Separation Science and Technology
Publication details, including instructions for authors and subscription information:
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To cite this Article Lee, Jae-Chun , Jeong, Jinki , Chung, Kang-Sup and Kobayashi, Mikio(2005) 'Active Facilitated Transport and Separation of Co in Co-Ni Sulfate Solution by Hollow Fiber Supported Liquid Membrane Containing HEH(EHP)', Separation Science and Technology, 39: 7, 1519 — 1533

To link to this Article: DOI: 10.1081/SS-120030794

URL: http://dx.doi.org/10.1081/SS-120030794

SEPARATION SCIENCE AND TECHNOLOGY
Vol. 39, No. 7, pp. 1519–1533, 2004

Active Facilitated Transport and Separation of Co in Co–Ni Sulfate Solution by Hollow Fiber Supported Liquid Membrane Containing HEH(EHP)

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ABSTRACT

The active facilitated transport and separation of cobalt in mixed solution of cobalt and nickel sulfate were studied using hollow-fiber supported liquid membrane (HF-SLM). The HF-SLM contained 2-ethylhexylphosphonic acid mono-2 ethylhexyl ester [HEH(EHP)], which was diluted in kerosene as a carrier. The permeation rates and separation factors were found to be dependent on the operational variables such as the

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DOI: 10.1081/SS-120030794
0149-6395 (Print); 1520-5754 (Online)
www.dekker.com

concentrations of metal and hydrogen ions in the feed solution, the carrier concentration in the liquid membrane, and the flow rate of feed solution. The permeation rate of cobalt was 10^{-6}–10^{-7} mol/m^2 s and the separation factor of Co/Ni was 10–220 under the experimental conditions. By recirculating the strip solution, a tenfold enrichment of cobalt was possible and cobalt with 95% purity was recovered from the low concentration solution in which the concentrations of both cobalt and nickel were 17 mol/m^3.

Key Words: Cobalt; Nickel; Metal ion separation; Hollow fiber-supported liquid membrane; HEH(EHP).

INTRODUCTION

The effective separation of cobalt and nickel remains one of the important tasks in the field of hydrometallurgy because both metals are contained together in ore and show a similar chemical behavior. Cobalt and nickel have often been separated by the chemical or reductive precipitation method from aqueous solution and such processes are still employed in commercial plants.[1–5]

The demand for higher purity metals has encouraged the development of improved separation methods and the solvent extraction has been introduced for the separation of cobalt and nickel. The application of solvent extraction has been reported to provide not only much improved selectivity for cobalt, but also high recovery of cobalt from solution.

Recent trends toward environment friendly technology drew attention to nondispersive solvent extraction, which appears to meet the requirements for performance and economics in replacing the conventional dispersive solvent extraction. One of the outstanding characteristics of nondispersive solvent extraction is that the organic solvent is not dispersed in the aqueous phase since the membrane acts as a semipermeable barrier between two phases. Various configurations are currently applicable to obtain nondispersive contact between the aqueous and organic phases using a porous solid support such as a membrane contactor,[6] a supported liquid membrane (SLM),[7,8] and a contained or flowing liquid membrane.[9,10] Among nondispersive solvent extraction technologies, the SLM is expected to be one of the most promising candidates for future commercial applications in the separation of metals ions.

Many have conducted research on the separation of cobalt by the nondispersive solvent extraction using the SLM. Chaudry et al.[11] investigated the extraction of cobalt by flat-sheets supported liquid membrane (FS-SLM) containing *bis*(2-ethylhexyl) phosphoric acid (D2EHPA) as a carrier and reported the dependence of the complexation reaction rates and diffusion resistance on temperature with which the permeability of cobalt increases. Several studies[12–15] were attempted to develop a mathematical model for transport mechanisms in the separation of cobalt by FS-SLM impregnated with 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester [HEH(EHP)] or D2EHPA as a cobalt carrier. The FS-SLM, including the works mentioned previously, has been widely employed in the separation of cobalt. However, only a few studies were reported for the separation of cobalt by nondispersive solvent extraction using the hollow-fiber supported liquid membrane (HF-SLM).[16,17]

In the present study, the active facilitated transport and separation of cobalt from mixed solution of cobalt and nickel sulfate were investigated using HF-SLM from Hoechst Celanese Corporation. The HEH(EHP) was used as a carrier of cobalt. The effects of the operational variables on the separation factors as well as the permeation rates were examined: the hydrogen and metal concentrations in the feed solution, the carrier concentration in the liquid membrane, and the flow rate. In addition, the enrichment of cobalt in the strip solution was obtained by recirculating the feed and/or strip solutions. The stability of SLM is a very important subject as discussed in our previous work.[16] Experiments in this study were conducted for periods during which SLM is expected to be stable.

EXPERIMENTAL

In this study HEH(EHP) (commercial name PC88A) from Daihachi Chemical Industry Co. was used as a cobalt carrier without further purification. Commercial EP grade kerosene from Junsei Chemical Co. was used as the diluent. The hollow fiber module manufactured by Hoechst Celanese Co. (Liqui-Cel.; Material, polypropylene; number of fibers, 3600; fiber length, 16 cm; fiber I.D., 240 μm; total surface area, 0.4 m^2; fiber wall thickness and porosity, 30 μm and 30%; pore diameter, 0.03 μm) was used as the liquid membrane support. This module is a simple shell-and-tube type device without any shell-side baffles.[17] The HF-SLM was prepared by flowing the organic solution through the tube side of fibers and soaking the microporous fiber walls for at least 30 min. Before soaking, the fiber was cleaned by flowing $0.5 \times 10^{-3}\,\mathrm{mol/m^3}$ sulfuric acid solution and methyl alcohol and dried by nitrogen flow. Distilled water was circulated to remove the residual organic solution on the fiber wall after soaking.

The liquid membrane solutions were prepared by diluting the carrier in kerosene at a predetermined weight ratio. The cobalt and nickel sulfate solutions were used as the feed solution after adjusting acidity by adding NaOH or H_2SO_4. In a single-pass flow mode to measure the permeation rate and separation factor of cobalt, the strip solution was $0.5 \times 10^{-3}\ \mathrm{mol/m^3}$ sulfuric acid. The strip solution of $1.5 \times 10^{-3}\ \mathrm{mol/m^3}$ sulfuric acid was used in the recirculating flow mode for the enrichment of cobalt.

The acidity of feed and strip was adjusted automatically to maintain the constant value throughout the experiment. The feed and strip solutions were passed through the lumen and shell side of fibers, respectively.

The concentration of metal ions in the aqueous phase was measured by the inductively coupled plasma-atomic emission spectrometry (ICP-AES, JOBIN YVON, JY 38+). Figure 1 shows the schematic diagram for the separation of cobalt and nickel ions by HF-SLM containing HEH(EHP) as a cobalt carrier.

RESULTS AND DISCUSSION

Permeation Rate

Figure 2 shows the effect of cobalt concentration in the feed solution on the cobalt permeation rate in the single-pass flow mode. The permeation rate

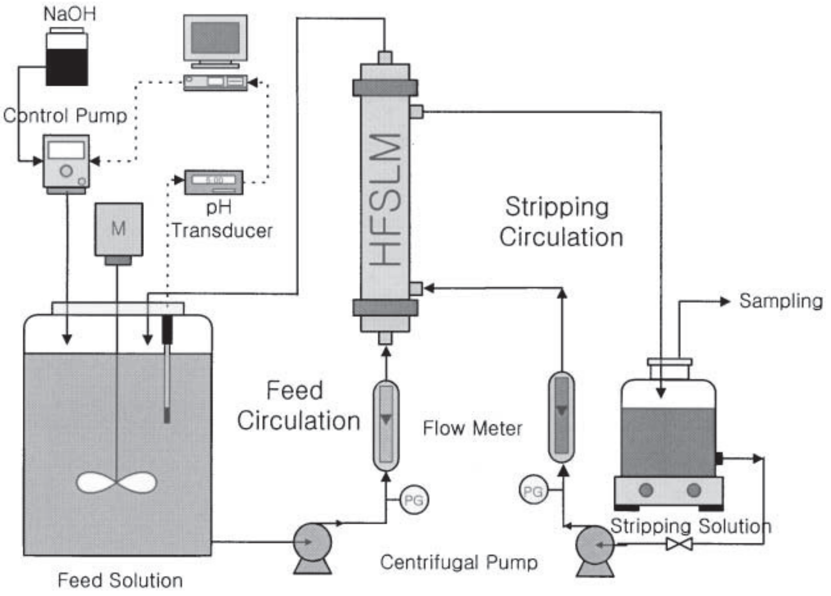

Figure 1. Schematic diagram of experimental apparatus for the separation of cobalt using HF-SLM.

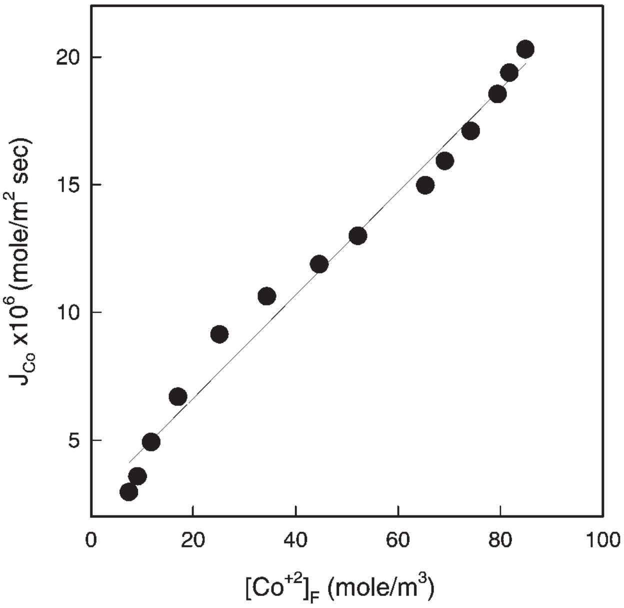

Figure 2. Effect of $[Co^{+2}]_F$ on the permeation rate in a single component solution ($pH_F = 5$, flow rate $= 1 \times 10^{-3}$ m^3/min, carrier = 10 wt%).

increases linearly with the cobalt concentration. The permeation rate is known to be proportional to the cobalt concentration in the feed solution at normal concentration ranges as reported previously by Youn et al. using FS-SLM.[15] The value of the cobalt permeation rate is found about $3–20 \times 10^{-6}$ mol/m^2 sec at concentrations of 7.5–85 mol/m^3. This value is about 40% of that obtained by using FS-SLM under similar experimental conditions.[15] This is mainly due to the reduction of effective membrane area for mass transport resulting from wall contact of 3600 hollow fibers contained in the one-inch diameter tube. In view of this, the HF configuration method appears to be important to increase the effective membrane area. The research to elucidate this effect has been reported.[18]

The permeation rates of cobalt in mixed solutions of cobalt and nickel at various conditions are shown in Figs. 3–6.

Figure 3 shows the competitive permeation of cobalt with various cobalt and nickel concentrations in the feed solution. The permeation rate increases linearly with concentration in the feed solution of low concentration range. At higher concentrations over 50 mol/m^3, the permeation rate becomes maximum. This result agrees with the experimental data reported

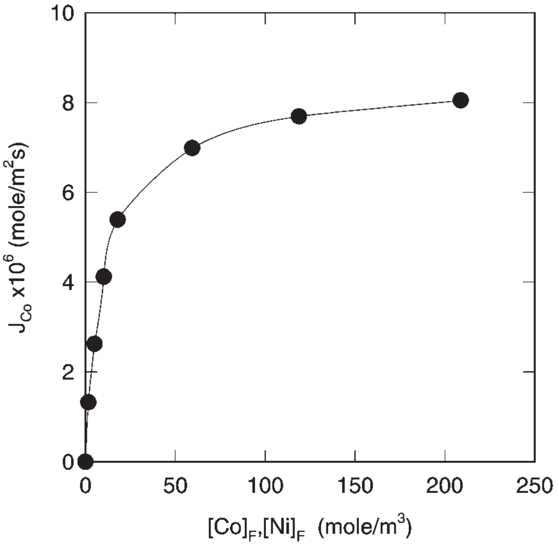

Figure 3. Effect of $[Co^{+2}]_F$ and $[Ni^{+2}]_F$ on the permeation rate of cobalt in a mixed components solution ($[Co^{+2}]_F = [Ni^{+2}]_F$, $pH_F = 4.7$, flow rate $= 1 \times 10^{-3}\,m^3/min$, carrier $= 10\,wt\%$).

by Teramoto[20] and Haan et al.[21] When metal concentration is low, the diffusion of metal species through the boundary layer in the feed phase is rate controlling.[20] Then, the permeation rate was increased linearly with concentration.

In the meantime, in regions of high cobalt concentration, more formation of cobalt-carrier complexes depletes the free carrier by retarding the diffusion of the complex in the liquid membrane and consequently limiting the cobalt permeation rate. In this case, the diffusion of the carrier from strip-membrane to the feed-membrane interface is rate determining. Then, the permeation rate becomes maximum.[20] Youn et al.[15] suggested that the hydrogen transfer resistance in the boundary layer of feed solution plays a significant role in the retarding cobalt permeation in addition to the decrease in the free carrier concentration in their model prediction using FS-SLM.

Figure 4 shows the effect of bulk hydrogen concentration in the feed solution on the permeation rate. The permeation rate decreases sharply with the increase of the hydrogen ion in regions of concentration higher than $0.01\,mol/m^3$. The increase of hydrogen ion depresses the formation of the cobalt–carrier complex at the interface between the feed and liquid membrane according to the equilibrium relationship. In regions of low hydrogen

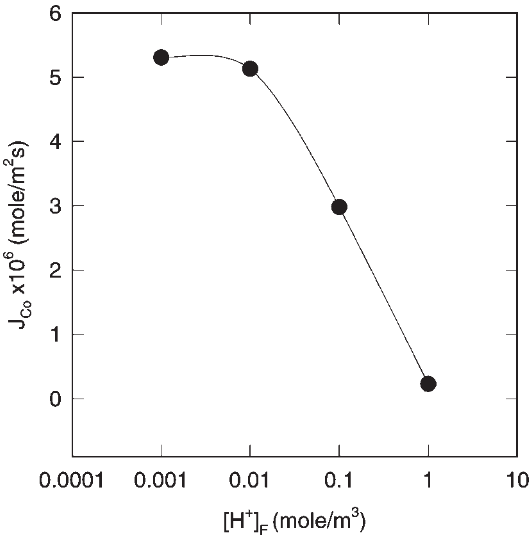

Figure 4. Effect of $[H^+]_F$ on the permeation rate of cobalt in a mixed components solution ($[Co^{+2}]_F$, $[Ni^{+2}]_F = 17\,mol/m^3$, flow rate $= 1 \times 10^{-3}\,m^3/min$, carrier $= 10\,wt\%$).

concentration below about $0.01\,mol/m^3$, the effect of hydrogen is negligible and the permeation rate is limited to an almost constant value due to the saturation of carrier at the interface between the feed and liquid membrane.

The permeation rate of cobalt at various flow rates of feed solution is shown in Fig. 5. The flow of feed solution in HF is lamina since the Reynold number is 29.8 at the maximum flow rate of $1.23 \times 10^{-3}\,m^3/min$ as used in this study. Under the lamina flow conditions, the increase of flow rate enhances the permeation rate of cobalt. The concentration gradient of cobalt in lumen of HF is developed because cobalt is depleted transferring from the feed solution to the liquid membrane. The fast flow reduces the concentration gradient between the bulk solution and the interface by compensating cobalt instantaneously. This should enhance the mass-transfer efficiency and result in the increase of cobalt permeation.

Figure 6 shows the permeation rate of cobalt at various carrier concentrations in liquid membrane. The permeation rate of cobalt increases with the carrier concentration. This occurs because the cobalt–carrier complexes between cobalt and carrier are formed in greater numbers at the interface as carrier concentration increases. At the same time, Fig. 6 shows that the increasing permeation rate reduces with the carrier concentration. The increase in the carrier concentration increases the viscosity of liquid membrane and thereby retards the diffusion of the cobalt–carrier complexes and

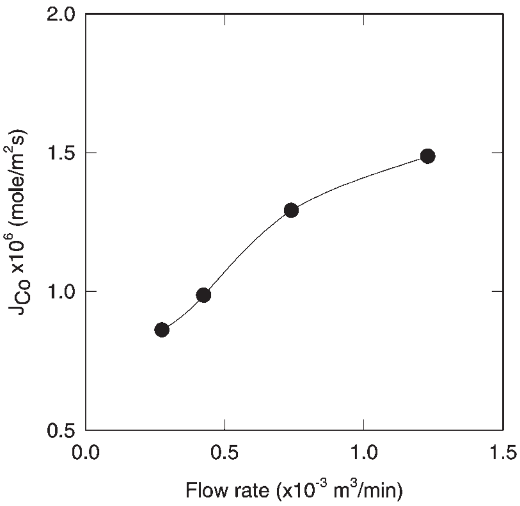

Figure 5. Effect of flow rate on the permeation rate of cobalt in a mixed components solution ($[Co^{+2}]_F = 0.85\,mol/m^3$, $[Ni^{+2}]_F = 85\,mol/m^3$, $pH_F = 6$, carrier = 30 wt%).

free carrier in liquid membrane.[22] Therefore, the permeation rate is expected to decrease at higher concentrations over a certain range where the complex formation increases slightly with carrier concentration and the effective diffusivities of the complex and free carrier decrease rapidly.[17]

Separation and Enrichment in the Strip Solution

Figures 7 and 8 show the separation factor of cobalt with flow rates at various pH of the feed solution and carrier concentration. The separation factor is defined as follows:

$$\text{Separation factor} = \frac{J_{Co}/[Co^{+2}]_F}{J_{Ni}/[Ni^{+2}]_F}$$

where J_{Co} and J_{Ni} are the permeation rates of cobalt and nickel, respectively. Their feed concentrations are $[Co^{+2}]_F$ and $[Ni^{+2}]_F$.

The separation factor of cobalt increases with flow rate at the carrier concentration of 10 wt%. However, the flow rate shows relatively little effect on the separation factor at the carrier concentration of 30 wt%. At the same time, the effect of the hydrogen concentration in the feed solution is observed to be negligible. As stated in detail in our previous paper,[17] the increase in

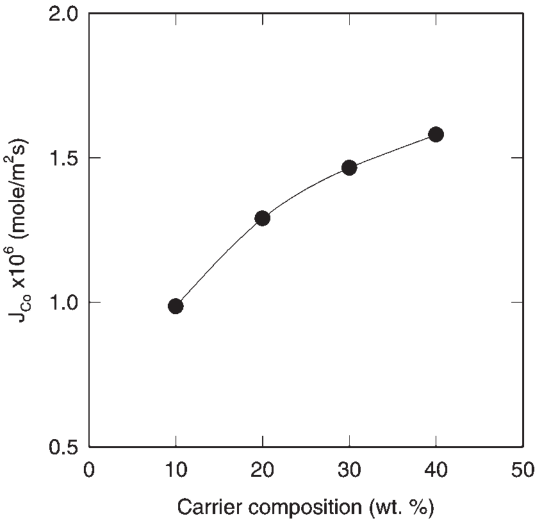

Figure 6. Effect of carrier concentration on the permeation rate of cobalt in a mixed components solution. ($[Co^{+2}]_F$, $[Ni^{+2}]_F = 0.85\,mol/m^3$, $pH_F = 4.7$, flow rate $= 0.275 \times 10^{-3}\,m^3/min$).

the flow rate enhances the mass transfer in the feed solution and results in the increase of cobalt permeation, consequently resulting in the observed increase in the separation factor. When the carrier concentration increases from 10 to 30 wt%, the separation factor of cobalt decreases. Though, the permeation rate of cobalt is enhanced by the abundant formation of cobalt–carrier complexes at higher carrier concentrations, the separation factor of cobalt decreases because the permeation rate of nickel is enhanced by the increase of the nickel–carrier complexes at the same time. The permeation rate of cobalt is remarkably affected by the changes of pH in the feed solution as shown in Fig. 4, while the separation factor of cobalt is little affected due to the simultaneous effect of pH on the permeation rate of nickel as well as cobalt. By comparing the results with the concentration ratio of cobalt and nickel shown in Figs. 7 and 8, it can be seen that the separation factor of cobalt becomes higher in higher nickel concentration range since the permeation rate of nickel does not increase significantly with its concentration. It could be presumed from the experimental results that a low concentration of carrier and high flow rate are favored for a higher separation factor of cobalt in mixed solutions of cobalt and nickel.

One of the outstanding characteristics of SLM is the enrichment of the component required. This can be achieved by the facilitated transport characteristics inherent to the liquid membrane. The enrichment effect is

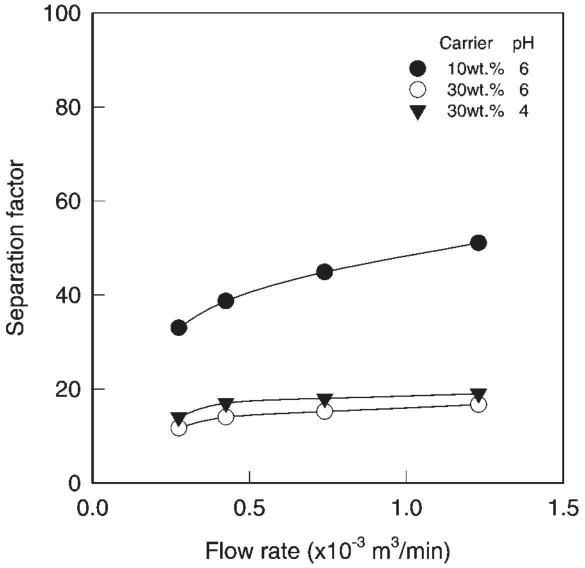

Figure 7. Effect of flow rate on the separation factor of cobalt in a mixed components solution ($[Co^{+2}]_F$, $[Ni^{+2}]_F = 0.85\,mol/m^3$, $pH_F = 4$–6, carrier = 10–30 wt%).

not easy to confirm in FS-SLM which has a small membrane surface area unlike the case in HF-SLM. In this study, the enrichment of cobalt in the mixed solution of cobalt and nickel using HF-SLM is demonstrated and summarized in Figs. 9 and 10.

Figure 9 shows the variation of cobalt concentration in the strip solution with time. The initial concentrations of both cobalt and nickel in the mixed feed solution were 17 mol/m^3, and the sulfuric acid solution of 1.5×10^{-3} mol/m^3 was used as the strip solution. The amount of $1 \times 10^{-3}/m^3$ of the feed solution and strip solution was recirculated continuously for the enrichment of cobalt. After 70 min, about 95% of cobalt in the feed solution was transferred to the strip solution. At the final stage of the enrichment, an active facilitated transport of cobalt occurs from the feed solution of 0.85 mol/m^3 Co towards the strip solution of 16.15 mol/m^3 Co. The permeation rate of cobalt is kept relatively constant during the initial 30 min and decreases slowly with time. In contrast, the permeation rate of nickel increases with time after the initial 30 min. This occurs since more carrier in membrane can participate in the formation of complexes with nickel, as cobalt in the feed solution is depleted. The recovery rate of cobalt increases with the operation time but the purity of cobalt in the strip solution decreases. In the enrichment operation

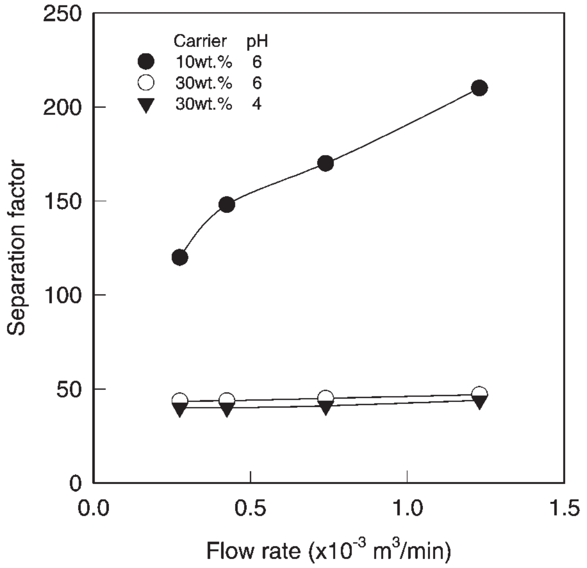

Figure 8. Effect of flow rate on the separation factor of cobalt in a mixed components solution ($[Co^{+2}]_F = 0.85\,mol/m^3$, $[Ni^{+2}]_F = 85 mol/m^3$, $pH_F = 4–6$, carrier = 10–30 wt%).

for 30 min, about 65% of cobalt is recovered and the purity of the cobalt is 92.8%. By extending the operation time to 70 min, the recovery rate of cobalt increases up to 95%, while the separation factor decreases from 13 to 2.5, suggesting that cobalt with 85% purity can be obtained.

Figure 10 shows the experiment results for cobalt enrichment in which the cobalt and nickel concentrations in feed solution were maintained at 17 mol/m^3. The feed solution was passed without recirculation. The acid concentration in $1 \times 10^{-3}\,m^3$ strip solution was maintained at $1.5 \times 10^{-3}\,mol/m^3$ and recirculated continuously for enrichment of cobalt. As the initial concentration of each metal in the feed is kept constant by supplying the fresh feed solution without recirculation, the permeation rate of cobalt does not decrease with time during the enrichment of cobalt while that of nickel does not increase. Therefore, the reduction of the cobalt permeation rate in Fig. 9 should be attributed to the decrease of the cobalt concentration in the feed solution. By recirculating the strip solution, a tenfold enrichment of cobalt is possible, and cobalt with 95% purity is recovered from low concentration solution in which the concentrations of both cobalt and nickel in the mixed feed are 17 mol/m^3.

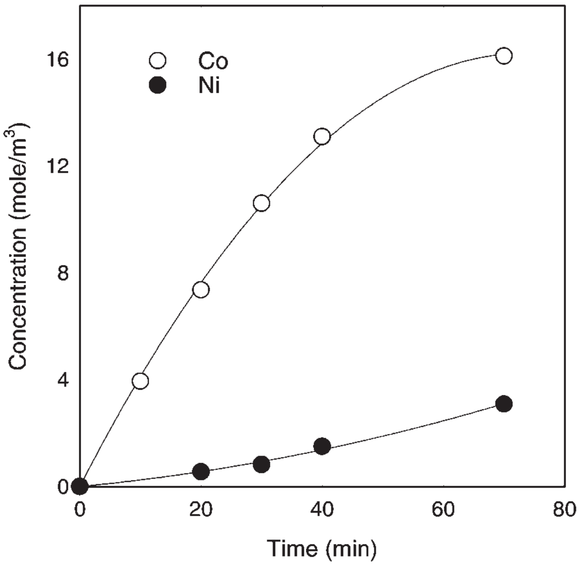

Figure 9. Enrichment of cobalt in the strip solution from a mixed solution of cobalt and nickel sulfate with time by recirculating the feed and strip solution (initial $[Co^{+2}]_F$, $[Ni^{+2}]_F = 17\,mol/m^3$, $pH_F = 4.5$, carrier = 10 wt%, feed flow rate = $1 \times 10^{-3} m^3/min$, feed and strip volume = $1 \times 10^{-3} m^3$).

From the results in Figs. 9 and 10, it can be seen that the transport of cobalt through SLM may takes place effectively against the concentration gradient of cobalt in virtue of the driving force obtained as a consequence of the concentration gradient of counter ion, hydrogen. It is estimated that about 15% of hydrogen ions in the strip solution are consumed to enrich the cobalt of $180\,mol/m^3$ in the strip solution. However, in a practical system, excessive amounts of hydrogen in the feed solution are not favorable in an economical point of view. In some cases, the strip solution containing the metal ions may be recirculated as a feed for the further separation. In such a case, since the excess amount of the hydrogen should be neutralized by adding the alkaline reagent, it is very important to maintain the hydrogen concentration at a minimum level below which the permeation rate is not affected.

CONCLUSIONS

The active facilitated transport and separation of cobalt from a mixed solution of cobalt and nickel sulfate were studied using HF-SLM. The

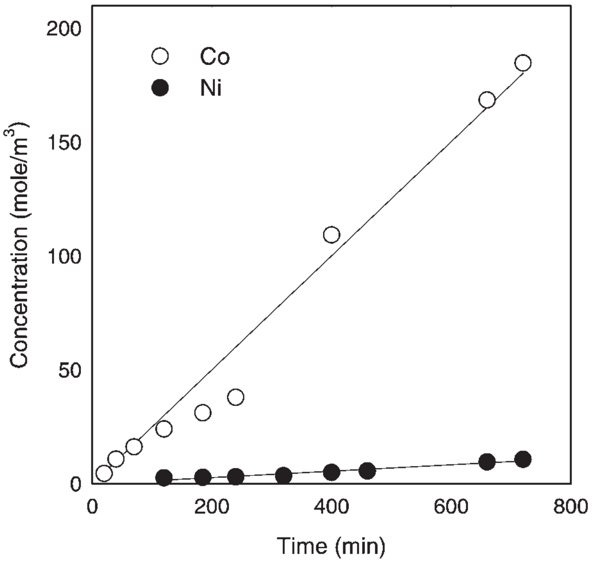

Figure 10. Enrichment of cobalt in the strip solution from a mixed solution of cobalt and nickel sulfate with time by recirculating the strip solution ($[Co^{+2}]_F$, $[Ni^{+2}]_F = 17\,mol/m^3$, $pH_F = 4.5$, carrier = 10 wt%, feed flow rate = $1 \times 10^{-3} m^3/min$, strip volume = $1 \times 10^{-3} m^3$).

permeation rate of cobalt through HF-SLM was proportional to the cobalt concentration in the feed solution. At a low carrier concentration of 10 wt%, fast flow increased the permeation rate of cobalt and, consequently, the separation factor. However, the flow rate showed relatively little effect on the separation factor at a high carrier concentration of 30 wt%. It could be presumed from the experimental results that a low concentration of carrier and high flow rate are favorable for a higher separation factor of cobalt in mixed solution of cobalt and nickel. The permeation rate of cobalt was remarkably affected by the changes of pH in the feed solution, while the separation factor of cobalt was little affected due to the simultaneous effect of pH on the permeation rate of nickel as well as cobalt. The separation factor of cobalt becomes higher at higher nickel concentrations, since the permeation rate of nickel did not increase significantly with its concentration. In this study, the enrichment of cobalt in a mixed solution of cobalt and nickel using HF-SLM was demonstrated. By recirculating the strip solution, a tenfold enrichment of cobalt was possible and cobalt with 95% purity was recovered from low concentration solution in which the concentrations of both cobalt and nickel were $17\,mol/m^3$.

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Received July 2003
Accepted November 2003

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