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J. M. Ortiz De Záarate[a]; A. Velázquez[a]; L. Peña[a]; J. I. Mengual[a]
[a] DEPARTAMENTO DE FÍSICA APLICADA I (TERMOLOGÍA), FACULTAD DE FÍSICA UNIVERSIDAD COMPLUTENSE, MADRID, SPAIN

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Influence of Temperature Polarization on Separation by Membrane Distillation

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J. M. ORTIZ DE ZÁRATE, A. VELÁZQUEZ, L. PEÑA, and J. I. MENGUAL*
DEPARTAMENTO DE FÍSICA APLICADA I (TERMOLOGÍA)
FACULTAD DE FÍSICA
UNIVERSIDAD COMPLUTENSE
28040 MADRID, SPAIN

ABSTRACT

Nonisothermal mass transport through three PTFE membranes has been studied. Aqueous solutions (0.1, 0.2, and 0.3 M) of sodium chloride were employed. The experiments were carried out under a temperature difference of 30 K and at mean temperatures of 25 and 45°C. The initial concentration and the stirring rate were changed independently. The stirring rate was varied among 0, 75, 150, 250 and 350 rpm. The results were interpreted on the basis of the membrane distillation theory. A separation between the two components increasing with time was obtained, and a model has been developed which permits us 1) to obtain concentration values from volume values, 2) to define a separation coefficient, and 3) to quantify the influence of stirring rate on the separation. The model has been applied to the experimental results and the agreement may be considered good.

INTRODUCTION

The transport of liquids through membranes under nonisothermal conditions has been known since the beginning of the century. Early experiments performed with dense membranes involved fluxes of very small magnitude. This fact led to the conclusion that the process was of a diffusive nature and therefore it was called "thermal osmosis" or "thermoosmosis" (1–4). Consequently, the possibilities for industrial application were scarce.

In the late 1960s, larger nonisothermal water transport was found through porous and hydrophobic partitions. Here it is worth noting the

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*To whom correspondence should be addressed.

contributions of Findley and coworkers (5, 6) who used various membranes such as asbestos paper plus silicone, glass fiber plus Teflon, etc. The subject was not reconsidered until the mid-1980s (7–17). In these papers new membrane materials such as PTFE (polytetrafluoroethylene), PVDF (polyvinylidene fluoride), and PP (polypropylene) were employed. The water fluxes reported in Refs. 5 and 17 were up to three or four times greater than those of Refs. 1–4. The magnitudes of the fluxes suggested that it was a new phenomenon of a nondiffusive nature. Nowadays the phenomenon is explained by saying that the transport mechanism is "membrane distillation."

The membrane material is water repellant, so liquid water cannot enter the pores of the membrane unless a pressure exceeding the LEPW (liquid entry pressure of water) of the membrane is applied. A liquid–vapor interface is then formed on either side of the water-repelling membrane. Due to a temperature difference, evaporation takes place at the hot interface and, after passage of the vapor through the pores, condensation takes place at the cold interface. Obviously, for membrane distillation to proceed, it is essential that the entrance of liquid water to the hydrophobic pores be prevented. Such a membrane allows the passage of water and volatile solutes in the vapor phase but excludes the flow of liquid water and its nonvolatile solutes. This leads to the possibility of obtaining separation of the components of aqueous solutions in some cases. There are studies in the literature of thermal separations of aqueous solutions of several nonvolatile solutes through different porous partitions. For example, Mita et al. (18) studied the separation of aqueous solutions of sodium chloride and potassium chloride through AP-20 Millipore filters, Sarti and Gostoli (19) employed sodium and ammonium chloride solutions and PTFE membranes, Schofield et al. (20) used aqueous solutions of sodium chloride and PVDF membranes, etc. In previous papers that refer to hydrophobic membranes and pure water (21, 22), it has been experimentally shown that the water flux is affected by the presence of the unstirred liquid layers adjacent to the membrane on both sides. The phenomenon is known as "temperature polarization," and it is expected to influence separation when aqueous solutions are employed.

In research leading to the present paper, experiments using aqueous solutions of sodium chloride were carried out with three PTFE membranes. Separation of the components was obtained by means of membrane distillation through the membrane. In order to quantify this effect, a separation coefficient has been defined. The influence of some relevant parameters has been analyzed, and special attention has been paid to the stirring rate.

EXPERIMENTAL

Materials

We have studied aqueous solutions of sodium chloride with solute concentrations of 0.1, 0.2, and 0.3 M. The materials employed in the experiments were pure water (deionized and doubly distilled) and pure proanalysis-grade sodium chloride. The membranes used were commercial PTFE ones. All of them are grossly porous partitions, with irregular cavities going through the membrane thickness. They have a hydrophobic nature and are industrially used in microfiltration processes. Their main characteristics, as specified by the manufacturer, are:

- Gelman TF-1000: nominal pore radius = 1 μm; thickness = 178 μm; porosity = 80%; liquid entry pressure of water = 0.47 atm.
- Gelman TF-450: nominal pore radius = 0.45 μm; thickness = 178 μm; porosity = 80%; liquid entry pressure of water = 1.36 atm.
- Gelman TF-200: nominal pore radius = 0.2 μm; thickness = 178 μm; porosity = 80%; liquid entry pressure of water = 2.72 atm.

Apparatus

The apparatus used (Fig. 1) was substantially similar to the ones described in previous papers (21–23). The central part of the experimental device is a stainless steel cell which essentially consists of two equal cylindrical chambers each having a volume of 270 cm^3. The membrane was fixed between the chambers with the help of a PVC holder. Three Viton O-rings were employed to ensure the absence of leaks in the assembly. The membrane surface area exposed to the flow was 2.75×10^{-3} m^2. Each semicell was connected to a different external solution container by means of a peristaltic pump and the corresponding silicone conductions. The circulation assures that the concentration of the solution is the same inside each chamber and its corresponding container. Both external containers are cylindrical and graduated in such a way that the volume of liquid in them may be easily known. Knowledge of the volume in the silicone conductions permits us to know the total solution volume on each side of the membrane.

The temperature requirements were set by connecting each chamber, through the corresponding water jacket, to a different circulation thermostat. In order to ensure the uniformity of temperatures and concentrations inside each chamber, the solutions were stirred by a chain-driven cell magnetic stirrer assembly. Temperatures were measured with platinum

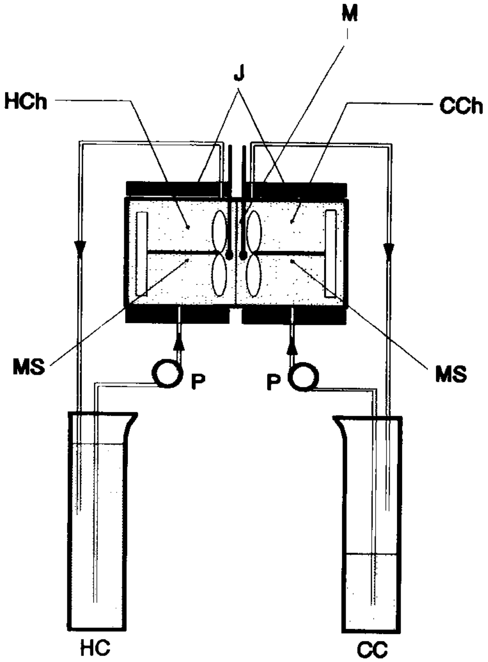

FIG. 1 Experimental assembly: M, membrane; HCh, hot chamber; CCh, cold chamber; J, thermostated jackets; MS, magnetic stirrer; P, peristaltic pump; HC, hot container; and CC, cold container.

resistance thermometers placed near both surfaces of the membrane. Under these conditions, the temperature was constant to within ±0.1°C.

As an experiment proceeds, the concentration of solute in each semicell varies. The solute concentrations of the samples periodically extracted from both containers are assessed by means of standard chemical titration for the chloride ion (Mohr titration). The error in these measurements is ±0.005 mol/L. It has been assumed that the volume extracted from both solution containers during the sampling procedure is negligible in proportion to the total volume, so that the method of measurement does not alter the results.

THEORY

Each experiment is started with the same solution (molar concentration of solute, C^0) on both sides of the membrane, and with a temperature difference (ΔT) maintained between the semicells. Since the vapor pressure of the hot solution is higher than that of the cold one, a flux of water takes place through the pores. Nevertheless, the solute cannot cross the membrane (it is nonvolatile), so its quantity remains constant on each side. Therefore, the solute concentration inside the chambers must change continuously during each experiment. Taking into account these facts, we have

$$C_{h/c}(t)\cdot V_{h/c}(t) = C^0\cdot V^0_{h/c} \tag{1}$$

where t is the time, $C_{h/c}(t)$ and $V_{h/c}(t)$ are the molar concentration of solute and the volume of solution at the hot/cold side of the membrane, and $V^0_{h/c}$ is the corresponding initial volume.

In the case of pure water, the difference in vapor pressures between the ends of the pores may be easily related to the difference in temperatures on both surfaces of the membrane, using standard thermodynamic procedures (16, 22). The presence of nonvolatile solutes may be first appreciated as a flux reduction (20). In the present paper the concentrations of the solutions are relatively small and, consequently, the water flux is expected to be practically independent of time during each experiment, in spite of the fact that the concentrations of both chambers change in time (obviously the validity of this assumption should be confirmed by the experimental results, as will be discussed later on). In this case, Eq. (1) leads to

$$C_{h/c}(t) = \frac{C^0\cdot V^0_{h/c}}{V^0_{h/c} \mp J\cdot t} \tag{2}$$

Equation (2) tells us that the flux of water originates in a growing concentration difference between the two chambers. The solute concentration grows on the hot side and decreases on the cold one, as was observed in the experiments. In order to study the time evolution of the concentration difference between the two chambers, a "separation coefficient," $s(t)$, is defined by

$$s(t) = \frac{C_h(t) - C_c(t)}{C^0} \tag{3}$$

It can be easily shown by using Eqs. (2) and (3) that $s(t)$ does not depend explicitly on the initial molar concentration, C^0.

On the other hand, for this paper it was experimentally tested that the rate of change of $C_{h/c}$ with time depends strongly on the stirring rate. This may be explained by taking into account two facts: 1) The water flux in membrane distillation is affected by the existence of unstirred liquid layers adjoining the membrane on both surfaces, and 2) the thickness of these unstirred liquid layers decreases with the stirring rate. In some previous papers which refer to pure water and to PTFE as well as to dense cellulose acetate membranes (21, 22, 24), we demonstrated that this effect, called temperature polarization, may be described by means of an empirical dependence of nonisothermal flux, $J(v)$, on the stirring rate, v, given by

$$\frac{v}{J(v) - J_0} = X + Y \cdot v \tag{4}$$

where J_0 is the nonisothermal flux without stirring and X and Y are adjustment parameters. The value corresponding to an infinite stirring rate (absence of temperature polarization effects), J_∞, is obtained from parameter Y. As a matter of fact, from Refs. 21, 22, and 24 it can be stated that the influence of temperature polarization is very important (the numerical values of the fluxes may be reduced up to 58% when comparing the results corresponding to $v = 250$ rpm with those obtained by extrapolation to an infinite stirring rate).

It is worth noting that Eq. (4) was obtained by taking into account only the effects of temperature polarization (the liquid employed was pure water). In the present paper we also consider the existence of concentration polarization which, in principle, should affect the flux measurements in a similar way. This point was considered and evaluated by Schofield et al. (20), who found slight flux reductions (about 3%) when comparing experiments carried out for pure water with experiments for 2.5 and 5 M aqueous solutions of sodium chloride. In what follows we are going to assume that, due to the fact that we are working with dilute solutions, the effect may be considered as practically negligible. The validity of this assumption will be discussed later. In any case, the effect of concentration polarization may be incorporated into Eq. (4) if it is assumed that the adjustment parameters X and Y include both temperature and concentration polarization effects.

One of the goals of this paper is to study the influence of the stirring rate on the separation coefficient. Equations (2)–(4) predict an increase in $s(t)$ with v. The exact form of this relation is a rather complicated quotient of second-grade polynomials on v. From this relation, simple algebra permits us to obtain the limit value corresponding to an infinite stirring rate. This value, $s_\infty(t)$, is

$$s_\infty(t) = \frac{J_\infty \cdot t \cdot (V_h + V_c)}{V_h \cdot V_c + J_\infty \cdot t \cdot (V_h - V_c) - J_\infty^2 \cdot t^2} \tag{5}$$

$s_\infty(t)$ represents the separation obtained in time t without liquid layers adjoining the membrane (that is, in the absence of both temperature and concentration polarization). Taking into account that $s_\infty(t)$ is the maximum separation that might be achieved at time t, this value may be compared with the experimental ones in order to quantify the influence of polarization effects in separation.

RESULTS AND DISCUSSION

The initial volumes were always $V_h^0 = 800$ cm^3 and $V_c^0 = 350$ cm^3. All the experiments were carried out with a temperature difference of 30 K. The mean temperature, initial molar concentration, and stirring rate were varied independently. The values of the mean temperature were 25 and 45°C. The initial concentrations were 0.1, 0.2, and 0.3 M. The values of the stirring rate were 0, 75, 150, 250, and 350 rpm.

The temperature difference causes the water flux to be from hot to cold. This flux causes a decrease in the volume and an increase in the solute concentration on the hot side and the opposite on the cold side. In each one of the experiments the concentration and the volume of the solution on each side of the membrane were determined independently as functions of time. The pairs of values (volume, time) were adjusted to straight lines by a least-squares procedure and then the volume fluxes were obtained.

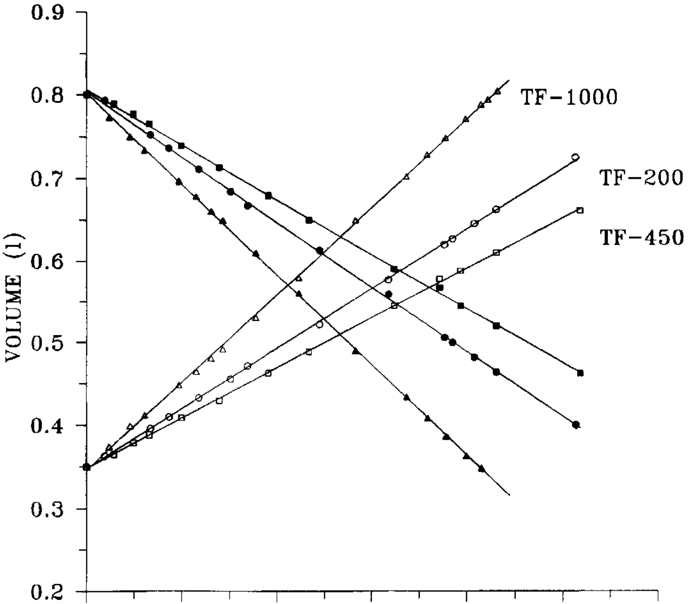

FIG. 2 Time evolution of volume on each side of the membrane. The filled (empty) symbols correspond to the hot (cold) side. $\overline{T} = 45$°C, $C^0 = 0.3$ M, $v = 350$ rpm.

As an example of the calculations carried out, in Fig. 2 we plot the results corresponding to each of the membranes in a particular case (mean temperature, $\bar{T} = 45°C$; initial molar concentration, $C^0 = 0.3$ M; stirring rate, $v = 350$ rpm). The correlation coefficient was 0.99 in the most unfavorable case for runs of at least 12 points, which confirms our previous assumption that the volume flux is practically independent of time (see Eq. 2). In Tables 1–3 the flux values are displayed for different experimental con-

TABLE 1
Solute Concentrations and Separation Coefficients Reached after 6 Hours with the Corresponding Volume Flux for the Different Experimental Conditions. Membrane TF-1000

$\bar{T}$ (°C)	C^0 (mol/L)	v (rpm)	$J \times 10^5$ (L/s)	C_c (mol/L)	C_h (mol/L)	s (%)
45	0.1	0	0.53	0.067	0.128	60
		75	1.04	0.062	0.138	75
		150	1.50	0.055	0.174	120
		250	1.90	0.049	0.197	150
		350	2.16	0.044	0.235	190
	0.2	0	0.49	0.146	0.224	40
		75	1.03	0.114	0.282	85
		150	1.43	0.090	0.427	170
		250	1.77	0.092	0.450	180
		350	2.03	0.090	0.455	180
	0.3	0	0.49	0.226	0.350	40
		75	1.02	0.171	0.450	95
		150	1.39	0.154	0.475	105
		250	1.76	0.142	0.551	135
		350	1.83	0.132	0.655	175
25	0.1	0	0.23	0.086	0.106	20
		75	0.47	0.075	0.117	40
		150	0.65	0.071	0.120	50
		250	0.80	0.067	0.125	60
		350	0.85	0.066	0.129	65
	0.2	0	0.23	0.170	0.214	20
		75	0.50	0.148	0.228	40
		150	0.70	0.138	0.240	50
		250	0.85	0.130	0.255	60
		350	0.96	0.121	0.261	70
	0.3	0	0.22	0.258	0.317	20
		75	0.50	0.219	0.327	35
		150	0.68	0.207	0.359	50
		250	0.84	0.192	0.372	60
		350	0.94	0.182	0.384	70

TABLE 2

Solute Concentrations and Separation Coefficients Reached after 6 Hours with the Corresponding Volume Flux for the Different Experimental Conditions. Membrane TF-450

$\bar{T}$ (°C)	C^0 (mol/L)	v (rpm)	$J \times 10^5$ (L/s)	C_c (mol/L)	C_h (mol/L)	s (%)
45	0.1	0	0.43	0.080	0.118	40
		75	0.84	0.067	0.127	60
		150	1.13	0.059	0.144	85
		250	1.41	0.055	0.165	110
		350	1.54	0.052	0.167	115
	0.2	0	0.44	0.165	0.235	35
		75	0.83	0.133	0.256	60
		150	1.10	0.122	0.291	85
		250	1.33	0.111	0.307	100
		350	1.49	0.095	0.312	110
	0.3	0	0.42	0.238	0.350	35
		75	0.73	0.216	0.378	55
		150	1.07	0.182	0.435	85
		250	1.26	0.174	0.471	100
		350	1.27	0.159	0.472	105
25	0.1	0	0.19	0.092	0.111	20
		75	0.38	0.082	0.114	30
		150	0.53	0.078	0.120	40
		250	0.63	0.073	0.122	50
		350	0.72	0.071	0.126	55
	0.2	0	0.20	0.174	0.213	20
		75	0.44	0.157	0.230	35
		150	0.57	0.142	0.237	50
		250	0.75	0.140	0.247	55
		350	0.82	0.134	0.256	60
	0.3	0	0.22	0.262	0.327	20
		75	0.44	0.241	0.340	30
		150	0.61	0.219	0.353	45
		250	0.72	0.206	0.370	55
		350	0.81	0.196	0.373	60

ditions ($\bar{T}$, v, and C^0) for the membranes TF-1000, TF-450, and TF-200, respectively. These tables show that the flux increases with both mean temperature and stirring rate, as expected.

On the other hand, these volume fluxes, J's, may be used to obtain the values of the solute concentration on both sides of the membrane, $C_{h/c}(t)$, via Eq. (2). These values may be compared with the ones obtained experimentally, and in this way a confirmation of the fitness of the membrane distillation model is reached. As an example of this procedure, in Fig. 3

TABLE 3
Solute Concentrations and Separation Coefficients Reached after 6 Hours with the Corresponding Volume Flux for the Different Experimental Conditions. Membrane TF-200

$\bar{T}$ (°C)	C^0 (mol/L)	v (rpm)	$J \times 10^5$ (L/s)	C_c (mol/L)	C_h (mol/L)	s (%)
45	0.1	0	0.47	0.079	0.114	35
		75	0.87	0.069	0.132	65
		150	1.18	0.052	0.146	95
		250	1.38	0.053	0.160	105
		350	1.57	0.048	0.170	120
	0.2	0	0.43	0.154	0.224	35
		75	0.83	0.128	0.271	70
		150	1.14	0.120	0.275	75
		250	1.41	0.111	0.322	105
		350	1.46	0.106	0.320	110
	0.3	0	0.51	0.235	0.347	35
		75	0.85	0.196	0.388	65
		150	1.15	0.173	0.403	75
		250	1.37	0.161	0.477	105
		350	1.47	0.154	0.493	110
25	0.1	0	0.21	0.090	0.111	20
		75	0.45	0.080	0.115	35
		150	0.60	0.074	0.122	50
		250	0.72	0.069	0.124	55
		350	0.80	0.070	0.129	60
	0.2	0	0.22	0.175	0.215	20
		75	0.46	0.159	0.217	30
		150	0.61	0.145	0.240	50
		250	0.73	0.139	0.250	55
		350	0.80	0.135	0.254	60
	0.3	0	0.24	0.255	0.312	20
		75	0.46	0.220	0.329	35
		150	0.62	0.227	0.369	50
		250	0.73	0.205	0.368	55
		350	0.79	0.197	0.379	60

the theoretical curve, obtained with Eq. (2), and the experimental points are displayed for the membrane TF-1000 (the mean temperature was 45°C, the initial concentration was 0.1 M, and the values of the stirring rate were 0, 75, and 350 rpm). The trend appearing in Fig. 3 is similar for the other membranes and under all the experimental conditions. A visual inspection of Fig. 3 permits us to conclude that the evolution of solute concentration with time is well predicted by theory (Eq. 2). Nevertheless, and in order

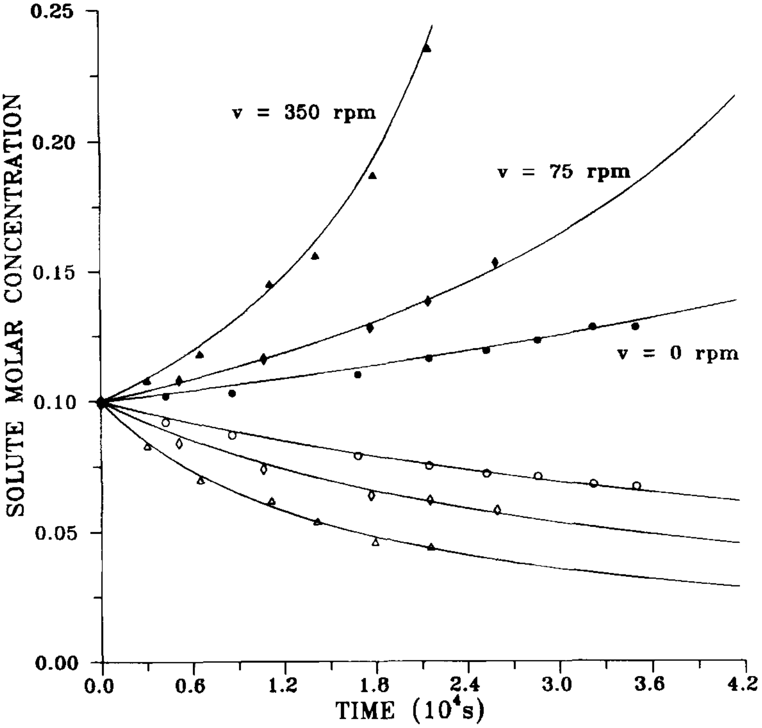

FIG. 3 Time evolution of solute molar concentration on each side of the TF-1000 membrane ($\bar{T}$ = 45°C). The filled (empty) symbols correspond to the hot (cold) side. The curves were obtained theoretically by using Eq. (2).

to be more rigorous, we evaluated the probability that the experimental points are due to statistical fluctuations from the theoretical curve. A statistical data analysis was used (χ^2-distribution procedure). The results obtained have always been better than 95%, which allows us to say that there is actually a significant relationship between the theoretical curve and the experimental points.

Something similar may be obtained via Eq. (1). By using Eq. (1), the solute concentrations may be obtained from the values of the volume. Again, and in order to check our above assumptions, this theoretical curve may be compared with the experimental values. In addition, this test has the advantage that it may be applied to the results of different experiments (different membranes, different mean temperatures, and different stirring rates, the only condition to be fulfilled being that the initial concentration is the same). In Fig. 4 the data corresponding to the membranes TF-1000

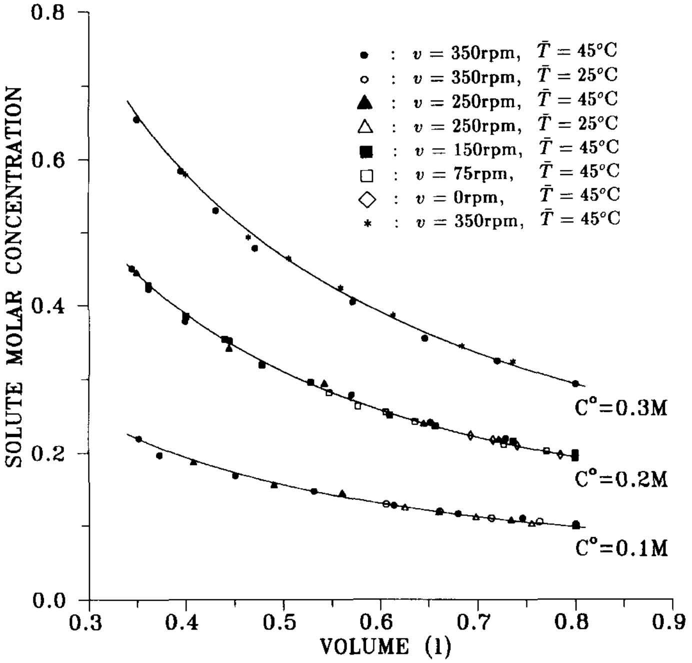

FIG. 4 Solute molar concentration versus volume on the hot side of the membrane. The curves were obtained theoretically by using Eq. (1). The asterisks refer to Membrane TF-200. All other symbols refer to Membrane TF-1000.

and TF-200 and various values of mean temperature and stirring rate are displayed together. As stated before, we can see that agreement between the experimental points and the theoretical curve is fairly good. The same statistical χ^2-analysis carried out above results in a 95% probability that there is a relationship between the experimental points and the theoretical curve.

In order to study the influence of the various parameters on the separation coefficient, an arbitrary time of 6 hours was chosen. The corresponding values of $s(t)$ are displayed in Tables 1–3 which correspond to membranes TF-1000, TF-450, and TF-200, respectively. As expected, the separation coefficient increases with mean temperature. Tables 1–3 show that the influence of the stirring rate on the separation is very important. In fact, separations obtained with the maximum stirring rate reach values up to three times higher than those obtained in the absence of stirring.

In order to check the validity of the hypothesis leading to Eq. (4), a nonlinear χ^2-minimization computer program has been used to fit the pairs

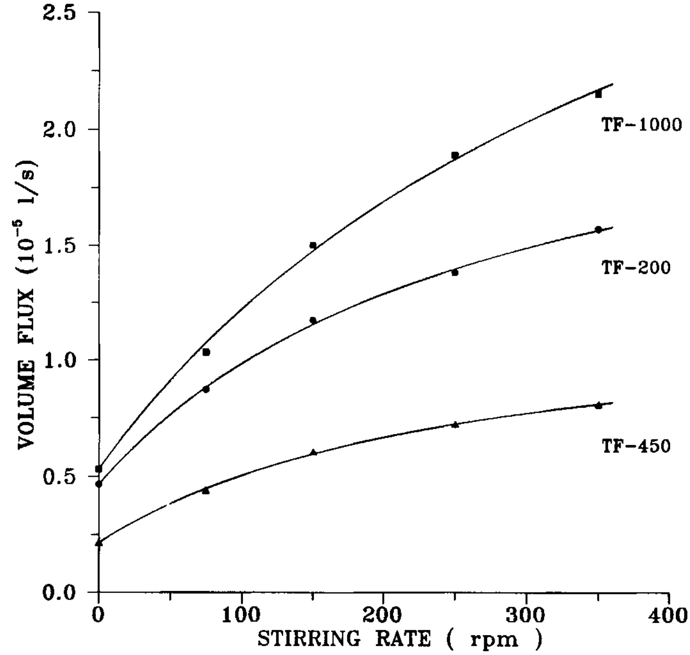

FIG. 5 Variation of volume flux with stirring rate. C^0 = 0.1 M, $\overline{T}$ = 45°C for Membranes TF-1000 and TF-200, and $\overline{T}$ = 25°C for Membrane TF-450.

of data $\{J(v), v\}$ obtained by following Eq. (4). The results are displayed in Fig. 5 for some cases (the trend is similar in all of them). The agreement between the experimental points and the theoretical curve (Eq. 4) is good. This fact shows that the effects of concentration polarization may be neglected or, at least, included in Eq. (4) itself, and thus the hypothesis leading to Eq. (4) is fulfilled.

Finally, if one extrapolates the values corresponding to $v = \infty$, the value of J_∞ is obtained. This value permits us to obtain $s_\infty(t)$ via Eq. (5). The results of these calculations are shown in Table 4. In order to quantify the numerical influence of stirring rate on separation, the values of $s_\infty(t)$ may be compared with those corresponding to a stirring rate of 350 rpm, $s_{350}(t)$ (which corresponds to the higher value of the stirring rate reached with our experimental setup). The result is that we are very far below the maximum value (about 71% in the most unfavorable case). As a consequence, the unstirred layers are not completely eliminated and their effects, which may be quantified, are very important. This suggests that this phenomenon must be taken into account in order to design new experimental devices.

TABLE 4

Extrapolated Values for $v = \infty$ of Volume Flux and Separation Coefficients after 6 Hours for the Different Membranes and Experimental Conditions. The Last Column (s_{350}) Has Been Taken from Tables 1–3

Membrane	$\bar{T}$ (°C)	C^0 (mol/L)	$J \times 10^5$ (L/s)	s_∞ (%)	s_{350} (%)
TF-1000	25	0.1	1.28	100	65
		0.2	1.53	120	70
		0.3	1.45	110	70
	45	0.1	3.20	700	190
		0.2	3.00	500	180
		0.3	2.78	350	175
TF-450	25	0.1	1.15	85	55
		0.2	1.35	100	60
		0.3	1.22	90	60
	45	0.1	2.50	280	115
		0.2	2.37	240	110
		0.3	1.87	160	105
TF-200	25	0.1	1.17	85	60
		0.2	1.15	85	60
		0.3	1.15	85	60
	45	0.1	2.45	250	120
		0.2	2.18	200	110
		0.3	2.25	210	115

NOTATIONS

C	molar concentration
C^0	initial molar concentration
V	volume
T	absolute temperature
t	time
J	volume flux
J_0	volume flux without stirring
s	separation coefficient
v	stirring rate
X	adjustment parameter
Y	adjustment parameter

Subscripts

h	value corresponding to the hot side
c	value corresponding to the cold side
∞	value corresponding to infinite stirring rate

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