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APPLIED ELECTROCHEMISTRY AND CORROSION PROTECTION OF METALS

Electrodialytic Recovery of Cobalt(II) with Liquid Membranes Based on Di(2-ethylhexyl)phosphoric Acid

T. Zh. Sadyrbaeva

Institute of Inorganic Chemistry, Riga Technical University, Salaspils, Latvia

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Abstract—Electrodialytic transfer of cobalt(II) from sulfuric acid solutions across liquid membranes containing di(2-ethylhexyl)phosphoric acid with addition of tri-*n*-octylamine in 1,2-dichloroethane was studied, with the metal electrodeposited from dilute solutions of various acids. The influence exerted by the electrodialysis current density and by the composition of aqueous solutions and liquid membranes on the rate of cobalt(II) transport and electrodeposition was examined.

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Cobalt is widely used in various industries as an alloying additive to steels and a component of magnetic alloys and catalysts, glass and ceramics making, medicine, manufacture of batteries, and production of paints and lacquers [1]. Cobalt(II) cations are commonly extracted from neutral and weakly acid solutions with acid organophosphorus reagents, such as di(2-ethylhexyl)phosphoric acid (D2EHPA) [2]. This reagent is also frequently used in membrane extraction as a cation-exchange carrier of metal ions [3]. To recover cobalt(II) from dilute solutions, D2EHPA has been used in various membrane extraction processes with emulsion [4], impregnated [5–8], bulk [9, 10], and combined [11] liquid membrane. Metal is transported in these systems as oppositely directed transports of Co²⁺ ions and hydrogen cations:

$$\operatorname{Co}_{(\operatorname{aq})}^{2+} + 2\operatorname{HR}_{(\operatorname{org})} \leftrightarrows \operatorname{CoR}_{2(\operatorname{org})} + 2\operatorname{H}_{(\operatorname{aq})}^{+}, \qquad (1)$$

where HR is D2EHPA, aq denotes the aqueous phase, and org, the organic phase.

The driving force of the transfer process is the pH gradient between the donating and receiving solutions, and, therefore, a strongly acid receiving solution is used. When a dc electric field is applied to the membrane system in the course of electrodialysis, the transport of

metal cations is combined with the transfer of hydrogen ions. In this case, there is no need to use a strongly acid receiving solution, the ion transfer across the membrane is intensified, and it becomes possible to isolate from the receiving solution the metal being recovered as a cathode deposit. It has been found previously that the D2EHPA-based liquid membranes under study provide effective single-stage extraction of metals into dilute solutions of various acids [12, 13]. The process of membrane extraction of silver(I) ions from nitric acid solutions under galvanostatic electrodialysis conditions with electrodeposition of the metal in the receiving solution has been studied and it was shown that the best quality cathode deposits of silver are formed in electrodeposition from 0.05 HClO₄ solutions [14].

The goal of the present study was to examine the electrodialytic transport of cobalt(II) ions from sulfuric acid solutions with liquid membranes based on technicalgrade D2EHPA with addition of tri-*n*-octylamine (TOA) in galvanostatic electrodialysis and to determine the optical conditions of extraction and electrodeposition of cobalt in the receiving solution.

EXPERIMENTAL

The process of electrodialysis of cobalt(II) ions was

membranes confined between vertical cellophane films in the system

$$(+) Pt 0.15 M H_2 SO_4 \qquad CoSO_4 \qquad D2EHPA \qquad Pt cathode solution (-) H_2 SO_4 \qquad TOA \qquad MA$$

studied in a four-chamber fluoroplastic cell with a liquid The process was performed in the galvanostatic mode, with a copper cathode occasionally used. The volume and thickness of the liquid membrane were 2 cm³ and 0.2 cm; the phase contact area was 7.1 cm². The volume of the electrode chambers was 17 cm³, and the volume of the receiving solution, 13 cm³. The anode chamber was separated from the chamber with the receiving solution by a MA-40 solid anion-exchange membrane. The cellophane films and solid membranes were preliminarily soaked in water. The role of the cathode solution was commonly played by 2.5×10^{-2} M HClO₄.

As the liquid membrane served solutions of technicalgrade D2EHPA (~63% the main substance) with addition of TOA (pure grade) in 1,2-dichloroethane. As a rule, 20 or 32 vol % solutions of D2EHPA with addition of 0.1 M of TOA were used. The donating solution was prepared by dissolution of cobalt sulfate CoSO₄·7H₂O (analytically pure grade) in sulfuric acid of the required concentration, as a rule, 0.01 M H_2SO_4 . The initial concentration of cobalt(II) was commonly 0.01 M. The content of cobalt(II) ions in aqueous solutions was determined spectrophotometrically with potassium rhodanide [15]. The optical density was measured with an SF-46 spectrophotometer. The properties of cathode deposits of cobalt were examined with a portable scanning electron microscope SEC Mini-SEM (South Korea).

In organic solutions, D2EHPA interacts with TOA to give an ion pair comprising an organic cation and an organic anion [16]:

$$HR + Oct_3N \leftrightarrows Oct_3NH^+R^-, \qquad (2)$$

where $Oct_3N = TOA$, and HR = D2EHPA.

The extraction of cobalt(II) into the liquid membrane can be described by the equation

$$Co_{(aq)}^{2+} + SO_{4(aq)}^{2-} + 2 \operatorname{Oct}_{3} \operatorname{NHR}_{(org)}$$
$$\Leftrightarrow CoR_{2(org)} + (\operatorname{Oct}_{3} \operatorname{NH})_{2} SO_{4(org)}.$$
(3)

In a polar organic solvent, the cobalt(II) complex being extracted is partly dissociated [17]:

$$\operatorname{CoR}_{2(\operatorname{org})} \leftrightarrows \operatorname{Co}_{(\operatorname{org})}^{2+} + 2R_{(\operatorname{org})}^{-}.$$
 (4)

Co²⁺ cations are transported via diffusion from the bulk of the donating solution to the interface with the liquid membrane and interact with the carrying agent. The CoR₂ complex being extracted diffuses across the liquid-membrane layer and dissociates at the interface with the receiving solution by a reaction reverse to (3). Co^{2+} cations appearing in the organic phase as a result of the partial dissociation of the complex being extracted are transferred across the liquid membrane due to electromigration. The carrying agent molecules diffuse back to the interface with the donating solution because of the concentration gradient. Hydrogen ions are transported across the liquid membrane into the receiving solution in the same direction as that for cobalt(II) cations by interacting with the carrying agent by the interphase reaction

$$2H^{+}_{(aq)} + SO_{4}^{2-}_{(aq)} + 2 \operatorname{Oct}_{3} \operatorname{NHR}_{(org)}$$
$$\Rightarrow 2HR_{(org)} + (\operatorname{Oct}_{3} \operatorname{NH})_{2} SO_{4(org)}.$$
 (5)

If an HX acid is contained in the receiving solution, X- anions interact with the extractive agent at the interface between the liquid membrane and the receiving solution, are transported across the organic phase in the reverse direction due to electromigration, and pass into the donating solution:

$$X_{\overline{(aq)}} + H_{(aq)}^{+} + Oct_{3}NHR_{(org)}$$

$$\Rightarrow Oct_{3}NHX_{(org)} + HR_{(org)}.$$
 (6)

It was found in preliminary experiments that, with dilute perchloric or sulfuric acid used as the receiving solution under electrodialysis conditions, cobalt(II) ions are transferred across the liquid membrane and the metal is electrodeposited at the cathode. The rate of Co^{2+} extraction grows with the current density increasing

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	<i>t</i> , min	Receiving solution, M	E	R	D	$J \times 10^{5}$,
<i>i</i> , mA cm ⁻²			%			mol m ⁻² s ⁻¹
2.1	175	$2.5 \times 10^{-2} \operatorname{HClO}_4$	99.7	32	45	1.3
2.8	98	$2.5 \times 10^{-2} \text{ HClO}_4$	99.7	41	27	2.1
4.2	55	$2.5 \times 10^{-2} \text{ HClO}_4$	99.0	36	6	2.4
5.7	40	$2.5 \times 10^{-2} \text{ HClO}_4$	98.4	30	2	2.4
2.8	107	$2 \times 10^{-2} \operatorname{H_2SO_4}$	100.0	64	15	2.3
4.2	46	$1 \times 10^{-2} \text{ H}_2 \text{SO}_4$	99.1	44	9	3.6
5.7	34	$0.1 \text{ H}_2 \text{SO}_4$	98.0	25	3	2.5

Table 1. Degree of extraction of cobalt(II) ions into the liquid membrane (*E*) and receiving solution (*R*), degree of electrodeposition (*D*), and flux *J* at various current densities. $c_{\text{D2EHPA}} = 20 \text{ vol }\%$, $c_{\text{TOA}} = 0.1 \text{ M}$

within the range 0–4.2 mA cm⁻². Further increase in the current density has no effect on the flux of metal ions across the liquid membrane (Table 1). A nearly full extraction of cobalt(II) from the starting solution containing 0.01 M of $CoSO_4$ is reached after 0.5–3 h of electrodialysis, depending on the current density. At high current density, the electrodialysis duration becomes shorter, but the extraction of cobalt(II) into the cathode solution and the degree of electrodeposition of the metal decrease. The maximum degree of electrodeposition of cobalt (~45%) in extraction of more than 75% of the metal into the receiving solution and nearly full extraction of Co^{2+} cations from the starting solution was obtained at a current density of 2.1 mA cm⁻².

As a rule, the process of galvanostatic electrodialysis ends in a sharp rise in the voltage in the system, with the instant of time at which this occurs being dependent on the current density (Fig. 1). It was found that the sharp decrease in the electrical conductivity of the system corresponds to a nearly full (\geq 98%) extraction of cobalt(II) into the organic phase and is due to desalination of the donating solution because of the transfer of Co²⁺ and H⁺ cations into the liquid membrane and transport of SO₄²⁻ anions across the solid anionexchange membrane into the anode chamber.

The current efficiency by transport of Co^{2+} cations in the system with 0.01 M of $CoSO_4$ is about 20% at current densities in the range 2.1–5.7 mA cm⁻². Charge is carried across the liquid membrane mostly by hydrogen cations from the donating solution and by anions (perchlorate ions or sulfate ions) from the receiving solution. Figure 2 shows kinetic dependences for the electrodialytic transport and electrodeposition of cobalt(II) ions at a moderate current density of 2.8 mA cm⁻². Under these conditions, full extraction of metal ions from the starting solution into the liquid membrane requires about 100 min. During this time, approximately 40% of cobalt(II) accumulates in the receiving solution and more than 25% of the metal is deposited on the cathode. The concentration of Co²⁺ cations in the cathode solution steadily grows in the course of the process, whereas in the organic phase, cobalt(II) accumulates till 80th min, after which the content of the metal in the liquid membrane falls. This



Fig. 1. Effect of the current density on the variation kinetics of the voltage *U*. $c_{\text{D2EHPA}} = 20$ vol %, $c_{\text{TOA}} = 0.1$ M, cathode solution 2.5×10^{-2} M HClO₄. (*t*) Process duration; the same for Figs, 2, 4, and 5. *i* (mA cm⁻²): (*1*) 5.7, (*2*) 4.2, (*3*) 2.8, and (*4*) 2.1.

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Fig. 2. Variation kinetics of content c_{Co} of cobalt(II) in (1) starting solution, (2) cathode solution, and (3) liquid membrane; and (4) electrodeposition of cobalt(II). $i = 2.8 \text{ mA cm}^{-2}$, $c_{D2EHPA} = 20 \text{ vol }\%$, $c_{TOA} = 0.1 \text{ M}$.

time corresponds to the beginning of a steep rise in the voltage across the system (Fig. 1, curve 3). It can be seen in Fig. 2 that, only after 80 min of the process, as the voltage increases due to a decrease in the electrolyte concentration in the donating solution, the rate of cobalt re-extraction from the liquid membrane starts to grow and the metal electrodeposition begins.

With the cobalt(II) concentration in the donating sulfuric acid solution increasing from 1×10^{-3} to 0.05 M, the rate of transfer of Co²⁺ ions across the liquid membrane grows; further increase in the concentration does not change the flux (Fig. 3a, curve 3). The degrees of metal extraction into the liquid membrane and into the receiving solution decrease as the initial concentration of cobalt(II) is raised. The highest current efficiency is about 24% at a metal concentration of 0.05 M in the starting solution. It should be noted that, in a system with low cobalt(II) concentration (1×10^{-3} M), the voltage at a given current density increases to the maximum extent already after 40 min of electrodialysis as a result of the full extraction of the metal into the liquid membrane.

An increase in the content of sulfuric acid in the donating solution within the range from 1×10^{-3} to 0.1 M leads to a substantial decrease in the degrees of extraction and rate of cobalt(II) transport across the liquid membrane (Fig. 3b). This is due to the decrease in the extracting capacity of D2EHPA upon an increase in the acidity of the aqueous phase. The optimal acidity of the starting cobalt(II) solution is 1×10^{-3} – 1×10^{-2} M of H₂SO₄. Under these conditions, the metal is quantitatively recovered from the starting solution with



Fig. 3. (a) Degrees of cobalt(II) extraction into (1) liquid membrane, E, and (2) cathode solution, R, and the transfer rate J vs. the concentrations of (a) cobalt(II), c_{Co} , and (b) sulfuric acid, $c_{\text{H}_2\text{SO}_4}$, in the starting solution. i = 2.8 mA cm⁻², t = 60 min, $c_{\text{D2EHPA}} = 32$ vol %, $c_{\text{TOA}} = 0.1$ M, cathode solution 2.5×10^{-2} M HClO₄; the same for Fig. 4.

a sufficiently high degree of re-extraction (R = 70-75%) at a moderate current density.

The influence exerted by the content of the carrying agent D2EHPA and by that of the additive TOA on the process of cobalt(II) extraction is illustrated by the data in Table 2. Variation of the D2EHPA concentration in the organic phase from 5 to 50 vol % at a constant concentration of the amine only slightly affects the rate of cobalt(II) extraction into the liquid membrane and on the rate of transmembrane transfer of metal ions. Raising the content of TOA in the liquid membrane from 0.05 to 0.4 M at a constant concentration of the D2EHPA carrying agent has no significant effect of the degree of cobalt(II) extraction into the organic phase, but results in that the rate of Co²⁺ ion transport across the liquid membrane somewhat decreases. An increase in the amine concentration enhances the transfer of perchlorate ions across the membrane from the receiving



Fig. 4. Effect of the concentrations of the carrying agent D2EHPA and TOA additive on the variation kinetics of the voltage *U*. c_{D2EHPA} (vol %): (1) 50, (2–4) 32, and (5) 5. c_{TOA} (M): (1, 5) 0.1, (2) 0.05, (3) 0.2, and (4) 0.4.

solution and leads to a decrease in the current efficiency by cobalt(II) ions.

The electrical conductivity of a system with a liquid membrane is, as a rule, determined by the composition of the organic phase. Solutions of technical-grade D2EHPA have low electrical conductivity because of containing a considerable amount (~16%) of a strongly associated monoalkylphosphoric acid [2]. Therefore, it is necessary to introduce electrically conducting additives into the liquid membrane. An increase in the TOA concentration in the liquid membrane results in that the electrical conductivity grows and the voltage in the system decreases, whereas raising the content of the carrying agent D2EHPA, by contrast, makes the electrical conductivity of liquid membranes lower (Fig. 4). As a rule, the voltage in the system gradually increases in the course of electrodialysis as a result of the decrease in the concentration of ions in the organic phase due to extraction of the neutral complex CoR₂. After the full extraction of Co²⁺ ions, the voltage sharply grows because of the desalination of the starting aqueous solution. In the case of a low D2EHPA concentration in the liquid membrane (5 vol %), the voltage starts to strongly grow beginning in the first minutes of electrodialysis (Fig. 4, curve 5). In this system, a deposit is formed on the cellophane film at the interface between the liquid membrane and the receiving solution.

The data in Table 3 illustrate the influence exerted by the composition of the receiving solution on the extraction and electrodeposition of cobalt(II). Co²⁺

Table 2. Electrodialytic transport of cobalt(II) ions at various liquid membrane compositions. i = 2.8 mA cm⁻², t = 60 min, receiving solution 2.5×10^{-2} M HClO₄

c _{D2EHPA} , vol %	с _{тоа} , М	E R %		$J imes 10^5$, mol m ⁻² s ⁻¹
5	0.1	68	17	0.9
20	0.1	54	14	0.7
32	0.1	59	17	0.9
50	0.1	69	15	0.8
32	0.05	65	17	0.9
32	0.2	59	13	0.7
32	0.4	61	14	0.7

cations are transferred across the liquid membrane with approximately the same efficiency into 1×10^{-2} M solutions of perchloric, sulfuric, hydrochloric, and nitric acids. The electrodeposition rate is the highest in the system with perchloric acid, whereas in solutions of sulfuric and nitric acids, no cathode deposit is formed after 45 min of electrodialysis. At a lower acid concentration $(1 \times 10^{-3} \text{ M})$, the organic phase appears in the aqueous solution in the course of the process. Under conditions of insufficient electrolyte content, organic cations start to be involved in the charge transport across the interface between the liquid membrane and the receiving solution. Raising the concentration of perchloric acid from 1×10^{-2} to 2.5×10^{-2} M leads to a decrease in the degree of cobalt(II) extraction, with no electrodeposition of the metal observed in this case. Such an influence of the pH of the receiving solution on the transfer rate distinguishes the electrodialysis process from the conventional membrane extraction with D2EHPA, which requires that the acidity of the reextracting solution should be high. An increase in the HClO₄ concentration enhances the flux of ClO₄⁻ anions across the liquid membrane, which leads to a lower current efficiency by Co²⁺ ions.

Raising the acid concentration in the receiving solution strongly affects the electrical conductivity of the membrane system and the run of kinetic curves describing the voltage variation (Fig. 5). In systems containing 10^{-4} to 2×10^{-2} M of H₂SO₄, the electrodialysis duration is limited by the sharp rise in voltage as a result of the

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t, min	Possiving solution M	Е	R	D	$J \times 10^5$,	
	Receiving solution, M		mol m ⁻² s ⁻¹			
45	$1 \times 10^{-2} \operatorname{HClO}_4$	52	3	7	0.7	
	1×10^{-2} HCl	57	12	3	1	
	$1 \times 10^{-2} \text{ H}_2 \text{SO}_4$	59	15	-	1	
	1×10^{-2} HNO ₃	58	10	-	0.7	
60	$1 \times 10^{-3} \text{ H}_2 \text{SO}_4$	88	Organic phase in solution			
	$1 \times 10^{-2} \mathrm{H}_2 \mathrm{SO}_4$	83	39	9	2.5	
	1×10^{-2} HCl	77	28	8	1.8	
	$2.5 \times 10^{-2} \operatorname{HClO}_4$	54	14	_	0.7	
		1	1			

Table 3. Electrodialytic transport of cobalt(II) ions in relation to the composition of the receiving solution. i = 2.8 mA cm⁻², $c_{\text{D2EHPA}} = 20$ vol %, $c_{\text{TOA}} = 0.1$ M



Fig. 5. Effect of the H₂SO₄ concentration in the cathode solution on the variation kinetics of the voltage U. c_{H2SO4} (M): (1) 1×10^{-4} , (2) 1×10^{-3} , (3) 2×10^{-2} , and (4) 0.1.



Fig. 6. Surface micrograph of a cobalt coating deposited from 2.5 M $HClO_4$ on a copper cathode at a current density of 6 mA cm⁻².

full extraction of the metal into the liquid membrane and desalination of the starting solution in several tens of minutes of the process, depending on the acid content. At a higher H_2SO_4 concentration, the gradual rise observed in the chronopotentiogram gives way to a decrease in the voltage because of the accumulation of water in the liquid membrane phase (Fig. 5, curve 4). Water is transferred into the organic phase in hydrate-solvate shells of the ions being extracted and due to electroosmosis [18]. The accumulation of water adversely affects the transport properties of liquid membranes.

Cobalt deposits on the platinum and copper cathodes, obtained in the course of electrodialysis, are dark compact coatings firmly adherent to the electrode. The properties of the cathode deposits were examined by scanning electron microscopy. The deposits obtained at a current density of 6 mA cm⁻² (the current density calculated per the liquid membrane area is 2.1 mA cm⁻²) have a finely crystalline structure (Fig. 6). A twofold increase in the current density makes the coatings more porous.

CONCLUSIONS

(1) In galvanostatic electrodialysis with liquid membranes containing di(2-ethylhexyl)phosphoric acid under the optimal conditions, the degree of electrodeposition of the metal reaches a value of 45% and the degree of re-extraction into the receiving solution exceeds 75%, with full extraction of cobalt(II) from the starting solution.

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(2) The rate of Co^{2+} extraction grows as the current

density is raised within the range 0-4.2 mA cm⁻² and

the cobalt(II) concentration in the donating solution

increases within the range from 1×10^{-3} to 0.05 M

and is only slightly changed when the concentration of

di(2-ethylhexyl)phosphoric acid in the organic phase is

varied from 5 to 50 vol %. An increase in the sulfuric acid

concentration in the starting solution (1 \times 10⁻³–0.1 M

and in that of tri-n-octylamine in the liquid membrane

(0.05–0.4 M) adversely affects the transmembrane

membrane into 1×10^{-2} M solutions of perchloric,

sulfuric, hydrochloric, and nitric acids are approximately

the same. As the acid concentration is lowered, the

organic phase may appear in the aqueous solution.

An increase in the acid concentration in the receiving

solution causes a decreases in the degrees of cobalt(II)

and copper electrodes as dark compact coatings with

finely crystalline structure, firmly adherent to the

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