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Batch dialysis of phosphoric acid. Quantification of acid and water transport
through Fumasep-FAD membrane

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Abstract

Diffusion dialysis of model aqueous solutions of phosphoric acid was investigated in a two-compartment batch cell equipped with an anion-exchange membrane Fumasep-FAD. The transport of acid through the membrane was accompanied by a significant water transport in the same direction. In order to quantify both these phenomena, the permeability coefficient of the membrane for acid and proportionality coefficient between the flux of water and that of acid were determined from the experimental dependences of acid concentrations and liquid volumes in both the compartments upon time. This study proved that the membrane used is highly permeable for phosphoric acid, i.e., the permeability coefficient was found to be in the range from 0.67×10^{-5} to $3.25 \times 10^{-5} \text{ m s}^{-1}$. Moreover, water transport can satisfactorily be quantified by only one proportionality coefficient with the value of 7.11.

Keywords: diffusion dialysis; anion-exchange membrane; phosphoric acid; batch cell; water transport

1. Introduction

Diffusion dialysis is a simple membrane separation technique using polymeric ion-exchange membranes. A characteristic of these membranes is the presence of negatively or positively charged fixed sites in their structures. The separation efficiency of such a membrane is then determined not only by complex interactions between the components transported and the membrane matrix but also by interactions between electrically charged species and charged sites. For the separation of acids from acid solutions, anion-exchange membranes are used, while the separation of alkalis needs cation-exchange membranes. Due to the diffusion character of the process, the transport rate is very low so that diffusion dialysis is suitable for the treatment of concentrated solutions. Although diffusion dialysis has a limited utilisation, i.e., in the separation of acids or alkalis from concentrated solutions, a relatively large number of papers dealing with this process have been published since the year 2000 [1-34]. These papers showed that namely inorganic acids can effectively be recovered from a series of acid solutions containing acids and their salts. The only exception is the recovery of hydrochloric acid from acidic solutions with Zn — in this case, a high zinc leakage through the membrane was observed [1,6,12].

The transport of components through the membrane is always accompanied by changes in liquid volumes on both the sides of the membrane. These changes are consequence not only of the component transport but also of the transport of water, which can occur in both the directions. In the direction from the dialysate side to the diffusate side, water is transported due to hydration effects, while its transport in the opposite direction is a result of osmosis. Although diffusion dialysis has extensively been investigated for many years, the transport of water has been studied only by several researchers [5,35-38].

In dialysis of a sulphuric acid and nickel sulphate mixture, Xu and Yang [5] quantified the transport of water by the apparent water osmosis coefficient, which was calculated from the dependence of the liquid volume on the feed side upon time obtained in a batch cell of a special construction. In continuous dialysis of the same mixture using the apparatus of an industrial scale, the volumetric expansion factor defined as the ratio of the volumetric liquid flow rate of the dialysate to that of the feed was used to quantify water transport. This characteristic was always higher than 1 and was strongly affected by cross-linking time. In the separation of hydrochloric

acid from acidic HCl/glyphosate liquor by continuous diffusion dialysis, the transport of water was quantified by the water osmosis coefficient which was calculated from the volumetric liquid flow rates of the feed and dialysate [35]. Zhang *et al.* [36] characterised the water quantity transported by the difference between the volumetric liquid flow rate of the dialysate and that of the feed, both the volumetric liquid flow rates being calculated from the component balance and volume balance. In this case, dialysis of an HCl + AlCl₃ mixture was investigated in a spiral wound diffusion dialysis membrane module equipped with the DF-120 commercial membrane. In literature [37], the mathematical model for the quantification of salt (NaCl) and water transport through the membrane was developed. The transport of sodium chloride and that of water was quantified by the permeability coefficients of the membrane to NaCl and water. In both the cases, the driving forces of the process were based on the chemical potential differences. The experiments were carried out with a conventional plate and frame dialysis module operated in batch mode. The comparison of the experimental NaCl concentrations in difusate solutions with the calculated ones revealed that a good agreement between the model and experiment exists. The same model was used to quantify mass transport and water osmosis in diffusion dialysis of hydrochloric acid solutions using the same apparatus and membrane (DF-120) [38].

With respect to the finding that limited attention was paid to water transport through polymeric ion-exchange membranes, the aim of this paper is to develop a simple mathematical model enabling satisfactory quantification of component and water transport. For this purpose, aqueous solutions of phosphoric acid were selected as model solutions. Moreover, phosphoric acid is a component of the etching solution (a mixture of acetic, nitric, and phosphoric acids) used in the semiconductor industry, and from spent solutions it can be recovered by diffusion dialysis with subsequent vacuum distillation [39]. From this point of view, experimental data on dialysis of solutions containing only phosphoric acid can be considered as reference data for quantification of dialysis of complex mixtures.

2. Theory

If compartment I of a batch dialyzer is initially filled with a solution (component A and water *w*) and compartment II with water, then unsteady state transport of component A in the direction from compartment I to compartment II exists — see Fig. 1. At the same time, the transport of water can occur in both the directions. Its resulting direction depends on what type of the water transport through the membrane prevails. In the direction from compartment I to compartment II, water is transported due to hydration effects, and the water flux, $J_{w,hydr}$, can simply be expressed as [38]

$$J_{w,hydr} = \xi J_A \quad (1)$$

where ξ is a proportionality coefficient and J_A is the molar flux of component A. The water transport in the opposite direction, i.e., the transport due to osmosis, $J_{w,osm}$, is quantified by the following equation

$$J_{w,osm} = P_{w,osm} (c_{wi}^{II} - c_{wi}^I) \quad (2)$$

In Eq. (2), $P_{w,osm}$ is the permeability coefficient of the membrane for water and c_{wi}^j ($j = I, II$) are the molar concentrations of water at the solution/membrane interfaces. The water concentration

can be calculated from the following equation

$$c_A \bar{v}_A + c_w \bar{v}_w = 1 \quad (3)$$

where c_A is the molar concentration of component A, \bar{v}_A and \bar{v}_w are the partial molar volumes of component A and water.

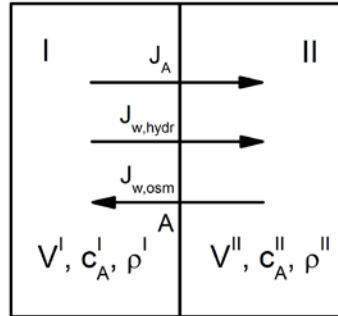


Fig. 1 Balance scheme of two-compartment batch dialyzer

The balance of molar amount of component A in compartment I and that in compartment II over the differential time interval, $d\tau$, results in

$$\frac{d(V^I c_A^I)}{d\tau} = -J_A A \quad (4)$$

$$\frac{d(V^{II} c_A^{II})}{d\tau} = J_A A \quad (5)$$

In Eqs (4) and (5), V^j ($j = I, II$) are the liquid volumes and A is the membrane area.

From the balance of the total mass in the individual compartments, one can obtain the following equations

$$\begin{aligned} \frac{d(V^I \rho^I)}{d\tau} &= -A(J_A M_A + J_{w,hydr} M_w - J_{w,osm} M_w) = \\ &= -A[(M_A + \xi M_w) J_A - P_{w,osm} (c_{wi}^{II} - c_{wi}^I) M_w] \end{aligned} \quad (6)$$

$$\begin{aligned} \frac{d(V^{II} \rho^{II})}{d\tau} &= A(J_A M_A + J_{w,hydr} M_w - J_{w,osm} M_w) = \\ &= A[(M_A + \xi M_w) J_A - P_{w,osm} (c_{wi}^{II} - c_{wi}^I) M_w] \end{aligned} \quad (7)$$

where ρ is density of liquid and M is the molar mass.

Using the substitution

$$\frac{d\rho^j}{d\tau} = \frac{d\rho^j}{dc_A^j} \frac{dc_A^j}{d\tau} \quad (j = \text{I, II}) \quad (8)$$

the set of Eqs (4) – (7) can be solved, so that the derivatives $\frac{dc_A^j}{d\tau}$ and $\frac{dV^j}{d\tau}$ ($j = \text{I, II}$) are obtained

$$\frac{dc_A^{\text{I}}}{d\tau} = -A \frac{[\rho^{\text{I}} - (M_A + \xi M_w)c_A^{\text{I}}]J_A + P_{w,\text{osm}}(c_{wi}^{\text{II}} - c_{wi}^{\text{I}})M_w c_A^{\text{I}}}{V^{\text{I}} \left(\rho^{\text{I}} - c_A^{\text{I}} \frac{d\rho^{\text{I}}}{dc_A^{\text{I}}} \right)} \quad (9)$$

$$\frac{dc_A^{\text{II}}}{d\tau} = A \frac{[\rho^{\text{II}} - (M_A + \xi M_w)c_A^{\text{II}}]J_A + P_{w,\text{osm}}(c_{wi}^{\text{II}} - c_{wi}^{\text{I}})M_w c_A^{\text{II}}}{V^{\text{II}} \left(\rho^{\text{II}} - c_A^{\text{II}} \frac{d\rho^{\text{II}}}{dc_A^{\text{II}}} \right)} \quad (10)$$

$$\frac{dV^{\text{I}}}{d\tau} = -A \frac{\left(M_A + \xi M_w - \frac{d\rho^{\text{I}}}{dc_A^{\text{I}}} \right) J_A - P_{w,\text{osm}}(c_{wi}^{\text{II}} - c_{wi}^{\text{I}})M_w}{\rho^{\text{I}} - c_A^{\text{I}} \frac{d\rho^{\text{I}}}{dc_A^{\text{I}}}} \quad (11)$$

$$\frac{dV^{\text{II}}}{d\tau} = A \frac{\left(M_A + \xi M_w - \frac{d\rho^{\text{II}}}{dc_A^{\text{II}}} \right) J_A - P_{w,\text{osm}}(c_{wi}^{\text{II}} - c_{wi}^{\text{I}})M_w}{\rho^{\text{II}} - c_A^{\text{II}} \frac{d\rho^{\text{II}}}{dc_A^{\text{II}}}} \quad (12)$$

The differential equations (9) – (12) are the basic equations describing the dependences of the component concentrations and liquid volumes in the individual compartments of a batch dialyzer upon time. The initial conditions are

$$\tau = 0, \quad c_A^{\text{I}} = c_{A0}^{\text{I}}, \quad c_A^{\text{II}} = c_{A0}^{\text{II}} = 0, \quad V^{\text{I}} = V_0^{\text{I}}, \quad V^{\text{II}} = V_0^{\text{II}} \quad (13)$$

where subscript 0 means the initial value.

The application of Eqs (9) – (12) needs the flux of component A through the membrane. This quantity can be obtained by solving the set of equations describing not only the transport of component A through the membrane (Eq. (15)) but also that through the liquid films (Eqs (14) and (16)), whose existences are supposed on both the sides of the membrane — see Fig. 2 in Refs. [2,8].

$$J_A^{\text{I}} = k_L^{\text{I}}(c_A^{\text{I}} - c_{Ai}^{\text{I}}) \quad (14)$$

$$J_{AM} = P_A (c_{Ai}^I - c_{Ai}^{II}) \quad (15)$$

$$J_A^{II} = k_L^{II} (c_{Ai}^{II} - c_A^{II}) \quad (16)$$

In Eqs (14) – (16), P_A is the permeability coefficient of the membrane for component A, and k_L^j ($j = I, II$) are the liquid mass transfer coefficients which can be estimated from equation

$$Sh = CRe_m^{0.50} Sc^{0.33} \quad (17)$$

where Sh is the Sherwood number, C is a constant, Re_m is the Reynolds number for mixing and Sc is the Schmidt number.

Supposing that the real state is not far from the pseudo-steady state ($J_A^I = J_{AM} = J_A^{II} = J_A$), then the solution to the set of Eqs (14) – (16) is in the form

$$J_A = P_A \frac{k_L^I k_L^{II} (c_A^I - c_A^{II})}{P_A (k_L^I + k_L^{II}) + k_L^I k_L^{II}} \quad (18)$$

If experimental data on the component concentrations and liquid volumes in both the compartments are at disposal, it is possible to quantify the diffusion dialysis process. For this purpose, a two-step procedure can be used. In the first step, the set of Eqs (9) – (12) with the initial conditions (13) is numerically integrated which results in obtaining the calculated data. If this step is followed by an optimisation method ensuring the best agreement between the experimental and calculated data, then the permeability coefficient, P_A , and proportionality coefficient, ξ , or permeability coefficient for water, $P_{w,osm}$, are found.

3. Experimental

The transport of phosphoric acid through the polymeric membrane was investigated in a batch dialyzer consisting of two identical well mixed compartments. Its description including experimental set-up and experiments is given in detail elsewhere [2,7,8,16]. In all the experiments, an anion-exchange membrane Fumasep FAD (Fumatech BTW GmbH, Germany) was used. The membrane area was $62.2 \times 10^{-4} \text{ m}^2$. At the beginning of the experiment, compartment I was filled with an H_3PO_4 ($1 \times 10^{-3} \text{ m}^3$), while distilled water ($1 \times 10^{-3} \text{ m}^3$) was poured into compartment II. At the constant initial acid concentration in compartment I (in the limits from 0.2 to 2.0 kmol m^{-3}), the rotational speed of stirrers was changed from 1.67 to 8.33 s^{-1} . In the course of each experiment, whose duration was 7 h, the heights of liquid levels (to determine the volume changes) were measured, and at the same time, samples from both the compartments were taken for analysis. The changes in the levels of liquid were measured with the help of a modified digital micro-meter screw with a needle. In such a way, the distance between a reference level (top of the cell) and the liquid level was determined and subsequently recalculated into the liquid volume. The concentration of phosphoric acid was determined by titration with a standard NaOH solution. The temperature was $25 \pm 0.5 \text{ }^\circ\text{C}$.

4. Data treatment and discussion

The transport of H_3PO_4 through the membrane was quantified by the permeability coefficient, P_A . For its determination, the experimental dependences of acid concentration in both the compartments upon time were utilised. For illustration, they are shown in Figs. 2 and 3. In the course of each experiment, the liquid volume in compartment I markedly decreased with time, while that in compartment II increased — see Figs 4 and 5. For this reason, water transport was characterised by the proportionality coefficient, ξ . In order to determine these characteristics, the procedure consisting of the following steps was used:

1. The initial estimates of P_A and ξ .
2. The numerical integration of Eqs (9) – (12), where the flux of acid was expressed by Eq. (18). For this purpose, the fourth order Runge–Kutta method was used, the integration step being 0.001 h. In this step, the calculated values of the liquid volumes and acid concentrations in both the compartments are obtained.
3. The calculation of the objective function

$$F = \sum_{i=1}^m \left[\left(\frac{c_{Ai}^{\text{I,exp}} - c_{Ai}^{\text{I,calc}}}{c_{Ai}^{\text{I,exp}}} \right)^2 + \left(\frac{c_{Ai}^{\text{II,exp}} - c_{Ai}^{\text{II,calc}}}{c_{Ai}^{\text{II,exp}}} \right)^2 + \left(\frac{V_i^{\text{I,exp}} - V_i^{\text{I,calc}}}{V_i^{\text{I,exp}}} \right)^2 + \left(\frac{V_i^{\text{II,exp}} - V_i^{\text{II,calc}}}{V_i^{\text{II,exp}}} \right)^2 \right] \quad (19)$$

where m is the number of experimental points in one time series. (Note: The objective function (19) was considered to be a function not only of permeability of the membrane but also of the constant C in Eq. (17). Moreover, the algorithm used enabled the calculation of the permeability coefficient for water, $P_{w,\text{osm}}$.)

4. The calculation of corrected values of P_A and ξ (and also those of C and $P_{w,\text{osm}}$) by the realisation of one step of an optimisation method. Here, a simplex method for nonlinear programming was used.
5. The procedure from 2) to 4) was repeated until a minimum of the objective function (19) was reached.

The adequacy of the model developed and represented by Eqs (9) – (18) can be evaluated by comparing the experimental data with the calculated data on the acid concentrations and liquid volumes in the individual compartments of the dialyzer. For the highest rotational speed of the stirrers, these comparisons are presented in Figs 2 – 5. Moreover, the root mean square deviations of the acid concentrations and liquid volumes are summarised in Table 1. Based on the graphical comparison and data in Table 1, it can be concluded that the model fits the data very well.

The characteristics of acid and water transport through the membrane are given in Table 2. As seen, the anion-exchange membrane Fumasep-FAD exhibits very high permeability for phosphoric acid. The permeability coefficient is in the range from 0.67×10^{-5} to $3.26 \times 10^{-5} \text{ m s}^{-1}$, which can be attributed to a relative small thickness of the membrane, i.e., approx. 80 μm . On the other hand, the permeability coefficient for water is negligible — it does not exceed $9 \times 10^{-14} \text{ m s}^{-1}$, which reflects the fact that water is transported by hydration. Other characteristic of water transport, i.e., the proportionality coefficient, ξ , lies in the relatively narrow range — from 6.10 to 8.96. With respect to this fact, the mean value of ξ was calculated and consequently used in the calculation of the remaining characteristics — see Table 3. Besides, new statistical characteristics were determined and are summarised in Table 4. Based on the data in Tables 3 and 4, it can be

concluded that water transport through the membrane can satisfactorily be quantified by only one proportional coefficient, i.e., $\xi_{\text{mean}} = 7.11$. This simplification has practically no effect on P_A , C and $P_{w,osm}$, and only elevated root mean square deviations of liquid volumes in compartments I and II at the initial acid concentration 1.0 and 2.0 kmol m^{-3} were found. In comparison with the experimental liquid volumes obtained at the highest initial acid concentration in compartment I, the calculated liquid volumes in compartment I were underestimated, while those in compartment II were overestimated.

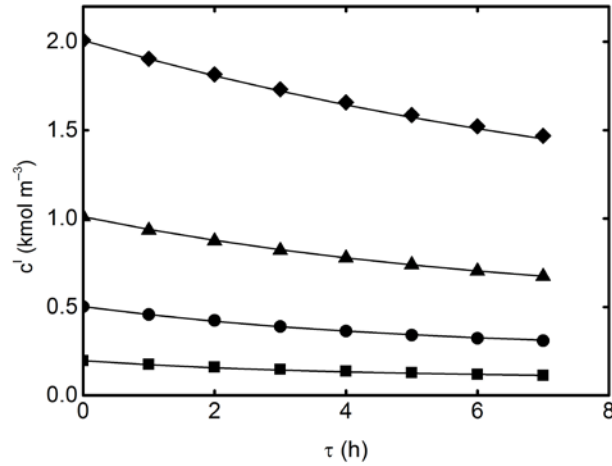


Fig. 2 Dependence of acid concentration in compartment I upon time for $n^I = n^{II} = 8.33 \text{ s}^{-1}$.
 c_{A0}^I [kmol m^{-3}]: ■ - 0.2; ● - 0.5; ▲ - 1.0; ◆ - 2.0; ——— calculated

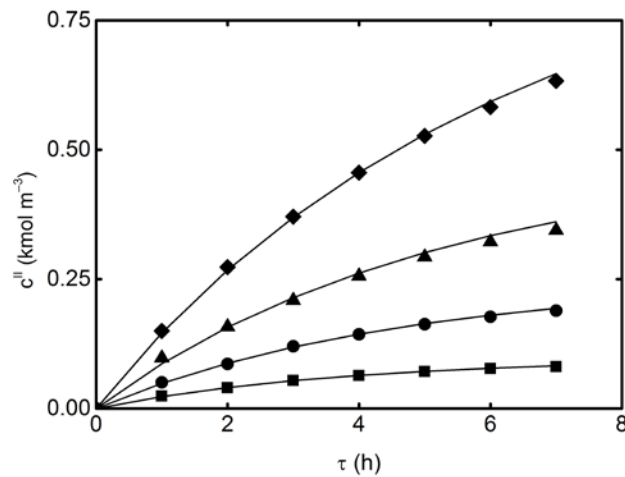


Fig. 3 Dependence of acid concentration in compartment II upon time for $n^I = n^{II} = 8.33 \text{ s}^{-1}$.
 For meaning symbols, see Fig. 2

Table 1 Root mean square deviations ($RMSD_y$) of acid concentrations and liquid volumes for $n^I = n^{II} = 8.33 \text{ s}^{-1}$

c_{A0}^I kmol m^{-3}	$RMSD_{c_A^I} \times 10^3$ kmol m^{-3}	$RMSD_{c_A^{II}} \times 10^3$ kmol m^{-3}	$RMSD_{V^I} \times 10^6$ m^3	$RMSD_{V^{II}} \times 10^6$ m^3
0.2	3.15	0.90	1.07	2.77
0.5	2.32	2.40	3.55	2.13
1.0	2.74	9.83	1.93	2.82
2.0	1.21	7.35	5.88	8.16

$$RMSD_y = \sqrt{\frac{1}{m} \sum_{i=1}^m (y_i^{\text{exp}} - y_i^{\text{calc}})^2} \quad (y = c_A^I, c_A^{II}, V^I, V^{II})$$

Table 2 Characteristics of acid and water transport

c_{A0}^I kmol m^{-3}	$P_A \times 10^5$ m s^{-1}	C	ξ	$P_{w,osm} \times 10^{15}$ m s^{-1}
0.2	3.26	0.516	6.64	6.76
0.5	1.49	0.528	6.73	3.61
1.0	0.89	0.615	8.96	24.39
2.0	0.67	0.585	6.10	85.34

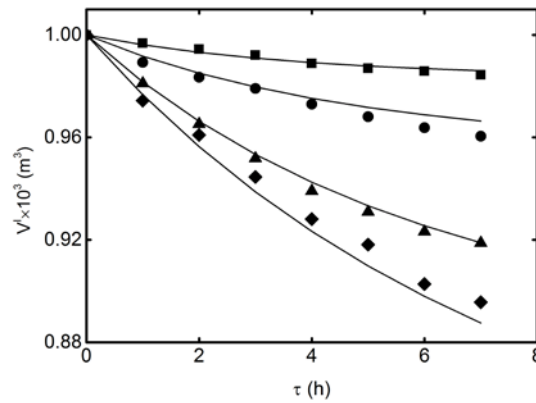


Fig. 4 Dependence of liquid volume in compartment I upon time for $n^I = n^{II} = 8.33 \text{ s}^{-1}$. For meaning symbols, see Fig. 2

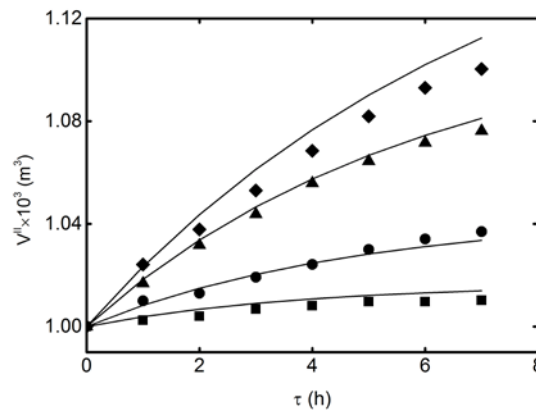


Fig. 5 Dependence of liquid volume in compartment II upon time for $n^I = n^{II} = 8.33 \text{ s}^{-1}$. For meaning symbols, see Fig. 2

Table 3 Characteristics of dialysis process for $\xi_{\text{mean}} = 7.11$

c_{A0}^I kmol m ⁻³	$P_A \times 10^5$ m s ⁻¹	C	$P_{w,osm} \times 10^{15}$ m s ⁻¹
0.2	3.27	0.516	4.79
0.5	1.50	0.528	4.77
1.0	0.88	0.615	12.28
2.0	0.68	0.586	6.07

Table 4 Root mean square deviations of acid concentrations and liquid volumes for $n^I = n^{II} = 8.33$ s⁻¹ and $\xi_{\text{mean}} = 7.11$

c_{A0}^I kmol m ⁻³	$RMSD_{c_A^I} \times 10^3$ kmol m ⁻³	$RMSD_{c_A^{II}} \times 10^3$ kmol m ⁻³	$RMSD_{V^I} \times 10^6$ m ³	$RMSD_{V^{II}} \times 10^6$ m ³
0.2	3.12	0.90	1.00	3.30
0.5	2.36	2.40	2.60	1.76
1.0	3.95	9.94	11.50	7.17
2.0	1.91	6.86	15.70	18.10

5. Conclusion

A new mathematical model was developed, which consists of four ordinary differential equations describing the dependences of component concentrations and liquid volumes in the individual compartments of a batch dialyzer upon time. It considers not only the transport of a component but also that of water due to hydration effects and osmosis. Using experimental data on dialysis of aqueous solutions of phosphoric acid, the acid and water transport through an anion-exchange membrane Fumasep-FAD (Fumatech GmbH, Germany) was quantified. For this purpose, the permeability coefficient and proportionality constant between the flux of water and that of acid were determined with the help of a two-step procedure combining the numerical integration of the basic differential equations and multivariable optimisation method for nonlinear programming.

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6. Symbols

A	membrane area, m ²
C	constant in Eq. (17)
c	molar concentration, kmol m ⁻³
D	diffusion coefficient, m ² s ⁻¹

d	diameter of stirrer, m
F	objective function
J	flux, $\text{kmol m}^{-2} \text{s}^{-1}$
k_L	liquid mass transfer coefficient, m s^{-1}
P	permeability, m s^{-1}
M	molar mass, kg kmol^{-1}
m	number of experimental points in one time series
n	rotational speed of stirrer, s^{-1}
Re_m	$(= nd^2/\nu)$ Reynolds number for mixing
$RMSD$	root mean square deviation of dependent variable, $\text{m}^3, \text{kmol m}^{-3}$
Sc	(ν/D) Schmidt number
Sh	$(= k_L d/D)$ Sherwood number
V	volume, m^3
\bar{v}	partial molar volume, $\text{m}^3 \text{kmol}^{-1}$
ν	kinematic viscosity, $\text{m}^2 \text{s}^{-1}$
ρ	density, kg m^{-3}
τ	time, h
ξ	proportionality coefficient

Superscripts and subscripts

A	referred to component A, i.e., phosphoric acid
calc	calculated
exp	experimental
hydr	hydration
i	solution/membrane interface
M	referred to membrane
osm	osmosis
mean	mean value
w	referred to water
y	referred to dependent variable
0	initial
I	referred to compartment I
II	referred to compartment II

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