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Recovery and Concentration of Metal Ions. I. Donnan Dialysis

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ABSTRACT

Transport of sulfates of divalent metals [M = Mn(II), Cu(II), Co(II), Ni(II)] in a system made up of multicomponent solution of MSO_4|Nafion membrane|sulfuric acid solution was studied. It was found that Donnan dialysis results in a recovery factor amounting to 80–90% with the concentration ratio $C_{M,strip}/C_{M,feed}$ not less than 30. The results of M(II)/H countertransport were compared with the results of simple dialysis of single and multiionic solutions. The technique of Donnan dialysis is efficient in preconcentrating ions for further separation in a multimembrane hybrid system employing a specific carrier of ions.

INTRODUCTION

Interest in the development of efficient techniques for separations of ions is a stimulant for constructing new membrane systems. Effective separation by membrane techniques is, however, a difficult problem when ions are of the same valency and are close in nature. A number of papers have dealt with the separation, recovery, or removal of Co(II), Ni(II), Cu(II), Mn(II), and Zn(II) sulfates from some industrial effluents, wastewaters, and dilute leach liquors in hydrometallurgical processing of lean ores. Some of these cations were separated by using liquid membranes containing properly selected specific carriers of divalent metals (1–5). Also, charged polymer membranes (6–9) separating ions by the charge

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and structure of complexes formed in a feed solution were reported. When applied in Donnan dialysis (DD), these membranes allow the concentration of metal ions from dilute solutions of salts (7, 10–21) in a transport system composed of

feed phase	cation-exchange membrane	strip phase
(M^{Z+}, A^-)	(IEM)	(H^+, A^-)

The effectiveness of polymer membranes in DD is, however, sometimes limited by low transmembrane fluxes and diminished selectivity. On the other hand, liquid membranes (LM) offer higher diffusivities and selectivities, being rather unstable and difficult to use in large-scale applications. Our general idea is to combine the properties of both membranes by adding a liquid membrane to Donnan dialysis in the sequence:

feed solution | IEM | LM | IEM | strip solution

The resulting multimembrane hybrid system is expected to be versatile in handling and to exhibit a selectivity not lower than that of liquid membranes containing the same selective carrier of metal ions. A detailed description of that system and the experimental results will be given in the next paper in this series.

In order to obtain the information required to design an efficient multimembrane system, we first examined sorption equilibria, simple permeation, and Donnan dialysis of some divalent metal ions using the perfluorosulfonic membrane Nafion-120. The results presented deal with fluxes as the fundamental property of ion-exchange membranes and with factors that characterize DD as the technique for recovering and preconcentrating metal ions.

EXPERIMENTAL

Materials

Commercially available perfluorosulphonic cation-exchange membrane (Nafion-120, Du Pont de Nemours, USA) was used in our experiments. The membrane was selected because of its high stability in corrosive organic media. Prior to the experiments the membrane was treated with 1 MH_2SO_4 (M = mol/dm^3) several times, washed with distilled water, and conditioned by boiling in water to obtain the stable expanded form. The pretreated membrane (thickness 0.28 mm, ion-exchange capacity 1.1 mol/dm^3) was converted into a metal ion form by soaking in a 0.5 M solution of metal sulfate (analytical grade, P.O.Ch., Gliwice, Poland).

Sorption of Metal Sulfates (Donnan Equilibria)

The membranes were equilibrated with salt solutions of 0.01–0.5 molarity for 24 hours at 298 K. After drying with filter paper, the membrane samples were weighed and placed in a thermostated cell (298 K) equipped with an immersion conductivity probe. The concentration of eluted salt was determined conductometrically, and the concentration of metal sulfate sorbed in the internal aqueous phase of a membrane, C_{int}, was calculated:

$$C_{\text{int}} = 1000n/V_{\text{w}}\ (\text{mol/dm}^3) \tag{1}$$

where n is the number of moles of desorbed salt and V_{w} (cm^3) is the volume of water in the membrane, calculated from the weight of a sample in its swollen and dry state.

Transport Experiments

Simple Dialysis

The permeation of single salts and competitive permeations of Ni(II), Co(II), Cu(II), and Mn(II) sulfates to water across Nafion-120 membrane were examined. The transports were performed in a two-chamber cell with a membrane of working area A equal to 0.8 cm^2. The feed and strip solution of 250 and 75 cm^3 volume, respectively, were thermostated (298 K) and intensively agitated. The initial ($t = 0$) concentration of the feed solution was 0.05 or 0.5 M. In the case of multiionic transports, the feed phase contained salts of 0.0125 molarity each. The amount of metal ions transported into the strip chamber was determined conductometrically, whereas in multiionic transports it was determined by using an atomic absorption spectrophotometer (Varian SPECTRAA-20ABQ).

The permeability coefficients P (cm^2/s) were calculated by fitting Eq. (2) to the experimental data:

$$Q(t) = -\frac{PALC_{\text{f}}}{6D} + \frac{PAC_{\text{f}}}{L}t \tag{2}$$

where $Q(t)$ is the number of moles of a salt transferred through a membrane after time t, A and L are the membrane area and thickness (0.027–0.030 cm, depending on the counterion), respectively, C_{f} is the initial concentration of a salt in a feed solution (mol/cm^3), and D is the diffusion coefficient.

Donnan Dialysis

In Donnan dialysis, sulfuric acid was used as the stripping agent. The cell was composed of two compartments of 0.2 cm^3 volume and the exposed membrane area was 3.14 cm^2. The solutions were pumped through the cell at a flow rate of 70 cm^3/min. The volumes of the feed and strip solutions were 250 and 50 cm^3, respectively. The membrane was used in its starting hydrogen form for the experiment. During the transport, feed and strip solutions were sampled (1 cm^3) to determine the concentration of metals by atomic absorption spectrophotometry.

In specific experiments, the composition and molarity of a feed (*f*) and strip (*s*) were

(A) $C_{M,f} = 0.5 \times 10^{-3}$ M M(II) | membrane | $C_{A,s} = 0.5$ M H_2SO_4
(B) $C_{M,f} = 0.5 \times 10^{-3}$ M M(II) | membrane | $C_{A,s} = 0.1$ M H_2SO_4
(C) $C_{M,f} = 2.5 \times 10^{-3}$ M M(II) | membrane | $C_{A,s} = 0.1$ M H_2SO_4
(D) $C_{M,f} = 2.5 \times 10^{-3}$ M M(II) | membrane | $C_{A,s} = 0.1$ M H_2SO_4
+ 0.01 M H_2SO_4

where M(II) = Co(II), Ni(II), Cu(II), and Mn(II) sulfate.

RESULTS AND DISCUSSION

Sorption of Metal Sulfates

Donnan equilibria describe nonexchange sorption of electrolytes into ion-exchange membranes. However, most of the membranes exhibit strong deviations from ideal behavior and absorb electrolytes in excess. The plots C_{int} vs C_{ext} drawn in Fig. 1 show relatively high sorption of the solutes into the Nafion membrane. The experimental distribution coefficient K_d, defined as the ratio C_{int}/C_{ext}, reaches 0.7–0.8 (Table 1) for each of the salts. Some explanation for the high sorption can be found in ion pairing of divalent cations and sulfate anions in solution. Note that the pK values for Co(II), Ni(II), Cu(II), Zn(II), and Mn(II) sulfates (22) are 2.47, 2.40, 2.36, 2.31, and 2.28, respectively. Consequently, for 0.05–0.5 M solutions the degrees of association ranges from 0.724 to 0.921, and the salts invade the internal solution of the membrane as undissociated species, mainly without ion exclusion effects.

Permeation of Metal Sulfates

The results, plotted in Fig. 2 as Q vs t curves, indicate the transports to be quasi-stationary. Thus it was possible to calculate permeability coef-

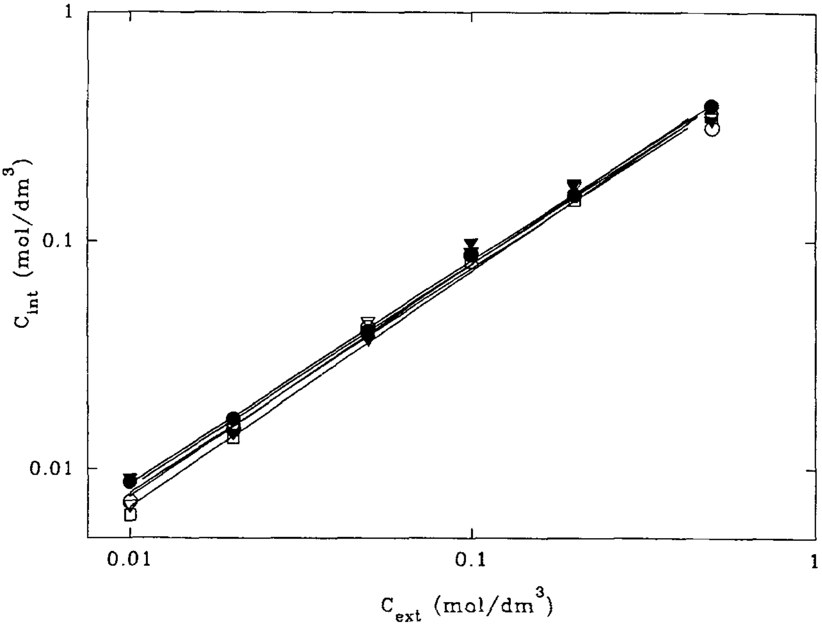

FIG. 1 Sorption isotherms for divalent metal sulfates: (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II) equilibrated with Nafion membrane.

ficients directly from the slope of curves, Eq. (2). The corresponding permeability and diffusion coefficients are listed in Table 1. The diffusion coefficients were calculated from

$$D_{MSO_4} = P_{MSO_4}/K_d(1 - V_p) \qquad (cm^2 \cdot s^{-1}) \tag{3}$$

where V_p denotes the volume fraction of a polymer in a swollen membrane. Numerically, the membrane permeability and diffusion coefficients are one order lower than the diffusion coefficients in aqueous solutions (23, 24). Few data are available in the literature concerning the permeability of Nafion membranes to the salts examined. For cobalt sulfate our results are comparable with those found by Heitner-Wirguin (25) for Nafion 125 and 110 membranes, i.e., P_{CoSO_4} = 4 and 3.6 × 10^{-7} cm^2·s^{-1}, respectively.

We also determined the permeability coefficients for sulfuric acid permeating the membrane in hydrogen form. Due to the Donnan exclusion effect, the sorption of H_2SO_4 is depressed and the permeability coefficients are smaller (see Table 1) than those observed for metal sulfates.

TABLE 1
Characteristics for Sorption and Permeation of Metal Sulfates through Nafion-120 Perfluorosulfonic Membrane in Simple Dialysis

Salt	Distribution coefficient K_d	Flux (mol/cm²s)	Permeability coefficient (cm²/s)	Diffusion coefficient (cm²/s)
Single salt systems, C_1^0 = 0.05 M				
$MnSO_4$	0.82	4.7×10^{-10}	2.7×10^{-7}	7.3×10^{-7}
$CuSO_4$	0.72	5.4×10^{-10}	2.9×10^{-7}	9.1×10^{-7}
$CoSO_4$	0.78	5.0×10^{-10}	2.6×10^{-7}	7.4×10^{-7}
$ZnSO_4$	0.77	4.3×10^{-10}	2.6×10^{-7}	8.2×10^{-7}
$NiSO_4$	0.80	3.8×10^{-10}	2.2×10^{-7}	6.1×10^{-7}
H_2SO_4	0.07	3.3×10^{-11}	2.0×10^{-8}	7.2×10^{-7}
Single salt systems, C_1^0 = 0.5 M				
$MnSO_4$	0.77	5.7×10^{-9}	3.0×10^{-7}	8.6×10^{-7}
$CuSO_4$	0.78	4.1×10^{-9}	2.2×10^{-7}	6.6×10^{-7}
$CoSO_4$	0.81	3.8×10^{-9}	2.0×10^{-7}	5.5×10^{-7}
$ZniSO_4$	0.74	3.5×10^{-9}	2.1×10^{-7}	7.2×10^{-7}
$NiSO_4$	0.76	3.3×10^{-9}	1.9×10^{-7}	5.6×10^{-7}
H_2SO_4	0.24	2.2×10^{-9}	1.3×10^{-7}	1.3×10^{-6}
Multiionic system, C_1^0 = 0.0125 M (for each salt)				
$MnSO_4$		7.0×10^{-11}	1.6×10^{-7}	
$CuSO_4$		7.7×10^{-11}	1.3×10^{-7}	
$CoSO_4$		5.9×10^{-11}	1.7×10^{-7}	
$NiSO_4$		7.1×10^{-11}	1.6×10^{-7}	
total MSO_4		2.9×10^{-11}	1.6×10^{-7}	

Nevertheless, diffusion coefficients calculated (Eq. 3) for sulfuric acid are comparable (0.05 M) or higher (0.5 M) than those for salts.

The results of simple dialysis presented in Table 1 show the permeability coefficients for metal sulfates to be very close to one another, indicating the separation of these salts to be impossible. The results of transport in the multiionic system seen in Fig. 3 prove this conclusion, i.e., the dialysis does not result in the separation of divalent cations. The permeability coefficients (Table 1) found in this experiment vary in the limits 1.3 to 1.7 $\times$ 10^{-7} $cm^2 \cdot s^{-1}$ for manganese and copper sulfate, respectively, and are even less differentiated than those found in single salt transports.

Donnan Dialysis

When an ion-exchange membrane is placed between two electrolyte solutions differing in composition, interdiffusion or/and internal ion-ex-

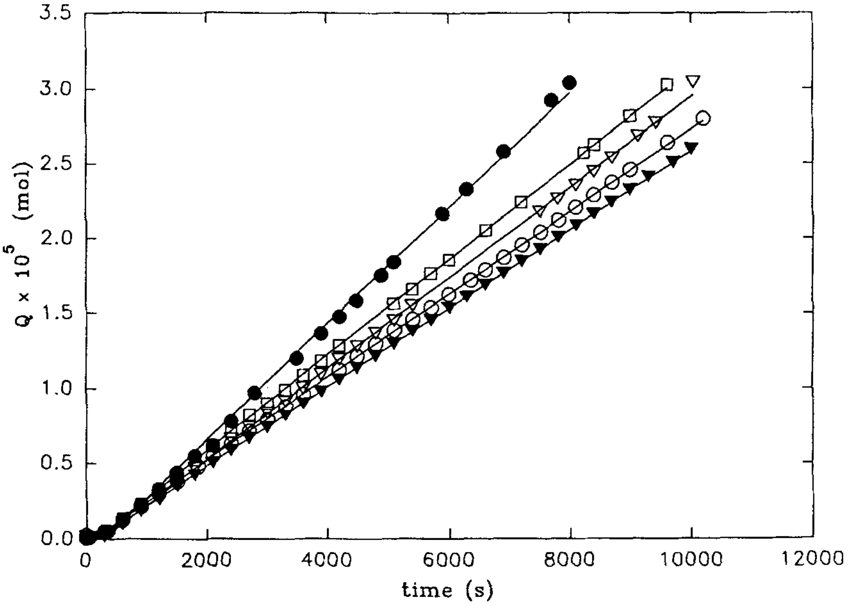

FIG. 2 Permeation of metal sulfates ($C^0_{M,f}$ = 0.5 M) through Nafion membrane–single-salt systems: (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II).

change of ions occurs in the membrane. The transport of metal ions is balanced by antitransported ions moving in the opposite direction. One can expect that the basic mechanism for DD is the exchange reaction between polymer fixed sites neutralized with metal or hydrogen ions. However, the sorption of metal sulfates and acid suggests that some amount of species can diffuse to the opposite side without the stoichiometry predicted by the ion-exchange reaction or by strictly balanced interdiffusion of ions.

In substance, the driving force for Donnan dialysis is the high concentration of a stripping agent that leads to effective pumping of ions from a feed solution and to a high recovery factor (RF):

$$RF = 1 - C_{M,f}/C^0_{M,f} \tag{4}$$

The efficiency of transport (EF) exceeds 1:

$$EF = C_{M,s}/C_{M,f} \gg 1 \tag{5}$$

because of the active pumping of ions. In reference to external solutions,

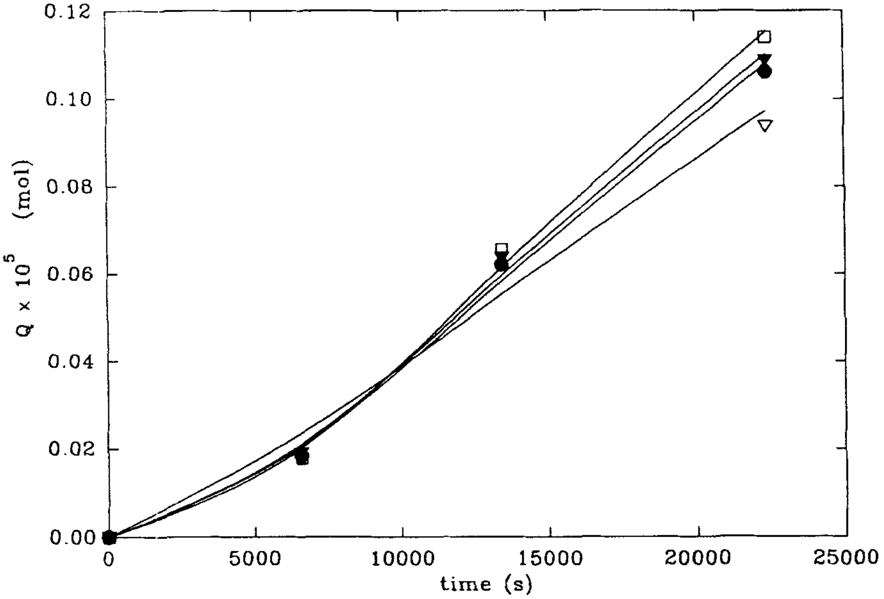

FIG. 3 Permeation of metal sulfates from multicomponent solution ($C^0_{M,f}$ = 0.0125 M, each of salts) through Nafion membrane: (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II).

this effect is known as "up-hill" transport, although within a membrane diffusion always occurs as a down-hill process.

Recovery and Concentration of Divalent Metals

In order to evaluate the recovery (RF) and efficiency (EF) factors for divalent metals, System A, which was treated as the reference system, was examined. The high concentration of acid in the strip solution (0.5 M) and the low concentration of the feed solution (0.0005 M) are typical for practical applications of Donnan dialysis. The results are drawn in Figs. 4(a–c) as the concentration $C_{M,f}$ and $C_{M,s}$ vs time curves and the resulting RF(t) and EF(t) plots. The recovery factors reach ~95% with EF in the 60 to 130 range whereas the separation of metal ions is negligible for each time of transport. These results are consistent with those published elsewhere by Trojanowicz et al. (21) who used the commercial cation-exchange membrane R-1010 (Rai Research Corp., USA) for Donnan dialysis of Cu(II), Mn(II), Ni(II), and Zn(II) salts. They found efficient preconcentration of metals without marked preference toward one metal.

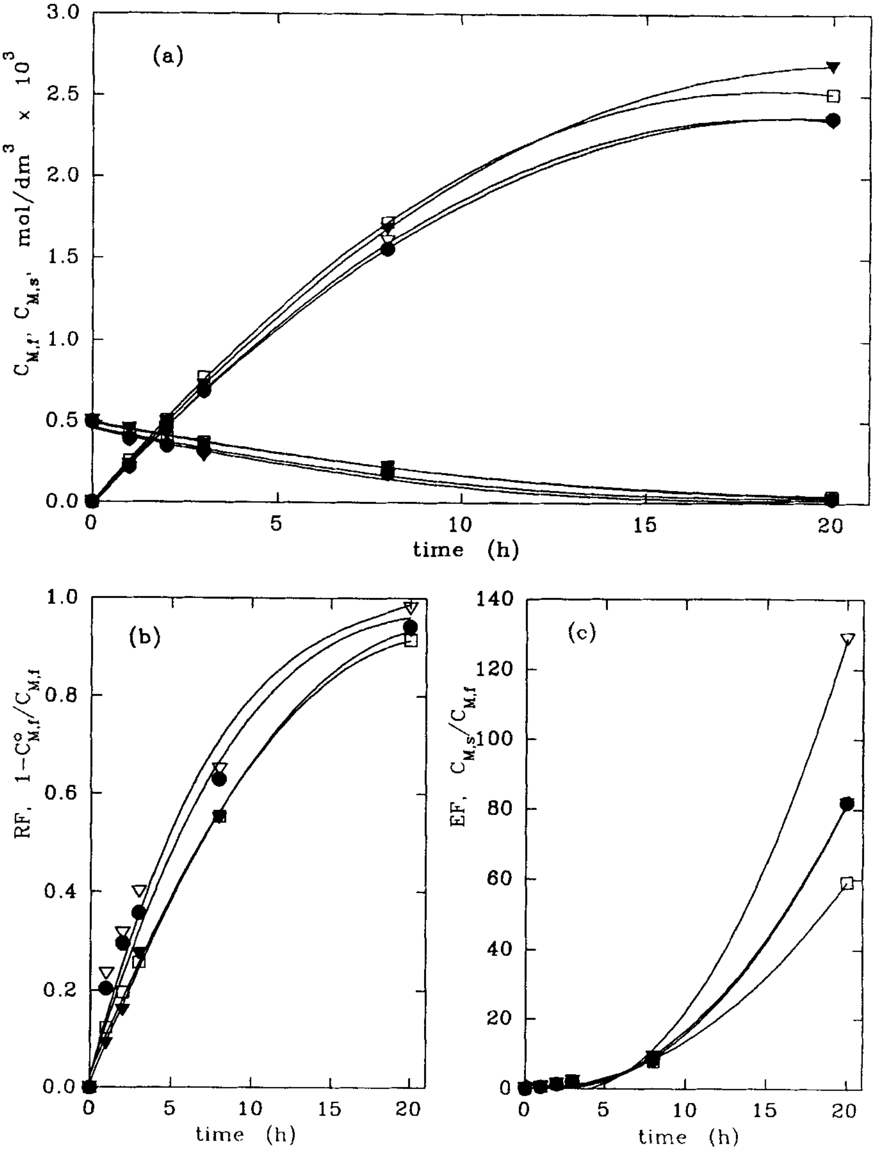

FIG. 4 Donnan dialysis of divalent metals; feed solution, 0.5×10^{-3} M MSO_4; strip solution, 0.5 M H_2SO_4: (a) concentrations of $C_{M,f}$ and $C_{M,s}$, (b) recovery factor, (c) efficiency factor of (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II).

Donnan Dialysis with Diluted Stripping Agent

In the designed multimembrane system (see Part 2 of this series), Donnan dialysis is coupled to a liquid membrane transport. Thus, the overall transport will be facilitated by complexing the permeating ions whereas the interfacial concentration of hydrogen ions (driving force for DD) will be diminished. It was therefore justified to carry out experiments with lower concentration of sulfuric acid in a stripping solution (0.1 M). The experimental results are seen in Figs. 5(a–c).

To compare the results of Donnan dialysis to simple dialysis, the quasi-steady-state fluxes were calculated from $C_{M,s}$ vs time plots. These fluxes correspond to the linear part of the curves observed and are characterized by correlation coefficients r not less than 0.999. The total flux for DD of metal ions in Systems A and C (see Table 2) is 4.4×10^{-9} to 1.4×10^{-8} mol/cm^{2}s, i.e., the DD fluxes are roughly 10–100 times higher than those found in competitive dialysis (2.9×10^{-10}) mol/cm^2·s). Decreasing the concentration of sulfuric acid from 0.5 (A) to 0.1 M (B) does not depress the fluxes proportionally. The ~15% lowering of fluxes in System B probably results from the flux saturation effect in DD known to be characteristic of reaction-diffusion transport (26).

Effect of Concentration of Feed Solution

To recognize the effect of concentration of salts in a feed solution, the molarity of the feed solution was increased to 2.5×10^{-3} M. The results in Fig. 6(a–c), compared with those in Fig. 4(a–b) and the fluxes in Table 2, prove that a 5-fold increase of sulfate concentration in the feed phase results in a nearly proportional increase of fluxes. Thus, the concentration of salts in a feed solution (usually very dilute) could be considered as the controlling factor of Donnan dialysis.

Effect of Acidification of a Feed Solution

During Donnan dialysis the counterions from the strip solution are transported into the feed phase. This occurs due to an equivalent exchange of cations between the feed and the strip solution as well as to a shunt flux of sulfuric acid as a consequence of Donnan sorption of a free electrolyte into an ion-exchange membrane. Comparing the results in Fig. 7 against those in Fig. 6 and the fluxes in Table 2 allows us to conclude that the transport from the acidified feed phase (0.0025 M metal sulfates dissolved in 0.01 M sulfuric acid) is diminished not more than ~10%. Thus, the acidification of the feed solution by the countertransported acid does not seem to affect the metal fluxes much, at least not in the systems examined.

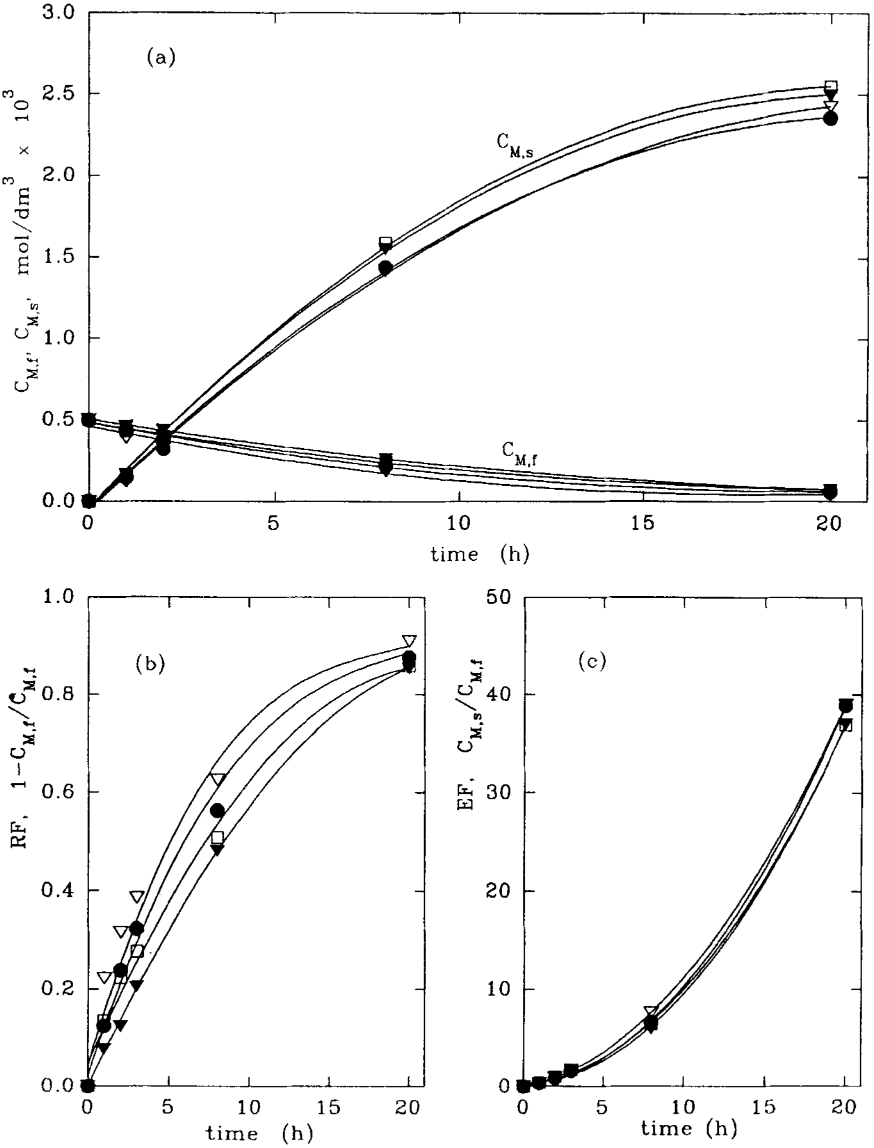

FIG. 5 Donnan dialysis of divalent metals; feed solution, 0.5 × 10^{-3} M MSO_4; strip solution, 0.1 M H_2SO_4: (a) concentrations of $C_{M,f}$ and $C_{M,s}$, (b) recovery factor, (c) efficiency factor of (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II).

TABLE 2
Steady-State Fluxes in Donnan Dialysis of Metal Sulfates through Nafion-120 Perfluorosulfonic Membrane

Salt (multiionic systems)		$C^0_{M,f}$ (M)	$C^0_{M,s}$ (M)	Flux (mol/cm^2·s)
(A)	$MnSO_4$	0.5×10^{-3}	0.5	1.2×10^{-9}
	$CuSO_4$	0.5×10^{-3}	0.5	1.1×10^{-9}
	$CoSO_4$	0.5×10^{-3}	0.5	1.0×10^{-9}
	$NiSO_4$	0.5×10^{-3}	0.5	1.1×10^{-9}
	Total MSO_4	2.0×10^{-3}	0.5	4.4×10^{-9}
(B)	$MnSO_4$	0.5×10^{-3}	0.1	7.9×10^{-10}
	$CuSO_4$	0.5×10^{-3}	0.1	8.7×10^{-10}
	$CoSO_4$	0.5×10^{-3}	0.1	8.6×10^{-10}
	$NiSO_4$	0.5×10^{-3}	0.1	8.6×10^{-10}
	Total MSO_4	2.0×10^{-3}	0.1	3.8×10^{-9}
(C)	$MnSO_4$	2.5×10^{-3}	0.1	3.4×10^{-9}
	$CuSO_4$	2.5×10^{-3}	0.1	3.5×10^{-9}
	$CoSO_4$	2.5×10^{-3}	0.1	3.6×10^{-9}
	$NiSO_4$	2.5×10^{-3}	0.1	3.4×10^{-9}
	Total MSO_4	1.0×10^{-2}	0.1	1.4×10^{-8}
(D)	$MnSO_4$	2.5×10^{-3}	0.1	3.2×10^{-9}
	$CuSO_4$	2.5×10^{-3}	0.1	3.1×10^{-9}
	$CoSO_4$	2.5×10^{-3}	0.1	3.1×10^{-9}
	$NiSO_4$	2.5×10^{-3}	0.1	3.0×10^{-9}
	Total MSO_4	1.0×10^{-2}	0.1	1.2×10^{-8}

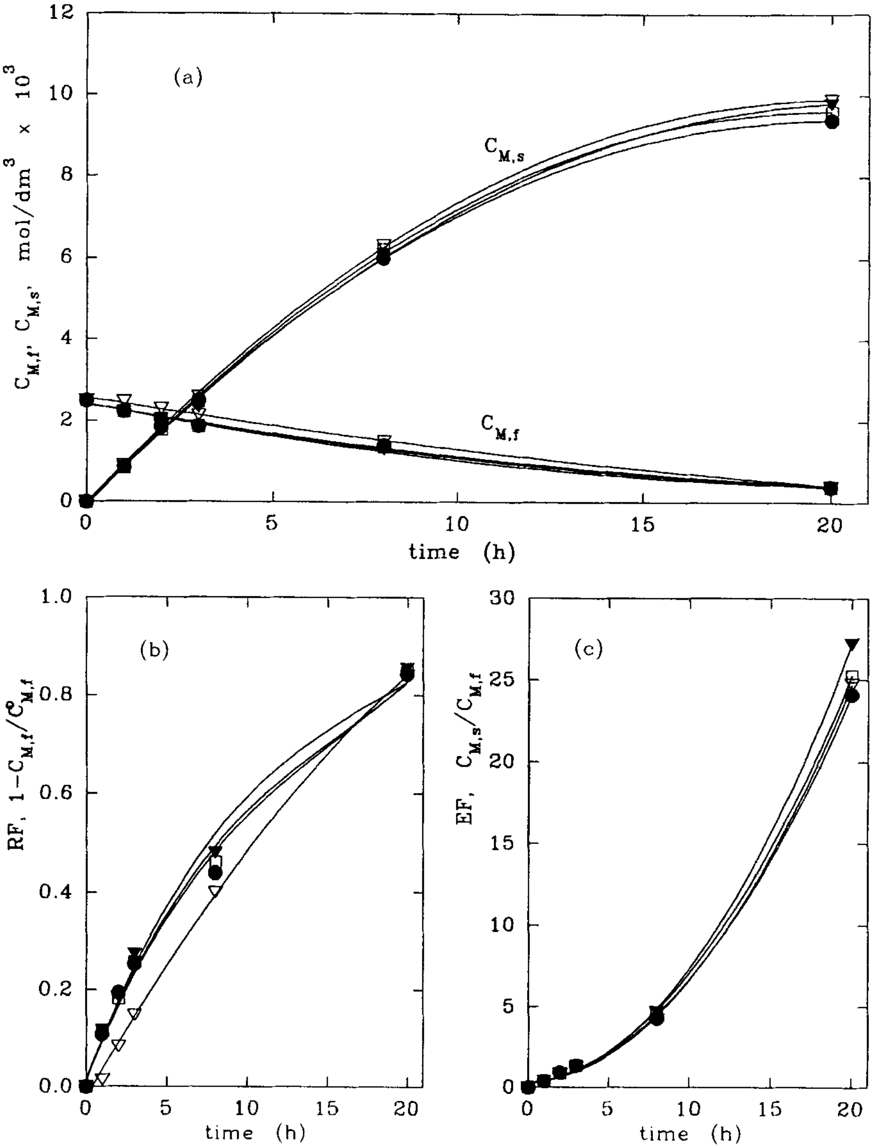

FIG. 6 Donnan dialysis of divalent metals; feed solution, 2.5×10^{-3} M MSO_4; strip solution, 0.1 M H_2SO_4: (a) concentrations of $C_{M,f}$ and $C_{M,s}$, (b) recovery factor, (c) efficiency factor of (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II).

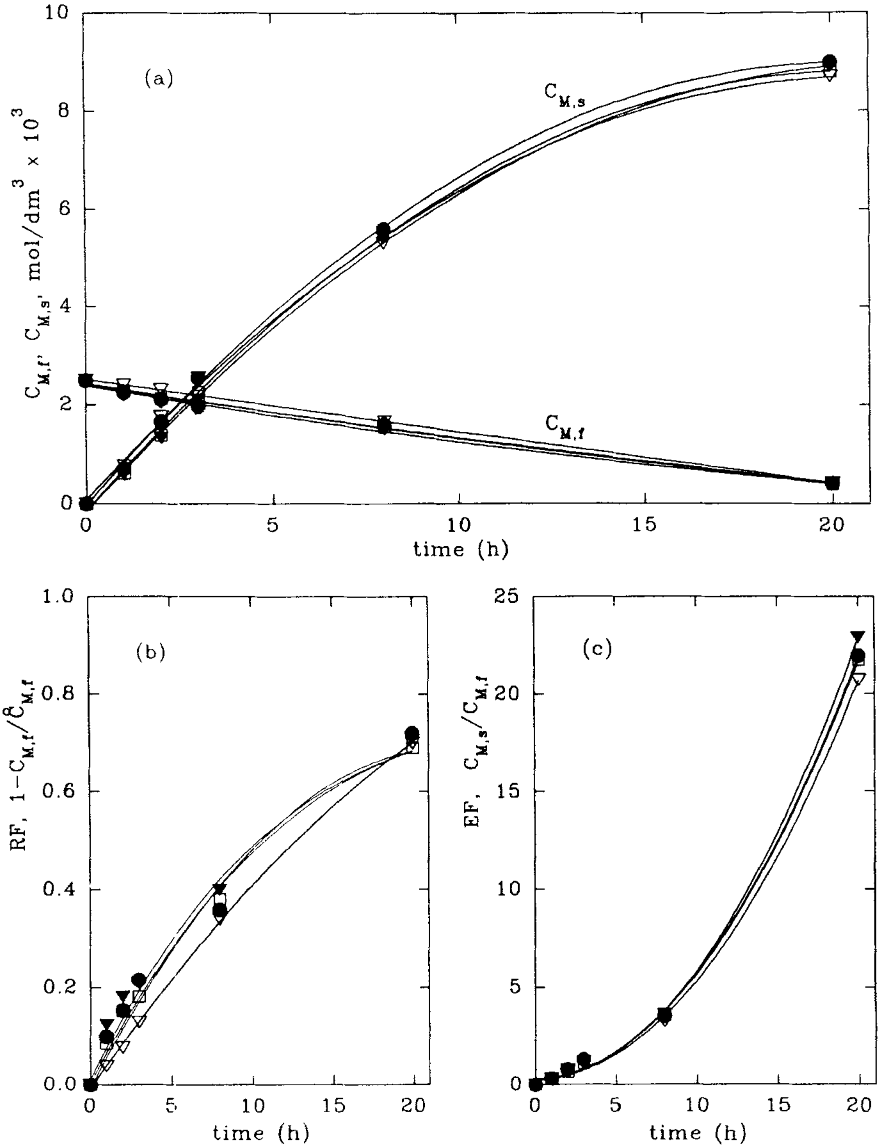

FIG. 7 Donnan dialysis of metal sulfates from acidified feed solution; feed solution, 2.5 × 10^{-3} M MSO_4 + 0.01 M H_2SO_4; strip solution, 0.1 M H_2SO_4: (a) concentrations of $C_{M,f}$ and $C_{M,s}$, (b) recovery factor, (c) efficiency factor of (○) Zn(II), (▽) Co(II), (□) Cu(II), (●) Mn(II), and (▼) Ni(II).

CONCLUSIONS

The results of our experiments demonstrated the fluxes of divalent metal ions in simple dialysis and Donnan dialysis to be remarkably different. Higher fluxes in DD point to the fast interchange of ions as the main mechanism of transport in the system. Indeed, the rates of the simple dialysis are limited because the membrane permeability is a product of diffusion and distribution coefficients.

The results prove the usefulness of Nafion membranes employed in DD for the effective recovery and concentration of Cu(II), Mn(II), Co(II), Ni(II) sulfates. However, both dialysis techniques examined are inefficient for the separation of these ions from multicomponent solutions.

The sorption of electrolytes and fluxes in DD allow to consider the perfluorosulfonic ion-exchange membrane as a component of multimembrane hybrid system for separation of divalent metals.

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