

Cobalt (II) membrane-extraction by DP-8R/Exxsol D100 using pseudo-emulsion based hollow fiber strip dispersion (PEHFSD) processing

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ABSTRACT

The transport of cobalt(II) from acidic sulphate solutions through PEHFSD was investigated using the extractant DP-8R in Exxsol D100. The acidic feed solution containing Co(II) was passed through the tube side, and pseudo-emulsions of DP-8R/Exxsol D100 and sulphuric acid was passed through the shell side in counter-current mode, using a single microporous hydrophobic polypropylene hollow fiber contactor for extraction and stripping. In PEHFSD the aqueous strip (sulphuric acid) solution was dispersed in the organic membrane solution in a reservoir tank with an impeller stirrer to form a strip dispersion. This pseudo-emulsion phase is circulated from the tank to the membrane module to provide a constant supply of the carrier solution to the membrane micropores. Several hydrodynamic and chemical parameters, such as variation in feed pH (3-7), cobalt concentration in feed ($0.17\text{-}1.7 \times 10^{-3}$ M), carrier concentration (0.16-1.28 M), etc., were investigated. Mass transfer modelling was performed and the validity of the model was evaluated with experimental data. Moreover, the system was

applied in the cobalt purification of synthetic lithium solutions derived from the processing of rechargeable batteries, with cobalt/lithium separation factor values around 25.

Keywords: Cobalt(II), DP-8R, Pseudo-emulsion based hollow fiber strip dispersion (PEHFSD), Lithium(I)

1. Introduction

Cobalt is a metal which is used in a wide number of industries, that due to its prize and re-use possibilities, it is of interest its recovery from the wastes that these industries generated.

In the hydrometallurgical processing of these wastes, recently developed membrane-extraction processes are of particular interest because of their versatility and the fact that they overcome problems encountered in other established separation technologies, i.e. liquid-liquid extraction. Thus, there are investigations to separate cobalt(II) from aqueous environments using liquid membrane technologies [1-8].

Also, to overcome some problems (stability and long-term performance) of several liquid membranes technologies, pseudo-emulsion based hollow fiber strip dispersion (PEHFSD) membrane processing, and other related membrane-dispersion-based processes, had been developed to provide the membrane separation system with an acceptable level of stability and also resulted in consistent performance. Recent references about the use of these technologies can be found elsewhere [9-23].

Since the survey of the literature suggests that, to date, no report is available concerning the use of a smart liquid membrane technology such as PEHFSD methodology for

cobalt transport and its application to the separation of Co(II)/Li(I), the present work studies the transport of Co(II) by PEHFSD, using DP-8R as mobile carrier. The influence of the various process variables, such as hydrodynamic conditions, metal and carrier concentration, pH of the feed solution, etc., on the metal transport in the PEHFSD processing have been investigated and a permeation model describing the transport mechanism is reported in order to optimize performance of hollow fiber module for the PEHFSD of Co(II) from acidic solutions. The system was used to separate Co(II) from Li(I) and the performance of DP-8R extractant was compared against two other acidic carriers such as Cyanex 272 and Acorga PT5050.

2. Experimental

2.1. Materials

DP-8R (di(2-ethylhexyl) phosphoric acid, Daihachi) was used as carrier for transport experiments in this investigation. Exxsol D100 (>99% aliphatic content, Exxon Chem. Iberia, Spain) was used as diluent for the organic phase. Other carriers used in this work were: Cyanex 272 (bis(2,4,4-trimethylpentyl) phosphinic acid, CYTEC) and Acorga PT5050 (2-hydroxy-5-nonylbenzaldehyde oxime, Avecia). All these organic compounds were used as received, whereas all other chemicals used in the present study were of AR grade. Stock cobalt(II) solution was prepared by dissolving the required amount of cobalt(II) sulphate (Fluka) in deionized water. The organic solution was prepared by dissolving the appropriate volume of the extractant in Exxsol D100 to obtain organic phases of different concentrations. The hollow fiber device used for the investigation was obtained from Membrana: Liqui-Cel 2.5x8 5PCG-354 contactor and

5PCS-1002 Liqui-Cel LLE). The main characteristics of the contactor are given in Table 1.

2.2. PEHSD experiments

The methodology used in the present investigation was the same as described in a previous work [15]. The view of PEHFSD using a single contactor in recirculation mode is shown in Fig. 1.

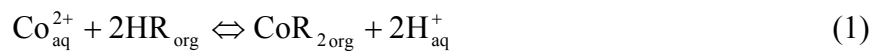
The volume of pseudo-emulsion phase used in the investigation was 400 cm³ (200 cm³ of the organic solution + 200 cm³ of the H₂SO₄ solution), whereas 2000 cm³ of feed solution were used throughout the transport experiments. Previous experiments had shown that 0.1 M H₂SO₄ solution is suitable for cobalt(II) recovery from cobalt-loaded DP-8R solutions. The pH value of the various feed phases used in this investigation was controlled by adding to the feed reservoir tank aliquots of NaOH or H₂SO₄ solutions when necessary.

All the transport experiments were carried out at 20° C.

Fibers permeability and metal recovery in the strip solution were determined by monitoring metal concentration by AAS (Perkin Elmer 1100B spectrophotometer) in the feed or strip solutions as a function of time.

3. Results and discussion

The extraction of cobalt(II) by an acidic extractant such as DP-8R can be represented in a simplified form by the next reaction:



where the subscripts aq and org represented to the aqueous and organic solutions, respectively. Thus, and in accordance with the above, the transport of cobalt(II) with this extractant is related to a counter-transport mechanism, where the difference in protons concentration between the feed and stripping solutions is the driving force for the transport process.

The cobalt flux, J_{Co} , through the module is given by:

$$J_{Co} = K \left[[Co]_f - \frac{D_{st}}{D_f} [Co]_{st} \right] \quad (2)$$

where K is the overall mass transfer coefficient, $[Co]_f$ and $[Co]_{st}$ are the metal concentrations in the feed and strip solutions, respectively, D_f is the cobalt distribution coefficient in the extraction process and D_{st} is the respective one in the stripping side. Since in the practice D_f is greater than D_{st} , the second term between the squares in the above equation can be neglected with respect to $[Co]_f$, and the material balance on the feed solution is:

$$-V \frac{d[Co]_f}{dt} = AK[Co]_f \quad (3)$$

where V is the volume of the feed solution, A is the effective membrane area in the module and t is the time.

From the above:

$$\ln \frac{[Co]_{f,t}}{[Co]_{f,0}} = -\frac{AK}{V} t \quad (4)$$

where $[Co]_{f,t}$ and $[Co]_{f,0}$ are the cobalt concentrations in the feed solution at time t and time zero, respectively.

However, under several experimental conditions, equation (4) is no longer valid, and the next equation is derived [24]:

$$[\text{Co}]_{f,0} - [\text{Co}]_{f,t} = \frac{[\text{HR}]k_m A}{nV} t \quad (5)$$

where $[\text{HR}]$ is the carrier concentration in the organic phase, n is the number of carrier molecules bound to the metal, and k_m is the membrane mass transfer coefficient. In this situation the metal flux is constant with time and plotting $[\text{Co}]_{f,t}$ versus time, a straight line must be obtained allowing to estimate k_m . The slope value of this straight line can be correlated to an initial mass transfer coefficient K_0 , defined as:

$$K_0 = \frac{[\text{HR}]k_m}{n[\text{Co}]_{f,0}} \quad (6)$$

The transport experiments carried out in this investigation at 1.7×10^{-4} M Co(II), gave trends as those shown in Fig. 2. As can be seen, at the beginning of the transport process there is an induction period and the plots of $\ln [\text{Co}]_{f,t}/[\text{Co}]_{f,0}$ versus time present a bending, but in the course of the run a straight line is obtained. Thus, in the first portion of the graphs, the permeability is entirely controlled by diffusion, and a plot of $[\text{Co}]_{f,t}$ versus time should give a straight line as derived by Eq. (5) allowing to the calculation of k_m and K_0 . In the region where representing $\ln [\text{Co}]_{f,t}/[\text{Co}]_{f,0}$ versus time a linear trend is observed, the overall mass transfer coefficient can be determined from the slope of the linear plot and Eq. (4).

3.1. Influence of the feed and pseudo-emulsion phases flow rate

Preliminary experiments were conducted to establish adequate hydrodynamic conditions. The permeability of the hollow fibers was studied as a function of the flow on the feed solution side, keeping constant the flow of the pseudo-emulsion phase at $100 \text{ cm}^3 \text{ min}^{-1}$.

Co(II) overall mass transfer coefficient ($K = 4.8 \times 10^{-5} \text{ cm s}^{-1}$) becomes virtually independent of the feed flow above $400 \text{ cm}^3 \text{ min}^{-1}$. This implies that the thickness of the feed phase boundary layer reached a minimum value. On the other hand, variation of the pseudo-emulsion phase flow rate ($100\text{-}400 \text{ cm}^3 \text{ min}^{-1}$) has a negligible effect on the overall mass transfer coefficient value.

3.2. Influence of DP-8R concentration on Co(II) permeation

$\ln [\text{Co}]_{f,t}/[\text{Co}]_{f,0}$ versus time plots at different carrier concentrations are reported in Fig.2. As mentioned before, two zones may be clearly differentiated in the experimental trend.

Considering the straight line zone, it can be concluded that a limiting slope corresponding to an overall mass transfer coefficient is obtained in this figure for all the plots. This fact is corroborated in Table 2, where the overall mass transfer coefficients at different carrier concentrations are given. Because K is independent of DP-8R concentration this region is representative of an aqueous diffusion film controlled transport process.

In the first zone of the curves, at short elapsed times, the membrane permeability is entirely controlled by diffusion. In this zone, Eq. (5) holds and a plot of $[\text{Co}]_{f,t}$ versus time gives straight lines as can be seen in Fig. 3. Calculating the slopes of the lines, K_0 values can be estimated by means of Eq. (6). The K_0 versus $[\text{HR}]_{\text{org},0}$ plot of Fig. 4 indicates that a linear tendency is achieved within the present DP-8R concentration range as could be expected from Eq.(6).

The slopes of the straight lines and Eq.(5) allow also to evaluate the membrane mass transfer coefficient. The calculated values are listed in Table 3. Though these values are

approximate due to the assumptions involved in their calculations, it seems that k_m values stand near constant within the present experimental conditions.

3.3. Influence of the diluent

The membrane diluent chosen as a water-resistant barrier in any liquid membrane process influences the membrane performance. In Fig. 5, the effect of the diluent on Co(II) transport is represented and Solvesso 100 is the diluent giving the best overall mass transfer coefficient compared with Exxsol D100. The different hollow fiber performance, using these two organic diluents, can be explained in terms of intrinsic diluent properties such as viscosity, volatility, surface tension, water solubility, etc.

3.4. Influence of the metal concentration

The influence of the metal concentration on the fibers permeation was also investigated and the results are presented in Fig. 6. The induction period was observed at every initial Co(II) concentration in the feed phase with the experimental data available, however it can be clearly observed that the overall mass transfer coefficient decreases as the initial cobalt(II) concentration in the feed phase increases. This should be attributable to an increase on the overall mass transport resistance at high metal initial concentrations which is related to the expected decrease of the distribution coefficient as the initial metal concentration in the feed phase increases.

3.5. Influence of the pH of the feed solution

In order to assess the role of feed phase pH, pH variation studies in the range 3.0 to 7.0 were carried out. The pseudo-emulsion phase consists of an organic solution of 0.36 M

DP-8R in Exxsol D100 and a stripping solution of 0.1 M H_2SO_4 . It is evident from Fig. 7 that the transport of cobalt increases with an increase in pH from 3.0 to 5.0, though at higher pH values it remained unaffected ($K = 4.8 \times 10^{-5} \text{ cm s}^{-1}$), this situation being representative of a transport process controlled by diffusion in the stagnant film of the feed solution [22].

3.6. Influence of feed, stripping and organic solutions volumes variation on Co(II) transport

Table 4 shows the effect of varying different volume ratios such as feed to stripping and feed to organic phases, under standard experimental conditions. Co(II) transport increased up to a ratio of 2.5/1 (feed/strip) and the value of K increased from 4.8×10^{-5} to $8.4 \times 10^{-5} \text{ cm s}^{-1}$. On the other hand, when the feed to organic ratio varies from 10/1 to 5/1, the overall mass transfer coefficient increases, thus, increasing the extraction of cobalt(II). This is due to more carrier being available for a fixed volume of feed.

3.7. Separation of Co(II) and Li(I): a case of study

Previous investigations had shown the practical importance that the separation of cobalt(II) from lithium(I) has in the LIB industry [25]. Thus, the present system had been evaluated in order to investigate its performance in such a role.

For these series of experiments, aqueous solutions of $1.7 \times 10^{-4} \text{ M}$ Co(II) containing various lithium concentrations at pH 5.0, were put into contact with a pseudo-emulsion phase composed of an organic solution of 0.36 M DP-8R in Exxsol D100 and a stripping solution of 0.1 M sulphuric acid. Results of these experiments were shown in Table 5, in which it can be seen that the presence of lithium in the aqueous feed

decreases the transport of cobalt, this probably being due to a population or crowd effect [26]. However, the separation factor cobalt/lithium, defined as:

$$\beta_{\text{Co/Li}} = \frac{K_{\text{Co}}}{K_{\text{Li}}} \quad (7)$$

indicated about the selective separation of cobalt and lithium within the present experimental conditions.

Moreover, the performance of the system was evaluated against two other acidic extractants such as Cyanex 272 and Acorga PT5050. The results of these experiments are given in Table 6, showing that the overall mass transfer coefficient is greater in the case of using DP-8R as carrier for the system, whereas the separation factor is similar in the case of DP-8R and Acorga PT5050 extractants and slightly greater than in the case of Cyanex 272.

4. Conclusions

The results of the permeation experiments confirm the validity of the derived model. At shorter elapsed times, when the carrier is almost all bound to Co(II) the mass transfer coefficient is the function of time and the rate of metal transfer is controlled by the diffusion in the membrane. After, when Co(II) concentration in the feed solution decreases metal permeation can be described by a time independent mass transfer coefficient, this situation being representative of an aqueous diffusion film controlled permeation process ($K = 4.8 \times 10^{-5} \text{ cm s}^{-1}$).

From experimental data it can be concluded that experimental conditions, i.e. using a pseudo-emulsion phase of 0.64 M DP-8R in Exxsol D100 and 0.1 M H_2SO_4 are suitable for the efficient extraction (higher than 98%) and stripping (higher than 70%) and

concentration of Co(II) under optimum operational conditions of i.e. pH of feed phase above 5.0.

On the other hand, separation of Co(II)/Li(I) with the required purity ($\beta_{\text{Co/Li}}$ around 25) is possible by the PEHFSD process and using DP-8R as mobile carrier. The performance of the system is better than when using Acorga PT5050 or Cyanex 272 as carriers, whereas Co(II)/Li(I) separation factors are similar in the case of DP-8R and Acorga PT5050 extractants and higher than in the case of Cyanex 272.

This technology is a good alternative to conventional technologies and should increase awareness of the potential for recovery of Co(II) from various sources.

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Table 1

Characteristics of the contactor used in the investigation.

Module length (cm)	28
No. of fibers	10000
Polymeric material	Polypropylene
Active interfacial area (m ²)	1.4
Fiber i.d. (d _i , cm)	20x10 ⁻³
Fiber o.d. (d _o , cm)	30x10 ⁻³
Fiber thickness (d _{org} , cm)	5x10 ⁻³
Fiber length (cm)	15
Porosity (ε)	25%

Table 2

Influence of DP-8R concentration in the organic solution on overall mass transfer coefficient of Co(II)

[DP-8R], M	Kx10 ⁵ , cm s ⁻¹
0.16	4.7
0.32	4.8
0.64	4.7
1.28	4.8

Experimental conditions as in Fig.2

Table 3

Membrane mass transfer coefficients at different carrier concentrations

[DP-8R], M	$k_m \times 10^9, \text{ cm s}^{-1}$
0.16	7.3
0.32	7.5
0.64	7.6
1.28	7.5

Table 4

Experimental values of the overall mass transfer coefficient for different volume ratios

	$K \times 10^5, \text{ cm s}^{-1}$
Feed:strip volume ratio	
10:1	4.8
5:1	6.4
2.5:1	8.4
Feed:organic volume ratio	
10:1	4.8
5:1	6.0

Feed phase: 1.7×10^{-4} M Co(II) at pH 5.0. Pseudo-emulsion phase: 0.32 M DP-8R in Exxsol D100 + 0.1 M H_2SO_4 . Feed flow rate: $400 \text{ cm}^3 \text{ min}^{-1}$. Pseudo-emulsion flow rate: $200 \text{ cm}^3 \text{ min}^{-1}$

Table 5

Cobalt transport in presence of lithium

[Li], M	$K \times 10^5, \text{ cm s}^{-1}$	$\beta_{\text{Co,Li}}$
-	4.8	-
1.7×10^{-4}	4.4	22.0
1.7×10^{-3}	4.1	25.4
1.7×10^{-2}	3.1	28.2

Feed phase: 1.7×10^{-4} M Co(II) and Li(I) at pH 5.0. Pseudo-emulsion phase: 0.32 M DP-8R in Exxsol D100 + 0.1 M H_2SO_4 . Feed flow rate: $400 \text{ cm}^3 \text{ min}^{-1}$. Pseudo-emulsion flow rate: $200 \text{ cm}^3 \text{ min}^{-1}$

Table 6

Cobalt transport in presence of lithium using different acidic carriers

Carrier	$K \times 10^5, \text{ cm s}^{-1}$	$\beta_{\text{Co,Li}}$
DP-8R	4.4	22.0
Cyanex 272	2.4	18.5
Acorga PT5050	1.7	21.5

Feed phase: 1.7×10^{-4} M (each) Co(II) and Li(I) at pH 5.0. Pseudo-emulsion phase: 10% v/v carrier in Exxsol D100 + 0.1 M H_2SO_4 . Feed flow rate: $400 \text{ cm}^3 \text{ min}^{-1}$. Pseudo-emulsion flow rate: $200 \text{ cm}^3 \text{ min}^{-1}$

Fig.1. Schematic view of PEHFSD operated in recycling mode. (1) feed solution into tube side, (2) pumps, (3) flowmeters, (4) hollow fiber membrane contactor, (5) pseudo-emulsion phase (DP-8R/Exxsol D100 and H₂SO₄) into shell side.

Fig.2. Influence of DP-8R concentration in the organic solution on Co(II) transport. Feed phase: 1.7×10^{-4} M Co(II) at pH 5.0. Pseudo-emulsion phase: DP-8R in Exxsol D100 + 0.1 M H₂SO₄. Feed flow rate: $400 \text{ cm}^3 \text{ min}^{-1}$. Pseudo-emulsion flow rate: $200 \text{ cm}^3 \text{ min}^{-1}$.

Fig.3. Total Co(II) concentration in the aqueous feed versus time at short elapsed times. Experimental conditions as in Fig.2.

Fig.4. Influence of DP-8R concentration in the organic solution on initial mass transfer coefficient of Co(II). Experimental conditions as in Fig.2.

Fig.5. Influence of the diluent in the organic solution on Co(II) transport. Feed phase: 1.7×10^{-4} M Co(II) at pH 5.0. Pseudo-emulsion phase: 0.32 M DP-8R in the diluent + 0.1 M H₂SO₄. Feed flow rate: $400 \text{ cm}^3 \text{ min}^{-1}$. Pseudo-emulsion flow rate: $200 \text{ cm}^3 \text{ min}^{-1}$.

Fig.6. Influence of metal concentration on Co(II) transport. Feed phase: Co(II) at pH 5.0. Pseudo-emulsion phase: 0.32 M DP-8R in Exxsol D100 + 0.1 M H₂SO₄. Feed flow rate: $400 \text{ cm}^3 \text{ min}^{-1}$. Pseudo-emulsion flow rate: $200 \text{ cm}^3 \text{ min}^{-1}$.

Fig.7. Influence of pH on Co(II) transport. Feed phase: 1.7×10^{-4} M Co(II) at various pH. Pseudo-emulsion phase as in Fig.6.

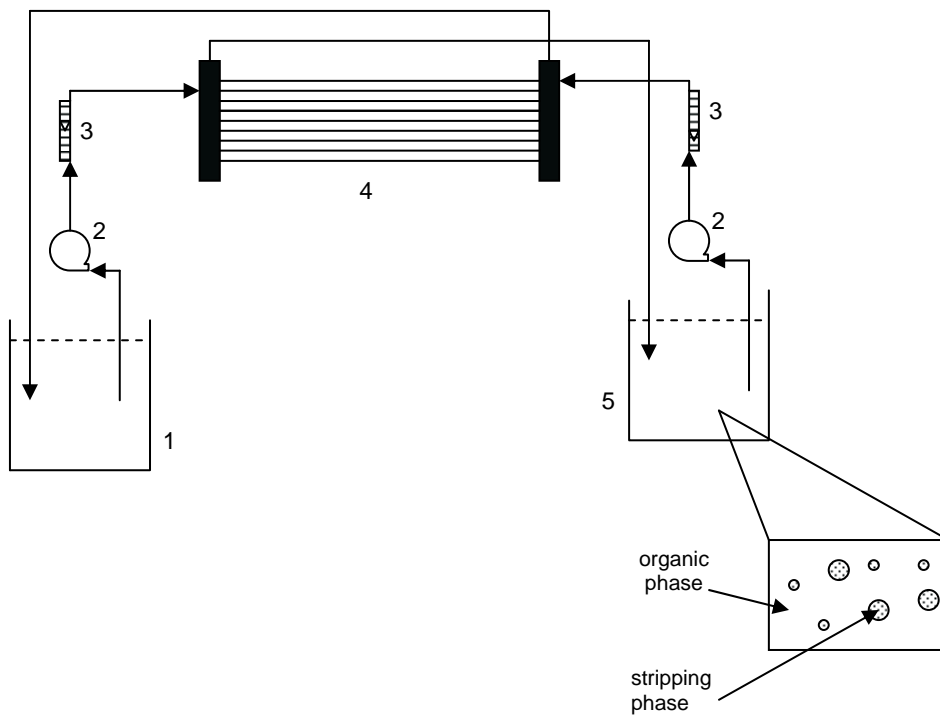


Fig. 1

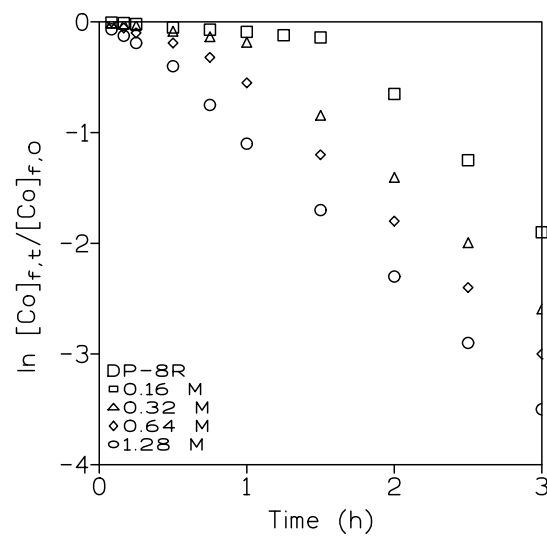


Fig. 2

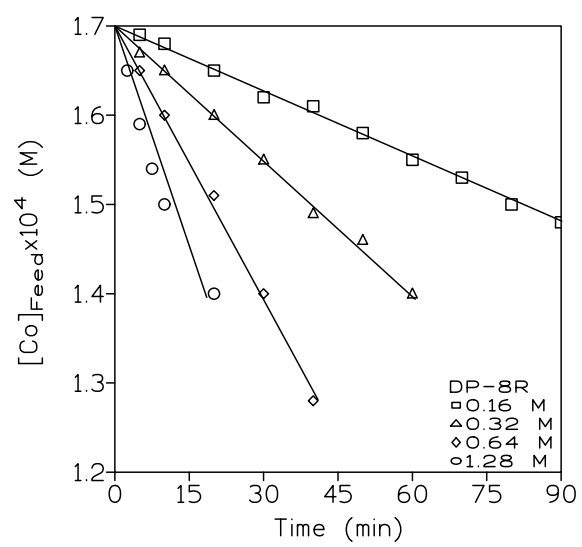


Fig.3

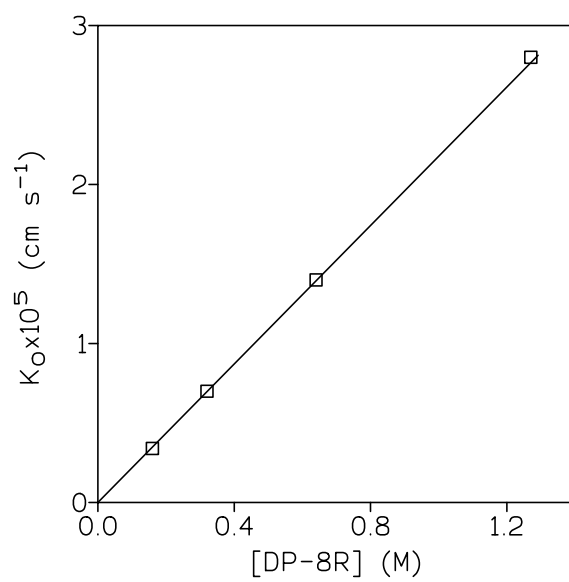


Fig. 4

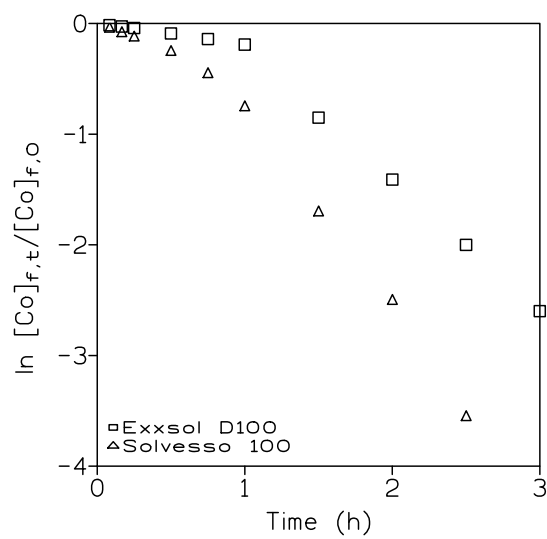


Fig. 5

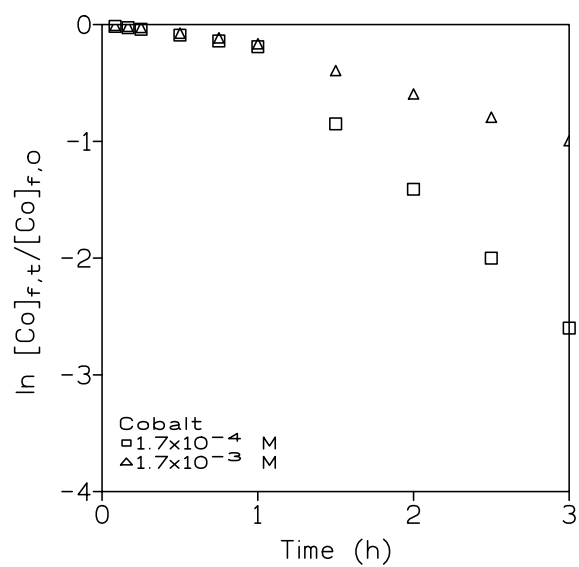


Fig.6

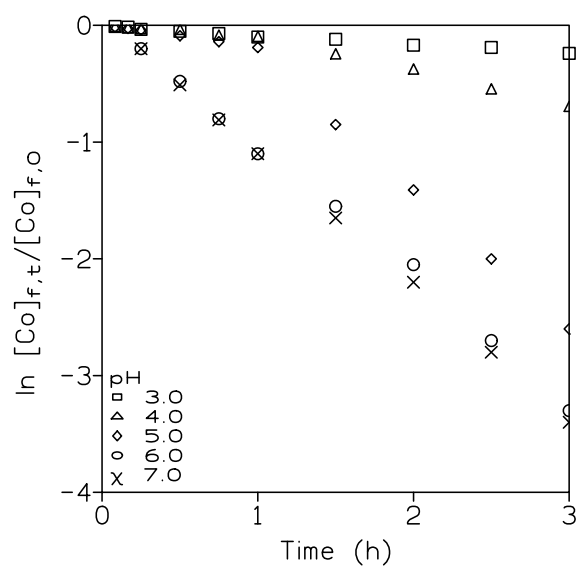


Fig. 7