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Separation of Carboxylic Acids from Carboxylates by Diffusion Dialysis

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Abstract: Acetic acid/sodium acetate and propionic acid/sodium propionate were separated by the diffusion dialysis technique using Neosepta AFN-7 and Selemion DSV membranes. Accounting for molarities of carboxylic acids and salts in broths produced by the pH controlled bacterial fermentation (pH 4–6), the experiments were carried out with carboxylates in excess of acids. The sorption equilibria established for acetic acid, propionic acid, and the sodium salts of both acids (single solute experiments) revealed a high sorption of acids in both membranes and the rejection of carboxylates. The partition coefficients were found to be from unity up to 2 for the acids and 0.04–0.05 for the salts. Reflecting a high sorption, the fluxes for the acids amounted from 1.5 to 2.0 $\mathrm{mol \cdot m^{-2} \cdot h^{-1}}$ and only 0.07–0.08 $\mathrm{mol \cdot m^{-2} \cdot h^{-1}}$ for the salts (one molar solutions). The separation factors computed upon the experiments performed with the ternary solutions were found between 20 to 37 for the Neosepta AFN-7 membrane and about 29 for the Selemion DSV membrane. Accounting additionally, for the results of the separation of lactic acid from sodium lactate reported in our earlier paper the results prove the diffusion dialysis to be applicable to the separation of mean strength and weak carboxylic acids from their salts.

Keywords: Diffusion dialysis, anion-exchange membranes, carboxylic acids, carboxylates, sorption

INTRODUCTION

The diffusion dialysis (DD) is an energy-saving separation technique devised for the recovery of strong acids from wastes of the steel-making, metal

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refining, electroplating and chemical industry. Since 1984, the technique has been adopted to the practice (1, 2). The acid/salt separation factors depend on valences of ions ranging from ~20 for the uni-uni valent components in a mixture like HCl/NaCl up to a hundred or more for $HNO_3/Al(NO_3)_3$ and $H_3PO_4/MgHPO_4$. The data published up to date have concerned mainly the separation of strong acids from salts (3–16).

In our search for a technique of the separation of lactic acid from sodium lactate we have examined the diffusion dialysis and found the separation factors $S_{acid/salt}$ amounting to ~20 for the Neosepta AFN-7 and ~30 for the Selemion DSV membranes (17). Accounting for the dissociation constant of lactic acid ($pK = 3.86$), the concentration of undissociated molecules in dilute solutions of the acid reaches 90% or higher of the total concentration. For the acid absorbed into a membrane dissociation is still hindered against its dissociation in a bulk. Thus, the molecular level fundamentals for the separation and transport of weak acids should be different from those for strong electrolytes.

A success made with lactic acid has motivated us to examine the diffusion dialysis for the separation of still weaker carboxylic acids from their salts, that is acetic acid ($pK = 4.76$) from sodium acetate and propionic acid ($pK = 4.87$) from sodium propionate. The experiments performed with those compounds were not of purely scientific interest. Carboxylic acids and carboxylates in mixtures are the products of the bacterial fermentation of carbohydrates (glucose, lactose, sacharose). Since acid molecules are toxic for bacteria, the pH of the broth cannot be lower than 4, and is mostly in the range of the pH 5–6. Certainly, for acids of different strength the pH 4 to 6 means different acid to salt ratio always, however, with an excess of salt to acid.

Referring to the low concentration of acids in broth we have examined the diffusion dialysis for the isolation of carboxylic acids starting from $c = 0.01 \text{ mol} \cdot \text{dm}^{-3}$.

As for any diffusional separation technique, the discrimination in transport rates in DD is due to the differences in the solubilities or diffusivities of permeants within a membrane. To highlight the fundamentals for the separation of weak acids from salts by DD technique, the sorption equilibria, the transport rates from single component solutions, and the separation from mixtures for acetic acid/sodium acetate (HAc/NaAc) and propionic acid/sodium propionate (HPr/NaPr) have been measured and discussed.

EXPERIMENTAL

Membranes

Two strong base anion exchange membranes, the same as for the experiments with lactic acid solutions (17) that is the Neosepta AFN-7 by Tokuyama Co.

Japan and the Selemion DSV by Asahi Glass Co., Japan were taken for the experiments. The characteristics of the membranes were published in (17).

The membranes were prepared in acetic and propionic forms (with acetic and propionic counterions). By a chemical nature, the membranes with both counterions are polysalts of a strong polymeric base neutralized by weak acids. As they undergo the hydrolysis, precautions were undertaken to differentiate between an exchanged and physically sorbed (dissolved) acid. The details of the experimental procedure were described in (17).

Experimental Techniques

To determine sorption, the samples of the membranes were equilibrated with acid or salt solutions of molarities from 0.1 to 1.0, and for each equilibrium state, the amount of solutes absorbed and the water content were measured (Tables 1, 2). The time of equilibration was five hours for acids and twelve hours for salts. The capillary electrophoresis (EA-100, Villa Labeco, Slovakia), has been used for determination of the amount of eluted acid/salt.

The permeation experiments for acids and salts from single solute solutions were carried out using an automatic set-up developed in our laboratory (17–19). For each component, the conductivity of a permeate in a receiver was monitored continuously, stored and processed automatically. The permeability coefficients were calculated on the basis of Fick's law adopted to the experimental findings (17–19):

$$\frac{l}{h}\ln\left[\frac{c_{\mathrm{f}}^{0} - c_{\mathrm{s}}^{\mathrm{t}}(1+b) + bc_{\mathrm{s}}^{0}}{c_{\mathrm{f}}^{0} - c_{\mathrm{s}}^{0}}\right] = -Pt \qquad (1)$$

In Eq. (1) symbols denote: l – membrane thickness; $h = [A(1+b)]/V_{\mathrm{s}}$; $b = V_{\mathrm{s}}/V_{\mathrm{f}}$; A – area of a membrane and P – permeability coefficient. The indexes correspond with feed (f) and permeate (s) solutions. The time of each permeation experiment was 10 hours. The accumulation of solutes in a permeate was controlled by measuring the conductivity (Elmetron CX-721, Zabrze-Mikulczyce, Poland). The relation between concentration of a solute and conductivity of a solution was predetermined for concentrations expected in the permeates for each of the compounds in study (17). Because of a low conductivity of acids, the accuracy in determining their concentration was ±7%. For the salts the accuracy was ±5%.

A scheme and details of the automatically controlled diffusion dialyzer used for the separation experiments were presented in (17). The ternary solutions contained 0.05 $\mathrm{mol \cdot dm^{-3}}$ of an acid and 0.2 $\mathrm{mol \cdot dm^{-3}}$ of a salt. The accumulation of these compounds in a permeate was controlled by

Table 1. Sorption Equilibria. Concentration of acetic acid and sodium acetate in Neosepta AFN-7 and Selemion DSV membranes and the partition coefficients (single solute experiments)

	Internal membrane solution			
External solution (mol · dm^{-3}) c	(mol · dm^{-3}) $\bar{c}$	Swelling g/g	Molality $\bar{m}$	k
Neosepta AFN-7 membrane				
Acetic acid				
0.07	0.14	0.457	0.31	2.00
0.23	0.32	0.457	0.72	1.40
0.48	0.57	0.458	1.26	1.18
0.75	0.84	0.460	1.87	1.12
1.03	1.13	0.462	2.52	1.10
Sodium acetate				
0.10	0.004	0.455	0.01	0.04
0.25	0.01	0.454	0.02	0.04
0.50	0.02	0.453	0.05	0.04
0.75	0.03	0.451	0.07	0.04
1.00	0.04	0.450	0.10	0.04
Selemion DSV membrane				
Acetic acid				
0.08	0.09	0.397	0.21	1.14
0.24	0.23	0.397	0.58	0.99
0.48	0.40	0.399	1.03	0.84
0.75	0.60	0.401	1.54	0.80
1.03	0.75	0.403	1.93	0.73
Sodium acetate				
0.10	0.003	0.396	0.01	0.03
0.24	0.01	0.395	0.02	0.03
0.50	0.02	0.394	0.04	0.03
0.75	0.03	0.392	0.07	0.03
1.00	0.04	0.391	0.10	0.04

measuring the pH and conductivity (Elmetron CX-721, Zabrze-Mikulczyce, Poland). Accounting for the nature of the components (acid, salt) forming a buffer, the concentrations of solutes were computed by solving the equations for the pH of a buffer and the polynomials for the conductivity (17). The method was tested by the experiments performed with model solutions containing acids and salts in molarities anticipated in the permeates. Additionally, at the end of each experiment, the concentration of acetic acid and acetate, and propionic acid and sodium propionate were determined by titration with standard NaOH and HCl solutions.

Table 2. Sorption Equilibria. Concentration of propionic acid and sodium propionate in Neosepta AFN-7 and Selemion DSV membranes and the partition coefficients (single solute experiments)

	Internal membrane solution			
External solution (mol · dm^{-3}) c	(mol · dm^{-3}) $\bar{c}$	Swelling g/g	Molality $\bar{m}$	k
Neosepta AFN-7 membrane				
Propionic acid				
0.07	0.13	0.427	0.30	1.83
0.23	0.38	0.428	0.90	1.65
0.48	0.74	0.429	1.75	1.53
0.75	1.12	0.431	2.67	1.50
1.03	1.53	0.432	3.64	1.48
Sodium propionate				
0.10	0.004	0.424	0.01	0.04
0.25	0.01	0.422	0.03	0.04
0.50	0.02	0.421	0.05	0.04
0.75	0.03	0.420	0.08	0.05
1.00	0.05	0.418	0.11	0.05
Selemion DSV membrane				
Propionic acid				
0.08	0.14	0.365	0.39	2.01
0.24	0.36	0.367	0.99	1.55
0.48	0.63	0.368	1.76	1.32
0.75	0.83	0.369	2.13	1.11
1.03	1.02	0.371	2.83	0.99
Sodium propionate				
0.10	0.004	0.364	0.01	0.03
0.24	0.01	0.363	0.03	0.04
0.50	0.02	0.362	0.05	0.04
0.75	0.03	0.360	0.08	0.04
1.00	0.04	0.358	0.13	0.04

RESULTS AND DISCUSSION

Sorption Equilibria for Carboxylic Acids and Sodium Carboxylates

Before discussing the results, it should be stressed that for learning the sorption equilibria with acetic acid and its salt, the membranes were prepared in the acetate form and correspondingly, for propionic acid and sodium propionate in their propionate form. Thus, only the physical sorption was measured, whereas any effect of exchange reactions was eliminated. After the earlier experiments (17), the Donnan rejection for carboxylates and some kind of dissolution of acids within water swelling a membrane were expected.

The corresponding results for the sorption of both acids and salts in Neosepta AFN-7 and Selemion DSV membranes, displayed in moles per volume of a swollen membrane ($\bar{c}$), in moles per kg of swelling water ($\bar{m}$), and as the partition coefficients, $k = \bar{c}/c$ are shown in Tables 1, 2.

The rejection of carboxylates has been confirmed. For the whole concentration range of NaAc and NaPr and for both membranes, the partition coefficient did not exceed 0.05. Different from the salts, the partition coefficients for acetic and propionic acids with the Neosepta AFN-7 and for propionic acid with the Selemion DSV membranes are all over unity for the whole range of concentrations in a bulk. Only for acetic acid and the Selemion DSV membrane the partition coefficients are below unity. Even more spectacular are the internal membrane molalities of the acids. As for the acids, the molarities and molalities do not differ much the direct confrontation is possible. From the data in Table 1, columns 1–4 it can be concluded that at c_{ext} 0.75–1.0 the molalities of the acids in the internal membrane solution are within 2.6–3.6, that is roughly four time over those in the bulk.

The equilibria for the sorption of aliphatic and aromatic acids in the ion exchange resins have already been studied in the fifties (20–22). Since the ion exchange resins and membranes are produced of polyelectrolytes of comparable chemistry, one can expect similar effects, irrespective of the geometry of the system. Reichenberg attributed the sorption of organic acids to:

a. simple dissolution in water swelling the resin,
b. "salting out" by polar groups,
c. London dispersion interactions, and
d. the polar attractive interactions (20).

For aromatic acids, the interaction between penetrants and polymers looks essential. For carboxylic acids, the dissolution in water and the polar attractive interactions should predominate. These acids are known to associate easily in the aqueous solutions forming the cycle dimers in the dilute solutions and open dimers or oligomers in more concentrated ones. More complex three-dimensional structures of associates incorporating water molecules may also form (23, 24).

Considering the membranes and acids studied here one can postulate that the carboxylic counterions play the role of centers where association through hydrogen bondings begins. The concentration of counterions in water swelling the membranes is 5.0–5.3 $\mathrm{mol \cdot kg^{-1}}$ and, together with the amount of the physically sorbed (dissolved) acid the total concentration of associating moieties is 7.8–8.8 $\mathrm{mol \cdot kg^{-1}}$. The molar ratio of the absorbed acid to counterions is 0.6–0.7. Because of the inhomogeneity of an internal membrane structure not every single counterion should participate in the association. Still, taking the high total concentration of acids within membranes into account, we suggest the formation of associated structure of carboxylic acids and water molecules as the origin for a high sorption. The need for the experiments that would clear this phenomena is evident.

Permeabilities of Carboxylic Acids and Sodium Carboxylates (Single Permeant Experiments)

On the basis of data recorded for each permeation experiment, by using Eq. (1), the permeability coefficients and the fluxes ($J = Pc_{\mathrm{f}}^{0}/l$) were calculated. The selected results presenting the accumulation of acetic acid and sodium acetate in the permeate from single solute solutions of molarity 1 mol dm^{-3} with Neosepta AFN-7 are seen in Fig. 1. Such a different sorption of the acids and salts is manifested by different rates of permeation. The concentration dependent fluxes of acetic acid, sodium acetate, propionic acid, and sodium propionate across Neosepta AFN-7 (a) and Selemion DSV (b) membranes are presented in Fig. 2. At the molarity of the compounds in the bulk 1 mol dm^{-3}, the fluxes of the acids are as high as 1.9–2.0 mol $\cdot$ $\mathrm{m}^{-2} \cdot \mathrm{h}^{-1}$ for acetic acid and 1.5–1.9 mol $\cdot$ $\mathrm{m}^{-2} \cdot \mathrm{h}^{-1}$ for propionic acid. For carboxylates, the fluxes do not exceed 0.08 mol $\cdot$ $\mathrm{m}^{-2} \cdot \mathrm{h}^{-1}$. Within the scope of this paper we have limited the molarities of acids in the bulk to one molar, yet, alike the sorption, the fluxes do not show the effects of saturation.

Separation of Carboxylic Acids from Carboxylates by Diffusion Dialysis (Ternary Solution Experiments)

Because the coupling between water and solutes may affect the individual fluxes, the experiments with ternary solutions have been performed to prove

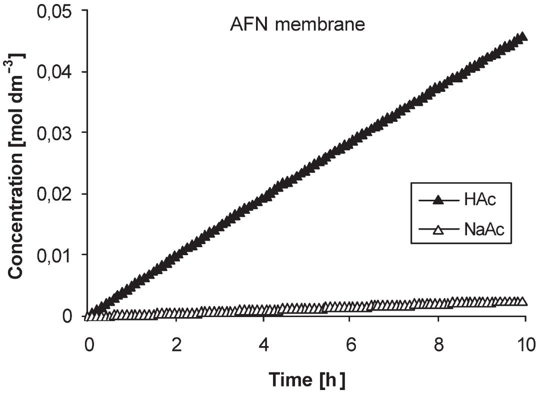

Figure 1. Accumulation of acetic acid/sodium acetate in the permeate with Neosepta AFN-7 membrane (single solute experiments; $c_{\mathrm{HAc}} = 1.0\ \mathrm{mol} \cdot \mathrm{dm}^{-3}$, $c_{\mathrm{NaAc}} = 1.0\ \mathrm{mol} \cdot \mathrm{dm}^{-3}$).

the separation ability of the system. Considering the fact that the concentration of carboxylic acids in broth is always below that of carboxylates (pH control fermentation), the experiments were carried out with the solutions containing 0.05 mol $\cdot$ dm^{-3} of the acid, and 0.2 mol $\cdot$ dm^{-3} of sodium carboxylate. Thus, the ratio of the components was rather unfavorable for the separation, and the final effect was to be revised.

The rates of the accumulation of the acids and salts in the permeates are seen in Fig. 3.

With these data, the dialysis coefficients meaning the flux per unit concentration difference between the feed and the permeate,

$$U = \frac{W}{A\Delta c} \tag{2}$$

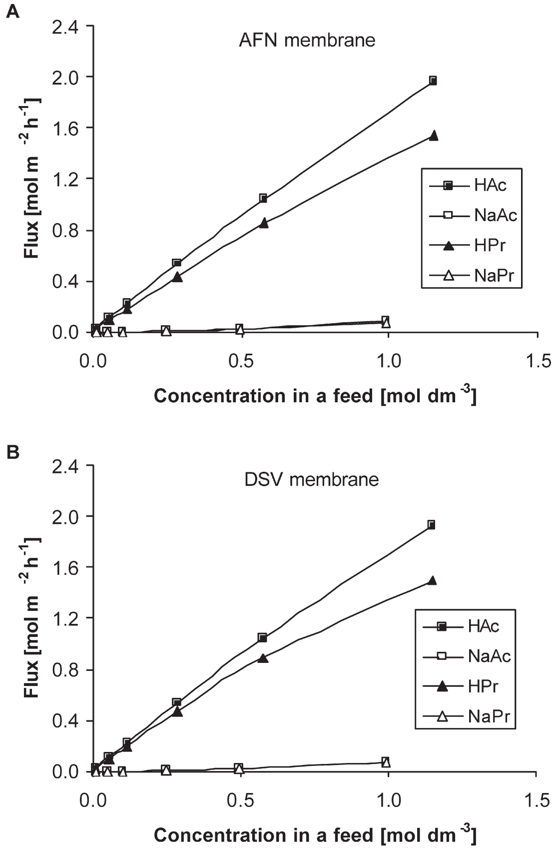

Figure 2. Transport of acetic acid, sodium acetate, propionic acid and sodium propionate across Neosepta AFN-7 (A) and Selemion DSV (B) membranes (single solute experiments).

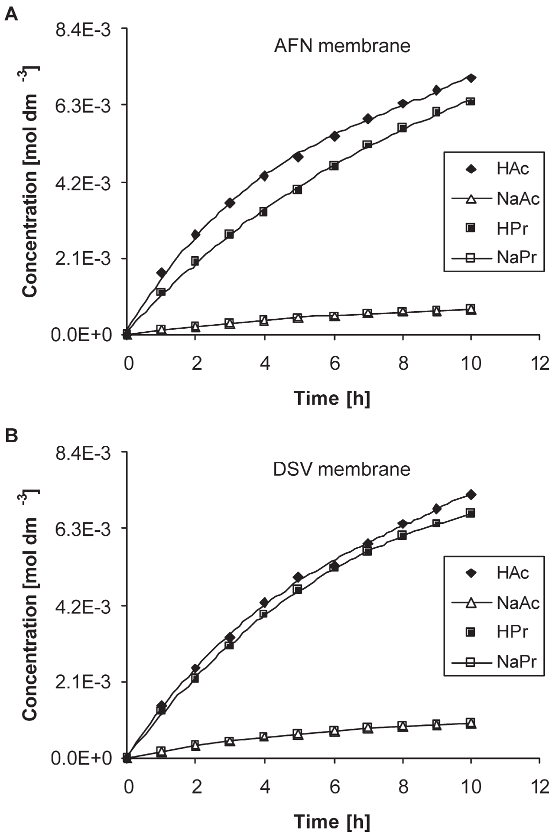

Figure 3. Accumulation of acetic acid, sodium acetate, propionic acid and sodium propionate in the permeate with Neosepta AFN-7 (A) and Selemion DSV (B) (ternary solution experiments: $c_{\mathrm{acid}} = 0.05\ \mathrm{mol \cdot dm^{-3}}$, $c_{\mathrm{carboxylate}} = 0.2\ \mathrm{mol \cdot dm^{-3}}$).

and the separation factors

$$S_{acid/salt} = \frac{U_{acid}}{U_{salt}} \tag{3}$$

were calculated (Table 3).

In the Equations (2) and (3), U is the dialysis coefficient ($\mathrm{m \cdot s^{-1}}$); W - the molar transport rate ($\mathrm{mol \cdot s^{-1}}$); A - the area of a membrane ($\mathrm{m^2}$); Δc - the logarithmic mean concentration difference between the feed and permeate at the beginning and the end of dialysis ($\mathrm{mol \cdot m^{-3}}$).

For both membranes the separation factors are high and amount from 20 to 37. Taking the separation factors for lactic acid/sodium lactate solutions from (17) we got more data to substantiate the final conclusions. Despite

Table 3. Separation factors for carboxylic acid/sodium carboxylates[b]

The acid	p*K*	Neosepta AFN-7	Selemion DSV
Acetic acid	4.76	37.3	28.1
Propionic acid	4.87	33.0	29.2
Lactic acid[a]	3.86	20.1	29.4

[a] taken from our paper (17).

[b] ternary solutions: concentrations of an acid 0.05 mol · dm^{-3}, of sodium carboxylate 0.2 mol · dm^{-3}.

both membranes contain the strong base functional groups, the separation factors differ not only in values but also in the response to the strength of acids (Table 3).

The separation factors for all acids with the Selemion DSV membrane are the same, within the experimental error. On the contrary the Neosepta AFN-7 membrane separate the weak acids: acetic and propionic more effectively than lactic acid.

As a conclusion, the results confirm the diffusion dialysis technique to be applicable for separating the mean strength and weak carboxylic acids from their salts.

CONCLUSIONS

A detailed analysis of the solubilities, permeabilities and separation ability of the diffusion dialysis membranes Neosepta AFN-7 and Selemion DSV for the separation of acetic, propionic and lactic acids (17) from the corresponding sodium salts leads to the following conclusions.

- Both membranes absorb acids in high amount and reject salts. The driving force for the high sorption seems to be the association of the acid with the water molecules incorporated in associates.
- The fluxes of the acids are high ranging 1.5-2.0 mol · $m^{-2} \cdot h^{-1}$ whereas those of the salts amount 0.07–0.08 mol · $m^{-2} \cdot h^{-1}$ only (single solute permeation).
- The separation factors found in the experiments with the ternary solutions and the Neosepta AFN-7 membrane are from 20 for lactic acid up to 37 for acetic acid.
- For the Selemion DSV membrane the separation factor is ~29, as irrespective to the strength of acid.
- The results prove the DD technique to be applicable for the dialytic separation of carboxylic acids from carboxylates.

SYMBOLS

A	area of a membrane (m^2)
c	concentration of an acid/salt in an external solution ($mol \cdot dm^{-3}$)
$\bar{c}$	concentration of an acid/salt within a membrane ($mol \cdot dm^{-3}$)
Δc	difference of concentration on feed and permeate side of a membrane ($mol \cdot m^{-3}$)
J	flux of a solute ($mol \cdot m^{-2} \cdot s^{-1}$)
k	partition coefficient of an acid/salt
l	thickness of a membrane (m)
$\bar{m}$	concentration of an acid/salt in a water swelling the membrane ($mol \cdot kg^{-1}$)
P	permeability coefficient of a solute ($m^2 \cdot s^{-1}$)
S	separation factor of a bicomponent solution systems
U	dialysis coefficient of an acid/salt ($m \cdot s^{-1}$)
V	volume of a solution (m^3)
W	transport rate of an acid/salt ($mol \cdot s^{-1}$)

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