Supported Liquid Membrane (SLM). An ELM process includes four steps: (1) emulsification, (2) dispersion, (3) settling, and (4) breaking the emulsion. The advantage of using an ELM is that it combines the extraction and stripping in a single operation, which eliminates equilibrium limitations and reduces metal concentrations in the feed to very low levels (Ho and Sirkar, 1992). Two main disadvantages of an ELM are swelling and leakage of the emulsion.

An SLM uses an organic liquid that is immobilized in the pores of a microporous membrane interposed between two aqueous solutions. One advantage of using a SLM is it can be selective for particular solutes. The major disadvantage of a SLM is its lack of long-term membrane stability (Ho and Sirkar, 1992).

An alternative to ELMs and SLMs is a Hollow-Fiber Contained Liquid Membrane (HFCLM). A HFCLM consists of microporous hollow fibers arranged in a shell-and-tube configuration (see Figure 1). HFCLM permeation-separation may be applied to almost any separation problem to which other liquid membrane techniques have been applied (Ho and Sirkar, 1992). The HFCLM can offer a very high surface area/volume ratio without dispersion or mixing of the two phases (Raghuraman and Wiencek, 1993).

A successful HFCLM process for simultaneously removing and recovering

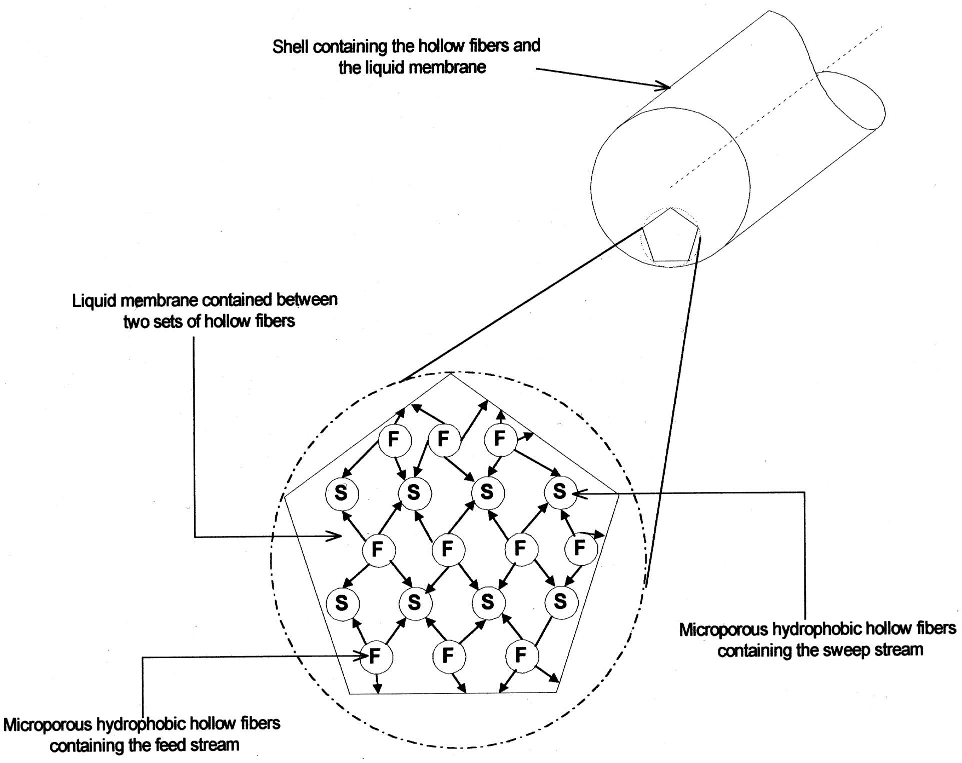

Figure 1. A configuration of the hollow-fiber contained liquid membrane (HFCLM) in a permeator shell.

a number of metals will require an appropriate extractant. Acid extractants react with metal cations to form neutral complexes that are preferentially dissolved by the organic phase, and the extent of extraction depends on the acidity of the aqueous phase. These extractants can be further subdivided into chelating and non-chelating species. The latter include carboxylic, phosphoric, and sulphonic acids and provide a very versatile series of reagents. The extent of extraction follows a combination of metal ion basicity and extractant acidity.

The most widely used organophosphoric reagent and the one used in this work is di(2-ethylhexyl) phosphoric acid (DEHPA, see Figure 2). Its chemical stability, low aqueous solubility, and good loading and stripping characteristics make it one of the most versatile extractants (Thornton, 1992). DEHPA is used commercially to recover uranium, vanadium, beryllium, yttrium, cadmium, copper, cobalt, zinc, rare earths and other valuable metals (Albright and Wilson Americas, 1992). However, these applications are at metal concentrations orders of magni-

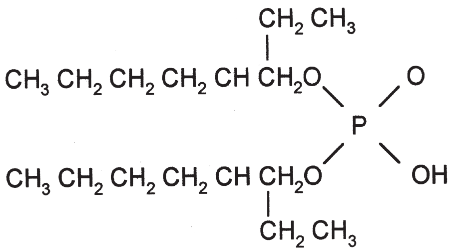

Figure 2. Chemical structure of DEHPA (Albright and Wilson Americas, 1992).

tude larger than the dilute concentrations considered herein. EHPA may be used alone or in combination with other synergistic extractants including trioctylphosphine oxide, tributyl phosphate, and dibutyl butyl phosphonate.

DEHPA has been shown to exist largely as dimers in organic diluents, leading to the formation of polymeric metal species at low metal loading. As metal loading increases these polymers dissociate, giving species such as MR_n, although these too may be polymeric with bridging oxygen atoms (Thornton, 1992). The major component in the organic phase is the diluent, which is required to reduce the viscosity of the extraction solution, provide a suitable concentration of extractant for the metal in the feed, reduce the tendency of extractants to emulsify with the aqueous feed, and improve dispersion and coalescence (Thornton, 1992). Taking into consideration these factors, we chose n-heptane as a diluent for this study.

The DEHPA-Metal extraction behavior for zinc, copper, cadmium and cobalt is as follows:

$$Zn^{+2} + \frac{1}{2}\overline{n(RH)_2} \leftrightarrow \overline{ZnR_2\ (n-2)\ HR} + 2H^+ \tag{1}$$

$$Cu^{+2} + \frac{1}{2}\overline{n(RH)_2} \leftrightarrow \overline{CuR_2\ (n-2)\ HR} + 2H^+ \tag{2}$$

$$Cd^{+2} + \frac{1}{2}\overline{n(RH)_2} \leftrightarrow \overline{CdR_2\ (n-2)\ HR} + 2H^+ \tag{3}$$

$$Co^{+2} + \left[\frac{n+2}{2}\right]\overline{(RH)_2} \leftrightarrow \overline{CoR_2\ (HR)_n} + 2H^+ \tag{4}$$

The stoichiometric factor n indicates the number of molecules involved in the reaction, and the bars indicate species in the organic phase. It can be seen that the extraction reactions for the dimerized DEHPA-Metals considered herein are similar. Table 1 shows the values of n obtained from the literature for the four metals at concentrations higher than those considered herein. The values of the stoichiometric factor depend on the metal and solvent used. Grimm and Kolarik (1974) extracted Zn(II) from nitrate solution with DEPHA in n-dodecane. A slope of (n/2), 1.7, was obtained. This slope of 1.7 instead of 1.5 was explained by the coexistence of ZnR_2-HR and ZnR_2-2HR in the region of $0.03M < [H\ _2R_2] < 0.1M$.

Laboratory results and pilot plant studies show that LMs are capable of reducing the level of cadmium from several hundred ppm to less than 1 ppm under batch or continuous flow conditions (Kitiwaga et al., 1977). Grimm and Kolarik (1974) extracted Cd(II) from nitrate solution with DEHPA in n-dodecane. A slope of 2.5 was obtained when they plotted log D vs. pH, indicating that the complex CdR_2-3RH was extracted. Raghuraman et al. (1995) extracted Cd(II) with DEHPA in tetradecane using ELM. They reported binary equilibrium data for Cd-DEHPA systems used in emulsion extractions. Predictive models that include aqueous phase nonidealities and all aqueous ionic equilibria were developed. The model overpredicted the organic phase loading in the low aqueous phase metal concentration region (less than 100 ppm). At a concentration of 1000 ppm cadmium, 100% extraction was achieved using 10% DEHPA in tetradecane when the equilibrium pH was greater than 3.5. Juang and Lo (1994) and Grimm and Kolarik (1974) suggested that only the extracted species $CoR_2 \cdot (HR)_2$ is formed in the organic phase.

The objective of this work was to investigate the use of HFCLM to simultaneously extract zinc, copper, cadmium, and cobalt from dilute aqueous solution using di(2-ethylhexyl)phosphoric acid (DEHPA) as the extracting agent. This study is unique in the attention paid to interference effects between the metals and in the low metal concentrations considered.

Table 1. Stoichiometric Factors from Literature for DEHPA-Metal Reactions

Metal	Stoichiometric Factor (n)	Source	Solvent
Zinc	3.0	Ajawin et al., 1983	n-heptane
Zinc	3.4	Grimm and Kolarik, 1974	n-dodecane
Copper	4.0	Grimm and Kolarik, 1974	n-dodecane
Cadmium	5.0	Grimm and Kolarik, 1974	n-dodecane
Cobalt	2.02	Juang and Lo, 1994	kerosene
Cobalt	2.0	Grimm and Kolarik, 1974	n-dodecane

Table 2. Characteristics of A/G Tech's Xampler UFP-5-E-3A Membrane Module

Material	Polysulfone
Nominal Molecular Weight Cutoff	5,000
Fiber Internal Diameter	1 mm
Module Length	33.7 cm
Module Diameter	0.9 cm
Membrane Area (based on inside)	0.007 m^2
Nominal number of Fibers	12
Lumen Nominal Holdup Volume	3 mL
Shell Nominal Holdup Volume	5 mL
Porosity	80 %
Wall Thickness (including skin)	0.3 mm

EXPERIMENTAL MATERIALS/METHODS

Materials and Sample Preparation Information

The sources for zinc, copper, cadmium, and cobalt were $ZnSO_4$-$7H_2O$, $CuSO_4$-$5H_2O$, $3CdSO_4$-$8H_2O$, and $CoSO_4$-$7H_2O$, respectively, all of which were analytical grade (Fisher Scientific). The pHs were adjusted to the desired value by continuous drop addition of 1% (v/v) Sulfuric Acid or 5N NaOH prepared with analytical grade reagents. DEPHA was provided by Albright and Wilson Americas. The aqueous samples were analyzed for metal content using a GBC 932 Atomic Absorption (AA) spectrophotometer. A mass balance provided the organic phase concentration.

The hollow fiber membrane module used in our experiments was a Xampler UFP-5-E-3A manufactured by A/G Technology Corporation (see Table 2). This polysulfone membrane material in this module is hydrophilic and macrovoid free. The tubing used in these experiments was Norton Pharmed NSF – 51 tubing (Fisher Scientific). This tubing is chemically resistant to the n-heptane organic phase. All experiments were conducted at room temperature.

Methods

The experiments were divided into two parts: Liquid-Liquid Extraction (LLE) experiments and Hollow Fiber Contained Liquid Membrane (HFCLM) experiments. In the first part, a system that extracts zinc, copper, cadmium and cobalt at equilibrium was investigated. In the second part, a polysulfone hollow-fiber membrane module was used to extract zinc, copper, cadmium, and cobalt from an aqueous solution to an organic solution under non-equilibrium conditions.

Liquid-Liquid Extraction Experiments

The effect of pH on Metal-0.3 mol/L DEHPA systems and the interference of the various metal ion combinations on the extraction were studied. The LLE experiments were performed by pipetting 50 mL of each solution, aqueous and organic, into a 125 mL separatory funnel. The pH of the mixture (organic and aqueous) was then adjusted and measured. The samples were then shaken at 110 rpm for 1 hour, allowed to settle in a separatory funnel, and the two phases were separated. The shaking time needed to insure equilibrium conditions was previously determined by measuring the percent of extraction as a function of the shaking time. The final pH of the aqueous phase was recorded as the equilibrium pH. Next, the aqueous samples were analyzed for zinc and/or copper and/or cadmium and/or cobalt.

Hollow-fiber Contained Liquid Membrane Experiments

In all the experiments involving the Hollow Fiber Contained Liquid Membrane (HFCLM) the aqueous phase was passed though the tube side and the organic phase was passed through the shell side countercurrently (see Figure 3). The

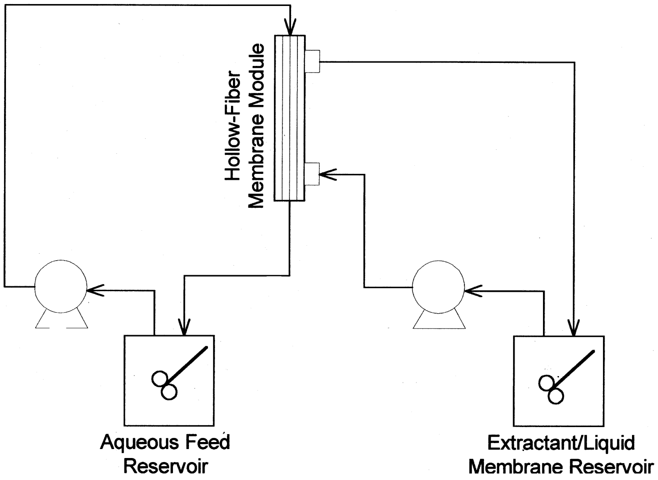

Figure 3. Schematic of a countercurrent hollow-fiber membrane module setup with 100% recycle.

aqueous outlet was kept at atmospheric pressure while the organic inlet was at 0.5 psig. These pressures allowed a stable aqueous/organic interface to be maintained at the shell side of the membrane. Both the aqueous and organic phases were recycled totally (100%). The aqueous and organic reservoirs were placed on magnetic stirrers. The reservoir volumes were 500 mL each, and the samples were approximately 5 mL. Samples were taken at 1, 5, 10, 20, 30, 45, 60, 90, 120, 180, 240, and 300 minutes to follow the extraction.

RESULTS AND DISCUSSION

Two types of experiments were performed to investigate the effectiveness of DEHPA as an extractant for copper, zinc, cobalt and cadmium metal ions from aqueous solution. Liquid-liquid extraction (LLE) was used to determine at equilibrium how much zinc, copper, cobalt and cadmium could be extracted with DEHPA. The hollow-fiber membrane module was used to determine the nonequilibrium behavior of nondispersive LLE. Values reported in this section are averages of three replicates.

Liquid-Liquid Extraction Experiments

The goal of these experiments was to find a system that simultaneously extracts zinc, copper, cadmium, and cobalt. To achieve this, a system that extracts all the above metal ions individually was studied. Then the effect of metal interfering ions was studied. Finally, a system that extracts zinc, copper, cadmium, and cobalt simultaneously was studied (Srinivas, 1997).

The effect of equilibrium pH on simultaneous extractions of zinc and copper, zinc and cadmium, zinc and cobalt, copper and cadmium, copper and cobalt, and cadmium and cobalt from aqueous solution with 0.3 mol/L DEHPA were investigated (Figures 4 through 9, respectively). The figures show the simultaneous extraction of the ion pairs along with single ion extraction results. As can be seen from Figure 4, 100% extraction of both zinc and copper was achieved when the equilibrium pH reached about 3.50. Figure 5 shows that about 100% extraction of zinc was achieved when the equilibrium pH reached about 2.30, whereas the maximum cadmium extraction was achieved when the equilibrium pH reached about 2.00, after which the extraction fell. About 100% extraction of both zinc and cobalt was achieved when the equilibrium pH reached about 3.90 (Figure 6). About 100% extraction of cadmium was achieved when the equilibrium pH reached about 3.00, whereas only about 60% extraction of copper was achieved at that pH for copper (Figure 7). Figure 8 shows that about 100% extraction of cobalt and 95% extraction of copper were achieved when the equilibrium pH reached

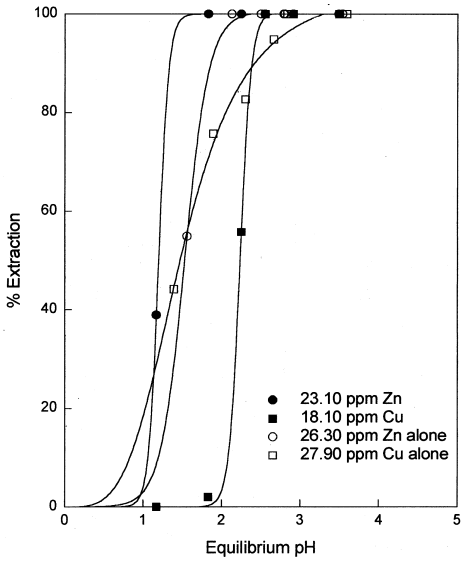

Figure 4. Simultaneous LLE of zinc and copper from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

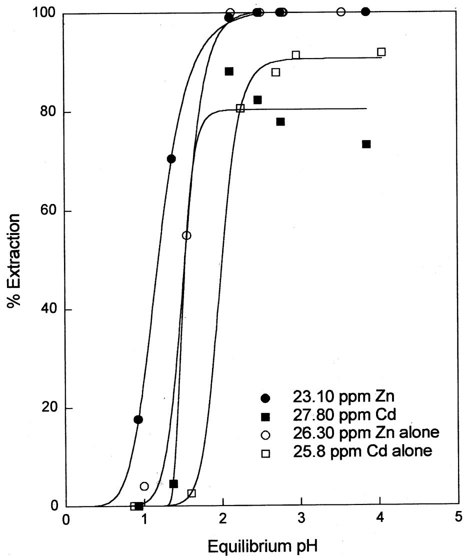

Figure 5. Simultaneous LLE of zinc and cadmium from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

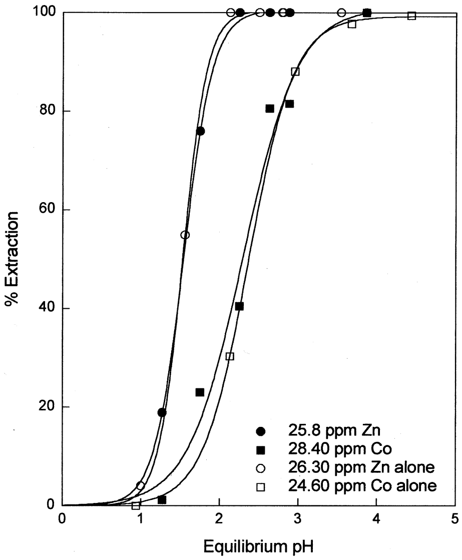

Figure 6. Simultaneous LLE of zinc and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

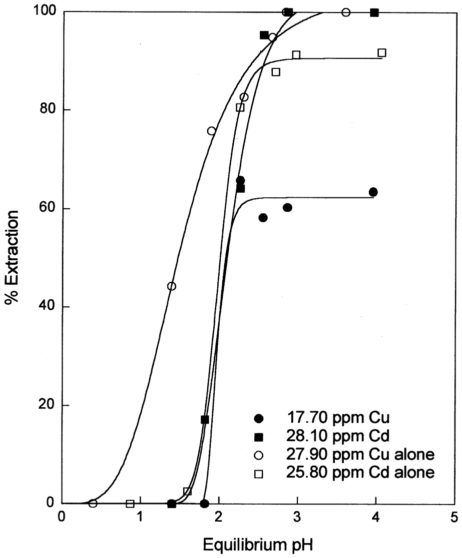

Figure 7. Simultaneous LLE of copper and cadmium from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

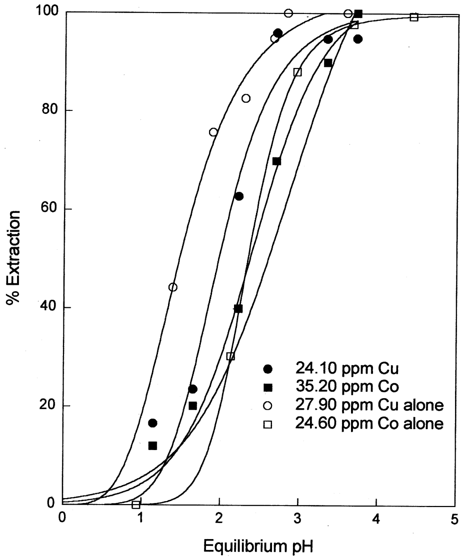

Figure 8. Simultaneous LLE of copper and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

about 3.70. About 80% extraction of cobalt and about 50% extraction of cadmium were achieved when the equilibrium pH reached about 3.60 (Figure 9). Comparing the two metal extraction results with single metal ion extraction, one can see that the extraction of both zinc and copper were affected by the presence of each other; the extraction of cadmium was affected by the presence of zinc, whereas extraction of zinc was not affected; the extraction of both zinc and cadmium were not affected by the presence of each other; the extraction of both copper and cadmium were affected by the presence of each other; the extraction of both copper and cobalt were affected by the presence of each other; and the extraction of both cadmium and cobalt were affected by the presence of each other.

The simultaneous extraction of zinc, copper, cadmium and cobalt from dilute aqueous solution was studied. The average initial concentrations of the zinc, copper, cobalt, and cadmium in the aqueous solution were 26.6, 29.25, 31.2 and 28.9 ppm, respectively. The percent extraction of the metal ions as a function of equilibrium pH is shown in Figure 10. About 100% extraction of all four metals was achieved when the equilibrium pH reached about 3.0. Comparing the results with single ion extraction, one can see that the extraction of zinc was not affected, whereas the extraction of copper, cadmium, and cobalt were improved. To fully understand this behavior, further study of the reaction mechanisms is needed along with numerical analysis of the data with the LETAGROP_DISTR program

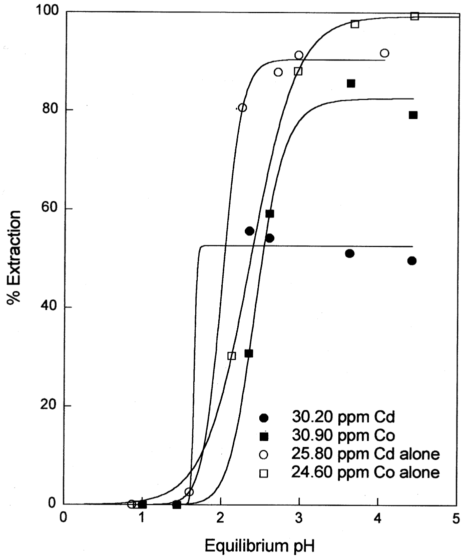

Figure 9. Simultaneous LLE of cadmium and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

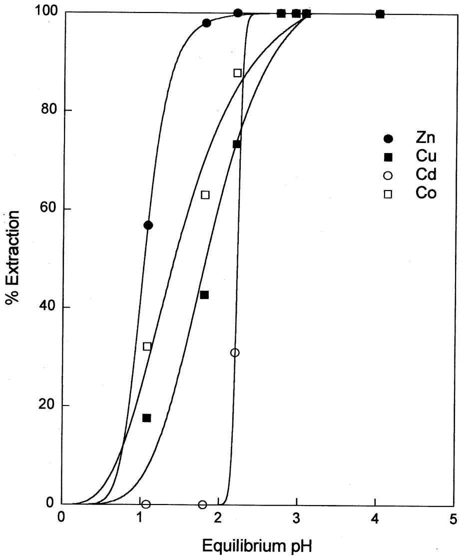

Figure 10. Simultaneous LLE of zinc, copper, cadmium and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane compared to single ion extraction.

(Liem, 1971; Rodriguez de San Miguel, et al., 1997). This program takes into account dimerization of the extractant in the organic phase and the formation of aqueous complexes of the metals with the buffer solutions employed. From Table 1, we can see that the stoichiometric factor, n, changes for each of the metals, and also, that it depends on the solvent used for the extraction. From the reaction rate constants of the DEHPA-metal reaction, an estimate of the affinity of the DEHPA for the metals could be studied and that could explain the anomalous behavior.

Hollow-Fiber Contained Liquid Membrane Experiments

HFCLM experiments were performed to study the nonequilibrium, simultaneous extraction of all four metals. Yezek (1996) studied the effect of flow rates on the extraction of zinc and copper systems with DEHPA using the Xampler UFP-5-E-3A module—the results of which suggested that the most efficient extraction occurs for a shell-side flow rate of 77 ml/min and a tube-side flow rate of 78 ml/min (Ilias et al., 1999). Based on this, we chose the organic and aqueous flow rates to be 78 ml/min.

The percent extraction of metal ions as a function of time at 100% recycle of the feed solution and the organic phase is shown in Figure 11. The initial con-

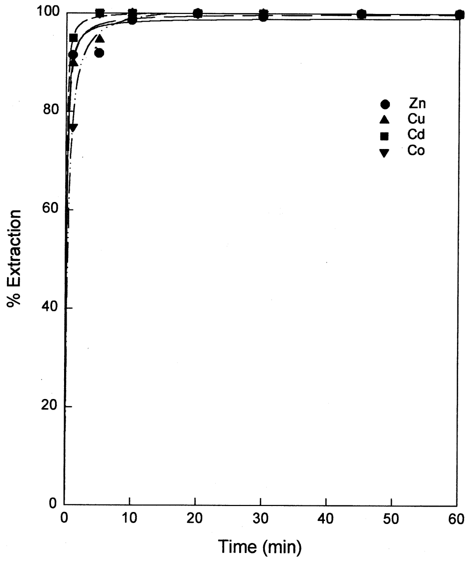

Figure 11. Simultaneous extraction of about 25 ppm of zinc, copper, cadmium, and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane using Hollow-Fiber Contained Liquid Membrane (HFCLM).

centrations of zinc, copper, cadmium, and cobalt in the aqueous solution were 28.00, 31.30, 23.50, and 31.50 ppm, respectively. As can be seen from Figure 11, nearly 100% extraction of all four metals was achieved within 20 minutes of extraction. The initial pH of the aqueous phase was set to be 5.45. The equilibrium pH was found to be 2.48.

To determine the effect of increasing the initial concentrations on the extraction, initial concentrations of zinc, copper, cadmium and cobalt of 114.40, 112.20, 141.20 and 105.80 ppm, respectively, were used. The percent extraction of each of the metal ions as a function of time is shown in Figure 12. About 100% extraction of zinc, copper and cadmium and about 97% extraction of cobalt were achieved within 60 minutes. The initial pH of the aqueous phase was 5.45, and the equilibrium pH was found to be 2.21.

To determine the effect of further increases in the initial concentration on the extraction, the metal ion concentrations were doubled from the previous experiment. The initial concentrations of zinc, copper, cadmium and cobalt were 214.00, 164.00, 190.00 and 266.00 ppm, respectively. The percent extraction of metal ions with time is shown in Figure 13. About 100% extraction of zinc and copper, 98% extraction of cadmium, and 99% extraction of cobalt were achieved

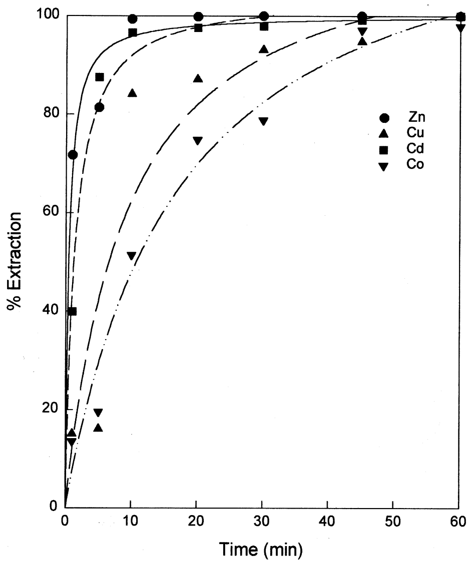

Figure 12. Simultaneous extraction of about 100 ppm of zinc, copper, cadmium, and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane using Hollow-Fiber Contained Liquid Membrane (HFCLM).

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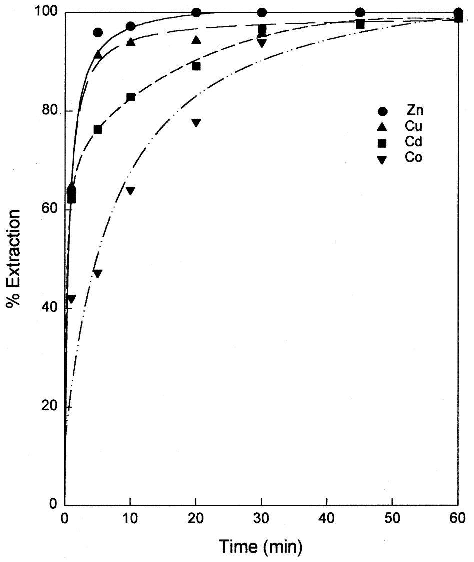

Figure 13. Simultaneous extraction of about 200 ppm of zinc, copper, cadmium, and cobalt from dilute aqueous solution with 0.3 mol/L DEHPA in n-heptane using Hollow-Fiber Contained Liquid Membrane (HFCLM).

within 60 minutes. The extraction of copper, cadmium and cobalt was affected by the increase in the initial concentration from 25 ppm to 200 ppm. The initial pH of the aqueous phase was set to be 5.22, and the equilibrium pH was found to be 2.24.

CONCLUSIONS

In this study, nondispersive liquid-liquid extraction (LLE) was investigated for the removal of metal ions from dilute wastewater using the concept of contained liquid membranes in hollow-fiber membrane modules. For extraction of metal ions from aqueous solution to an organic phase, DEHPA was investigated as a potential extracting agent. In the LLE experiments, nearly 100% extraction of all four metals (Zn, Cu, Cd and Co at about 25 ppm initially) was achieved when all of them were extracted simultaneously using 0.3 mol/L DEHPA in n-heptane. In the nonequilibrium hollow-fiber membrane experiments (LLE), the polysulfone membrane/DEHPA system was successful in extracting all four metals simultaneously.

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