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Separation of Cobalt and Nickel by Pertraction in a Rotating Film Contactor Using Cyanex 302 as a Carrier

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Abstract: The extraction behavior of Cyanex 272, Cyanex 301, and Cyanex 302 for recovery of cobalt and nickel from sulphate media was studied. Cyanex 302 was found to be the most suitable reagent for extraction of both metals and for Co(II)/Ni(II) separation. Competitive pertraction of Co(II) and Ni(II) in a rotating film contactor with Cyanex 302 as a carrier was studied. At appropriately chosen operational conditions Co(II) was preferentially removed, while Ni(II) remained in the feed solution. Pertraction process using Cyanex 302 as a carrier guarantees an excellent separation of Co(II) from Ni(II): the obtained separation factors are

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$K_{sep} \sim 1.0 \times 10^4$. Higher pH of the feed solution favors the recovery of both metals, but deteriorates Co(II)/Ni(II) separation ability. At neutral pH of the feed solution and strongly acidic acceptor solution, pertraction process can be used for simultaneous and complete recovery of both metals, also.

Keywords: Cobalt, nickel, separation, liquid membranes, Cyanex 302, pertraction

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INTRODUCTION

Cobalt and nickel are among the most important nonferrous metals. Certain raw materials such as laterite ores are the main sources for their production. Hydrometallurgical methods of leaching applying sulphate, chloride-sulphate, and chloride systems result in leach liquors containing mainly nickel and cobalt in company of some impurities, such as copper, iron, and aluminium. Usually, the majority of these impurities is removed by precipitation with lime, while the copper is cemented out with cobalt powder and recovered by filtration (1). The obtained purified leach liquors contain both Co(II) and Ni(II) ions. It is very difficult to obtain pure cobalt and nickel compounds from such solutions because of the similar physico-chemical properties of these metals. Using classical methods, such as precipitation, oxidation, and crystallization, it is impossible to separate the nickel-cobalt pair in a simple and economical way. Solvent extraction seems to be the most efficient method for separation of Co(II) and Ni(II) ions from solutions, including leach liquors (2–6). Generally, cobalt is preferentially removed with a suitable extractant, leaving nickel behind. Among a large number of studied extracting agents, some organophosphorus compounds have demonstrated best Co(II)/Ni(II) separation ability. Di-2-ethylhexyl phosphoric acid (D2EHPA) is the first widely used extractant for recovery of Co(II) and Ni(II) and for their separation (4–9). Nevertheless, the process of Co(II) and Ni(II) separation using D2EHPA as extracting agent requires very strict pH control and relatively high operational temperatures (above 50°C). Afterward, the development of phosphonic and phosphinic acid extractants led to improvement in Co(II)/Ni(II) separation factors. Co(II) was successfully separated from Ni(II) from sulphate solutions using the 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester (PC 88A) (10, 11) and the bis(2,4,4-trimethylpentyl)phosphinic acid (Cyanex 272) (9, 12–14). Sodium salts of D2EHPA, PC 88A and Cyanex 272 for Co(II)/Ni(II) separation have also been used (15, 16). Two thiophosphinic extractants, Cyanex 301 (whose active component is the bis(2,4,4-trimethylpentyl)dithiophosphinic acid) and Cyanex 302 [with bis(2,4,4-trimethylpentyl)monothiophosphinic acid as an active component], have recently become available from Cytec Industries Inc. These extractants have demonstrated good potential for recovery of Co(II) and Ni(II) and for their separation from aqueous solutions (9, 17–20).

The use of liquid membranes containing specific carriers offers an alternative method for recovery of various metal ions from solutions and their separation (21, 22). Solute transport across a liquid membrane is a combination of extraction and stripping in a single unit operation called pertraction. This process provides maximum yield of the extracted solute with minimum extractant and power consumption (23). Usually, two aqueous solutions, the feed (donor) solution F and the acceptor (strip) solution A, are intermediated by an organic liquid: the membrane solution M, which is practically insoluble in both aqueous solutions. The solute is transferred from the feed solution across the organic membrane to the acceptor solution under the effect of well-chosen thermodynamic conditions at the two interfaces. Among the existing techniques, the pertraction in a rotating film contactor merits particular attention, because of its stable and efficient continuous operation, avoiding any phase dispersion or phase intermixing (23–27).

The aim of this work was to study the process of Co(II) and Ni(II) removal from sulphate solutions applying batch pertraction in a rotating film contactor in order to recover and separate these metals.

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EXPERIMENTAL

Reagents and Analytical Methods

Cyanex 272, Cyanex 301, and Cyanex 302 were kindly supplied by Cytec Industries Inc. and used without further purification. Kerosene (White Spirit, France, bp = 155–170°C, $d_4^{20} = 0.748$), cobalt sulphate ($CoSO_4 \times 7H_2O$), nickel sulphate ($NiSO_4 \times 6H_2O$), ammonium sulphate, sulphuric acid, and ammonia (all p.a. grade reagents from Fluka) were used as received.

Metal ions concentrations in the aqueous solutions were measured by atomic absorption spectrometry using SpectrAA-100 (Varian) after appropriate dilutions. Concentrations of Co(II) and Ni(II) in the organic solutions were obtained from the mass balance. The acidity of the aqueous solutions was measured with a pH-meter HI (Hanna Instruments).

Experimental Equipment and Procedures

The equilibrium data on Co(II) and Ni(II) distributions between the organic and the aqueous phases were obtained using 100 mL separating funnels. The aqueous and organic phases (each of 20 mL) were mixed and shaken intensively. It was previously established that for the whole range of studied experimental conditions, 15 min of shaking were sufficient to attain the equilibrium. The organic phase was a solution of the studied extracting agent (Cyanex 272, Cyanex 301, or Cyanex 302, respectively) in kerosene and the aqueous phase was a sulphate solution of Co(II) and Ni(II) ions. Extraction of metal ions with ionic carriers *HA*, such as the active components of the three studied extractants, can be considered as counter-coupled transport, because the transfer of metal ions from the aqueous to the organic phase is accompanied by a transfer of the substituted protons in the opposite direction:

$$(Me^{2+})_{aq} + 2(HA)_{org} \longleftrightarrow (MeA_2)_{org} + 2(H^{+})_{aq} \tag{1}$$

Consequently, the aqueous phase acidity increases during the process of metal ions extraction. To maintain a desired pH, a portion of ammonium sulphate/ammonia buffer solution was added to the aqueous phase. The initial metal concentrations in the aqueous phase were determined prior to phase contact and their equilibrium concentrations after complete phase separation.

The studies of Co(II) and Ni(II) pertraction were carried out in a laboratory rotating film contactor made of Plexiglas®, which is described elsewhere (27). The lower part of the contactor is divided into four compartments: two for the feed and two for the acceptor solution. The organic membrane liquid occupies the common upper part of the contactor. In each compartment rotates a disc, coated by hydrophilic coating. The lower part of each disc is immersed in the corresponding aqueous solution and the upper part is immersed in the common organic liquid membrane. The aqueous solutions F and A form mobile liquid films on the corresponding disc surfaces, which contact the common organic membrane. Rotation of the discs provides continuous renewal of the aqueous films on the disc surfaces as well as an agitation of all three phases.

All equilibrium and kinetic studies were carried out in an air-conditioned room at temperature of 293 K. Pertraction studies were carried out at constant discs rotation velocity of 20 rpm.

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RESULTS AND DISCUSSION

Equilibrium Studies

Equilibrium distribution of Co(II) and Ni(II) between an acidic aqueous phase and an organic phase was evaluated as a function of aqueous solution acidity. The extraction behavior of three extractants from the Cyanex series was studied. The organic phase contained 0.1 M *HA* (the active component of the corresponding extractant) in kerosene and the aqueous phase contained both 5.0 mM Co(II) and 5.0 mM Ni(II) in ammonia sulphate solution. The obtained results, represented in Fig. 1, show that the extraction of both metals is strongly affected by the aqueous phase acidity. For all studied extractants, higher pH favors the recovery of both metals. It should be mentioned that Co(II) is extracted in preference to Ni(II) by any given extractant. The obtained results are in good agreement with the observation of Sole and

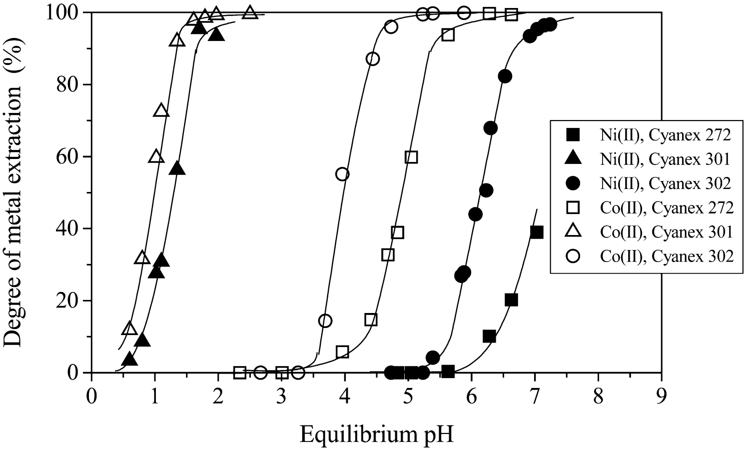

Figure 1. Effect of pH on metal extraction with Cyanex 272, Cyanex 301, and Cyanex 302 (the aqueous solutions contained both metals, $[\mathrm{Co}]_{aq} = 5.0\,\mathrm{mM}$, $[\mathrm{Ni}]_{aq} = 5.0\,\mathrm{mM}$, $[\mathrm{HA}]_{org} = 0.1\,\mathrm{M}$).

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Hiskey (17) and Tait (18) that extraction ability of phosphinic acid-based extractants, under comparable conditions, increases in the order:

$$\text{phosphinic acid} < \text{thiophosphinic acid} < \text{dithiophosphinic acid}$$

The most efficient extractant for both studied metals is the Cyanex 301. Since this extractant contains two sulphur donor atoms, it tends to form more stable complexes with divalent metal ions, such as Co(II) and Ni(II), than the extractants Cyanex 272 and Cyanex 302, which contain oxygen donor atoms. However, compared to the other two studied extractants, Cyanex 301 is apparently the less selective extractant for Co(II)/Ni(II) separation. The separation of Co(II) from Ni(II) ions using Cyanex 301 as extracting agent requires very strict pH control. Moreover, due to the high stability of the complexes formed between the metal ions and Cyanex 301, the stripping of loaded with metal ions organic phase after the extraction was very difficult. Even when the stripping was carried out with strongly acidic aqueous solutions it was not complete. In contrast to Cyanex 301, the stripping of loaded with Co(II) and Ni(II) ions other two studied extractants, Cyanex 272 and Cyanex 302, with 1.0 M H_2SO_4 was prompt and entire. Figure 1 shows also that these two extractants are appropriate for Co(II)/Ni(II) separation. At the experimental conditions, pH interval between 4.0 and 5.5 is favorable for Co(II)/Ni(II) separation using Cyanex 302, while pH interval between 5.0 and 6.5 is found to be suitable for this purpose with Cyanex 272 as extractant. While for the earlier commercialized extractant, Cyanex 272, more information is available, the Cyanex 302 is less studied. In contrast to Cyanex 272, Cyanex 302 provides almost complete extraction of both metals at the studied operational conditions. In addition, the lower solubility of Cyanex 302 in water (3 mg/L only) is a serious advantage, also (28). Therefore, our next studies were carried out with Cyanex 302 with the bis(2,4,4-trimethylpentyl) monothiophosphinic acid (R_2PSOH) as a chief component.

The influence of the initial metal concentration in the aqueous phase on metal recovery from ammonium sulphate media was studied with 0.1 M R_2PSOH in kerosene and individually aqueous solutions of Co(II) or Ni(II) ions, respectively. To reveal better this effect we carried out the studies at equilibrium pH, high enough for metal recovery: $pH = 6.00 \pm 0.05$ for Co(II) and $pH = 7.00 \pm 0.05$ for Ni(II) ions, respectively. The influence of the initial metal concentration on metal distribution ratios D at constant equilibrium pH is presented in Fig. 2. The obtained high-distribution ratios at low metal concentrations correspond to an almost complete extraction of both studied metals. Higher metal concentrations provoke a decrease of distribution ratios, because, at these conditions, there is a lack of free extractant to react with metal ions.

The expected positive effect of extractant concentration in the organic phase on metal extraction was observed, also. Figure 3 shows the results obtained with individually metal aqueous solutions of 5.0 mM Co(II) at $pH = 6.00 \pm 0.05$ and 5.0 mM Ni(II) at $pH = 7.00 \pm 0.05$, respectively. Distribution ratios of both metals rise with increasing of extractant concentration and, at high extractant concentrations, tend to constant values.

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Pertraction Studies

A liquid membrane (pertraction) process of Co(II) and Ni(II) recovery was studied in the previously described rotating film contactor. The feed

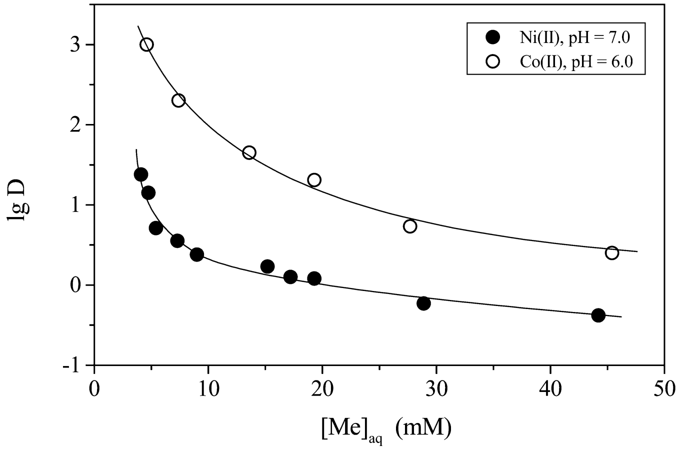

Figure 2. Influence of initial metal concentration on metal extraction with Cyanex 302 (individually metal aqueous solutions, $[R_2PSOH]_{org} = 0.1\,M$).

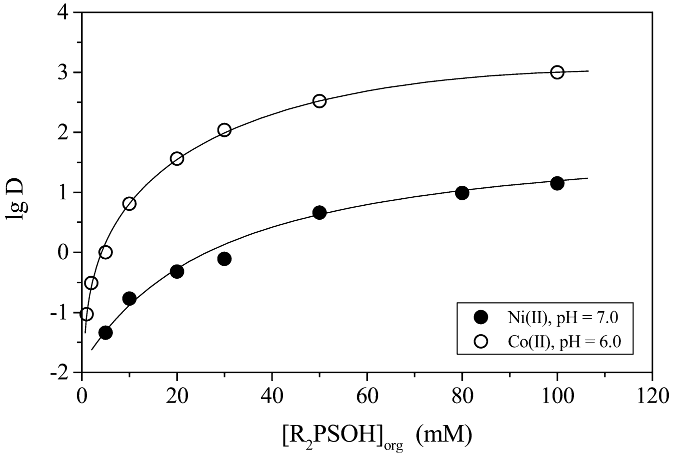

Figure 3. Influence of extractant concentration in the organic phase on metal extraction (individually metal aqueous solutions, $[Co]_{aq} = 5.0$ mM, $[Ni]_{aq} = 5.0$ mM).

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solution (250 mL) contained both 0.01 M Co(II) and 0.01 M Ni(II). In order to provide an appropriate and steady acidity of the feed solution during whole pertraction process, this phase was buffered by ammonium sulphate/ammonia buffer solution. A 0.1 M R_2PSOH in kerosene was used as a membrane solution (1250 mL) and a 1.0 M solution of H_2SO_4 as an acceptor phase (250 mL).

The interaction between the carrier and metal ions, according to Eq. (1) occurs at the first interface F/M. The formed metal-carrier complexes are then extracted to the organic membrane and, due to the concentration gradient created, transferred to the second interface M/A, where the conditions are favorable for decomposition of the complexes and transfer of Co(II) and Ni(II) ions into the acceptor solution. The acidity of the feed solution was monitored during the pertraction process. In spite of feed solution buffering, the transfer of protons toward this phase provoked a slight decrease of pH_F. To maintain the required acidity of the feed phase small portions of ammonia buffer solution were periodically added to the feed solution.

Co(II)/Ni(II) separation ability applying pertraction in a rotating film contactor was studied at constant discs rotation velocity of 20 rpm, varying the acidity of the feed solution. Figure 4 shows the evolution of dimensionless Co(II) and Ni(II) concentrations in the feed solution vs. time for three studied pH_F: 4.40 ± 0.05, 5.00 ± 0.05, and 5.60 ± 0.05, respectively. At lower pH_F, Co(II) ions were preferentially extracted, while Ni(II) ions remained in the feed. As expected, the increase of pH_F favored the extraction of both metals, and at $pH_F = 5.60 \pm 0.05$, the recovery of Ni(II) was not negligible. At these conditions, the extraction of Co(II) was also faster and almost complete. The accumulation of metal ions in the acceptor solution is

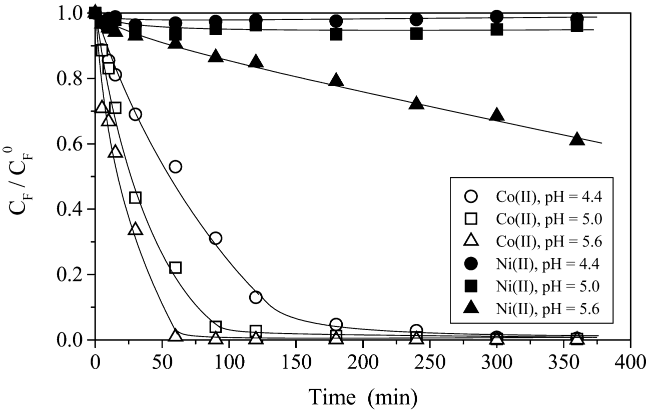

Figure 4. Effect of feed solution pH on Co(II) and Ni(II) extraction in a rotating film contactor (the feed solutions contained both metals).

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presented in Fig. 5. Reasonably, for both studied metals, better extraction from the feed led to an advanced metal accumulation in the acceptor phase. The obtained results demonstrate the high efficiency of Co(II) pertraction in a rotating film contactor using Cyanex 302 as a carrier. Even at the less favourable conditions ($pH_F = 4.40 \pm 0.05$), about 90% of the present Co(II) was accumulated in the acceptor solution. The trends in Figs. 4 and 5 are consistent with the results reported on Co(II) recovery applying other types liquid membranes and Cyanex 302 as carrier (9, 29). A comparison shows that the process of Co(II) recovery in the case of emulsion liquid membranes,

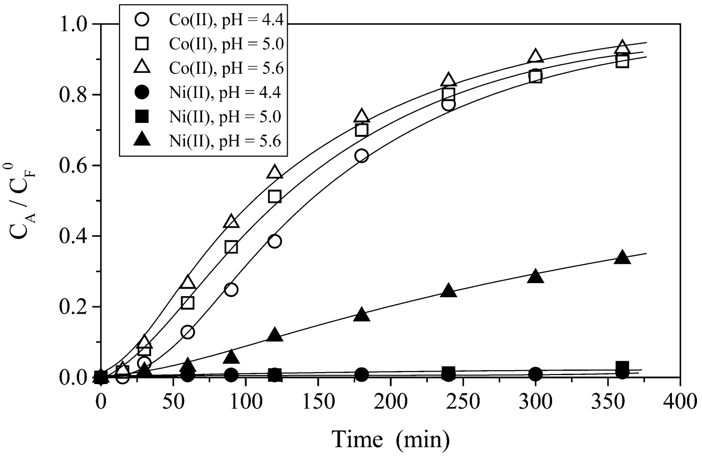

Figure 5. Effect of feed solution pH on Co(II) and Ni(II) accumulation in the acceptor solution (the feed solutions contained both metals).

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recently reported by Ribeiro et al. (29), is quite faster, but less efficient: as a result of membrane breakup, even at the best operational conditions (temperature of 323 K and pH ~5.5) less than 85% cobalt extraction was attained. In contrast, the process of Co(II) recovery with supported liquid membranes, proposed by Gega et al. (9), is very slow: about 24 h are required to accumulate 90% of the present Co(II) in the acceptor solution.

The separation factor of competing metal ions can be calculated according to

$$K_{sep} = \frac{[Co^{2+}]_A \cdot [Ni^{2+}]_F}{[Ni^{2+}]_A \cdot [Co^{2+}]_F} \tag{2}$$

The separation factors obtained at the experimental conditions are presented in Table 1. Obviously, the Cyanex 302 is an excellent extractant for Co(II)/Ni(II) separation. A comparison between the obtained separation factors shows that highest K_{sep} was found at the lowest studied pH_F. Higher pH_F causes a slight decrease of the separation factor, as a result of growing Ni(II) ions permeation at these conditions. The whole studied pH_F interval is found to be very suitable for Co(II)/Ni(II) separation using Cyanex 302 as a carrier. The obtained separation factors are higher than the reported earlier (9, 18). This is a result of better chosen operational pH_F, feed solution buffering, and the pertraction technique used. A positive influence of the solvent used is also possible.

The evolution of dimensionless amount of Co(II) and Ni(II) in all three phases vs. time in the case of $pH_F = 5.60 \pm 0.05$ is shown in Fig. 6. The results allow elucidating the way of extractant loading during the pertraction process. Due to the fast kinetics of Co(II) extraction, the concentration of the cobalt-carrier complex in the organic membrane increases quickly at the beginning of the experimental run. After about 1 h, owing to the stripping with sulphuric acid of the loaded carrier at the second interface M/A, the amount of Co(II) in the membrane liquid decreases continuously. The profile of the amount of nickel-carrier complex in the organic phase is similar, but lower, because of the slower Ni(II) ions extraction from the feed solution at these conditions, resultant of the small Ni(II) distribution ratio.

A simultaneous pertraction of Co(II) and Ni(II) applying rotating film contactor and using Cyanex 302 as a carrier at operational conditions

Table 1. Separation factors K_{sep} after 6 h pertraction at 20 rpm

pH_F	$\frac{[Co^{2+}]_A}{[Co^{2+}]_F}$	$\frac{[Ni^{2+}]_A}{[Ni^{2+}]_F}$	$K_{sep} = \frac{[Co^{2+}]_A \cdot [Ni^{2+}]_F}{[Ni^{2+}]_A \cdot [Co^{2+}]_F}$
4.40	183.222	0.016	11450
5.00	303.688	0.028	10850
5.60	5635.556	0.566	9960

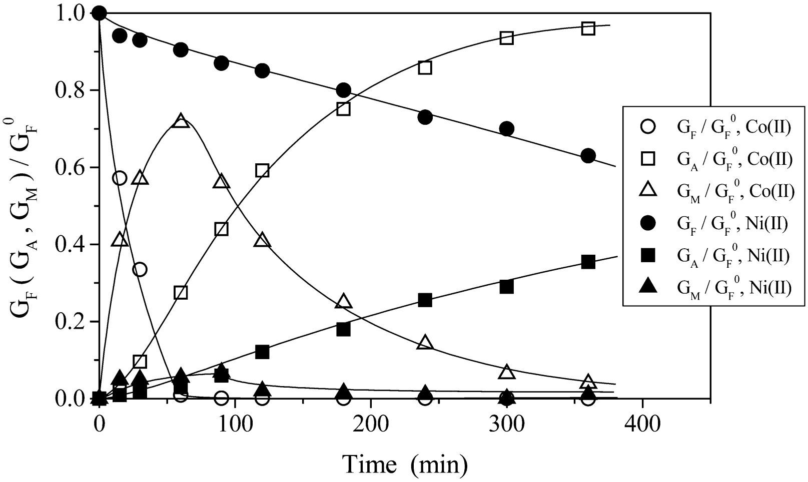

Figure 6. Evolution of dimensionless metal amount in feed, membrane, and acceptor solutions vs. time (the feed solution contained both metals, $[R_2PSOH]_M = 0.1\,M$, $pH_F = 5.60 \pm 0.05$).

favorable for both metals recovery was studied, also. For this purpose, the feed solution, containing both 0.01 M Co(II) and 0.01 M Ni(II), was buffered by ammonium sulphate/ammonia buffer solution to $pH_F = 7.10 \pm 0.05$. As an acceptor solution 1.0 M H_2SO_4 and as a membrane solution 0.2 M R_2PSOH in kerosene were used, respectively. The obtained results, shown in Fig. 7,

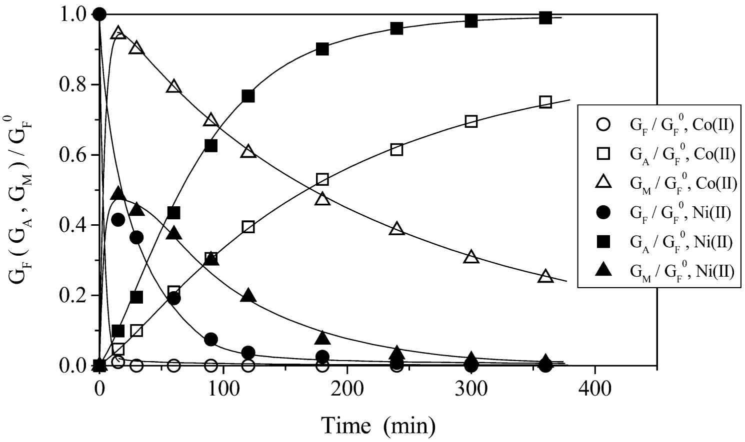

Figure 7. Evolution of dimensionless metal amount in feed, membrane, and acceptor solutions vs. time (the feed solution contained both metals, $[R_2PSOH]_M = 0.2\,M$, $pH_F = 7.10 \pm 0.05$).

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establish that, at these conditions, both Co(II) and Ni(II) can be successfully recovered. The extraction of Co(II) at these conditions was immediate, but its accumulation in the acceptor solution was hampered by the competing transport of present Ni(II). A comparison between the curves representing the evolution of Co(II) and Ni(II) amount in the membrane phase shows that the stripping of loaded with Ni(II) ions carrier was easier than the loaded with Co(II) ions. The higher transfer of metal ions to the acceptor phase in this case provoked a noticeable diminution of the acceptor phase acidity. Consequently, the thermodynamic conditions created at the second interface M/A were not suitable for complete decomposition of the stronger, cobalt-carrier complex and total accumulation of Co(II) ions in the acceptor solution. Thus, the described pertraction process led to an entire removal of both Co(II) and Ni(II) ions form the feed solution, but only Ni(II) ions were totally accumulated in the acceptor solution. To guarantee a complete stripping of the loaded carrier and accumulation of both metals in the acceptor phase the use of a stronger acidic solution as acceptor phase is necessary.

Any modification in metals charge during the pertraction processes was not observed.

The loaded with Co(II) organic phase, containing Cyanex 302, can be successfully stripped by cobalt-spent electrolyte, as proposed Tsakiridis et al. (30). Therefore, the described pertraction process allows obtaining of aqueous solutions suitable for cobalt and nickel electrowinning. Cobalt can be extracted from the feed and accumulated in the acceptor solution (cobalt spent electrolyte) applying pertraction in rotating film contactor at conditions suitable for cobalt separation from the present nickel ($[R_2PSOH]_M = 0.1\,M$, $[H_2SO_4]_A = 1.0\,M$, $pH_F = 4.4$, $T = 293\,K$). The obtained acceptor solution, enriched in Co(II), is suitable for cobalt electrowinning. In the second step, after complete cobalt removal from the feed phase, this solution, rich in nickel, is buffered to $pH = 7.0$ and used as a donor solution in another pertraction process with nickel spent electrolyte as strip solution ($[R_2PSOH]_M = 0.1\,M$, $[H_2SO_4]_A = 1.0\,M$, $pH_F = 7.0$, $T = 293\,K$). The enriched in Ni(II) acceptor solution could be a subject of nickel electrowinning, also.

CONCLUSIONS

The study of extraction behavior of three extractants from Cyanex series established that the Cyanex 302, with chief constituent the bis(2,4,4-trimethylpentyl) monothiophosphinic acid, is the most suitable extracting agent for Co(II)/Ni(II) separation, as well as for the recovery of both metals from sulphate media. Co(II) and Ni(II) can be successfully separated applying pertraction in a rotating film contactor with Cyanex 302 as a carrier. The pH_F interval between 4.0 and 6.0 was found to be the most appropriate for

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privileged Co(II) recovery. At these conditions, Ni(II) remained mainly in the feed solution. The obtained separation factors ($K_{sep} \sim 1.0 \times 10^4$) demonstrate that the proposed pertraction process using Cyanex 302 as a carrier guarantees an excellent Co(II)/Ni(II) separation. Higher pH of the feed solution favors the recovery of both metals, but deteriorates separation ability. At well-chosen operational conditions (neutral pH of the feed solution and strongly acidic acceptor solution), pertraction process can be used for simultaneous and complete recovery of both metals, also.

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