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# SEPARATION OF FERMENTATION PRODUCTS BY MEMBRANE TECHNIQUES. IV. ELECTRODIALYTIC CONVERSION OF CARBOXYLATES TO CARBOXYLIC ACIDS

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## ABSTRACT

For the salt-to-acid conversion of carboxylates by ED technique, the high resistance of weak carboxylic acid solutions limits a current and increases the energy consumption per unit of a product. Carrying experiments for the conversion of sodium lactate to lactic acid ($pK_{lactic} = 3.86$), we proved that the energy consumption can be lowered by filling the acid compartment in ED unit with the hydrogen conducting filler.

In this paper the results of the same procedure applied for the conversion of sodium acetate to acetic acid ($pK_{acetic} = 4.75$) and sodium propionate to propionic acid ($pK_{propionic} = 4.87$) are presented. It has been stated that with a filler in a unit the limiting current densities increase from 0.9 mA/cm$^2$ to 19.5 mA/cm$^2$ for the production of acetic acid and from 0.75 mA/cm$^2$ to 22 mA/cm$^2$ for

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propionic acid. With these current densities, the intensification factor found for these two conversions was within 30–35, and the energy consumption was in a range 0.3–0.5 kWh/mol.

Two phenomena coupled to electrotransport—osmotic dilution and back diffusion of acids—are known to depress the yield of conversion. By experiments performed for acetic, propionic, and lactic acids using three anion exchange membranes Neosepta AM-1, ACM, and AMX (Tokuyama Co., Japan) it has been stated that diffusional leakage of acetic acid is rather high (up to 1 mol/m$^2$h), whereas for lactic acid it is much lower (~0.1 mol/m$^2$h). Of three membranes tested, the Neosepta AMX was the most effective in preventing both effects, thus assisting in getting high-yield conversions.

## INTRODUCTION

Carboxylic acids are extensively used in the food and chemical industries. Acetic acid is not only applied as an acidifier, but also as a chemical reagent for the production of cellulose acetate, vinyl acetate, and esters. Propionic acid is a bacteriostatic agent used for the conservation of cereals. Lactic acid is a monomer for the production of biodegradable polymers and a component of good solvents. All these acids could be produced by the chemical synthesis or by the bacterial fermentation of carbohydrates. The natural fermentation seems to be preferable for the products used for food industry. Despite being known for long time, the fermentation technology is still developing, mainly because diversity of microorganisms in use but also because of the efforts put toward developing the continuous fermentation instead of the contemporary batch technique (1–4).

For high-yield fermentation a selection of microorganisms, nutrients, and organic additives are essential. It has been documented, however, that the fermentation products inhibit the fermentation process. At the concentration of acetic acid as low as 0.6% (v/v) *Acetobacter aceti* stop growing (5). To keep the bacteria active the broth could be neutralized to the optimal level (pH 5–6). Nonetheless, bacteria are not resistant for carboxylates either. With sodium acetate in a broth the growth of *Acetobacter aceti* becomes inhibited at the concentration ~2.4% (w/w). The inhibition by both products has been reported by a number of authors, and it is a common effect for the fermentations. Thus, the continuous removal of fermentation products seems to be the only effective method for keeping bacteria active for long time. Elaboration of the corresponding methods is one of the tasks needed before designing the continuous fermentation technology.

In this respect, a number of membrane techniques were examined. Well described are the systems for the so called "pH controlled fermentation" with a fermenter integrated with modules for ultrafiltration (UF) and electrodialysis (ED) operating consecutively (6–8). In those systems the UF module works for the recycling cells in a loop of a fermenter, whereas ED is for the conversion of carboxylates in a filtrate to carboxylic acid. The system for the fermentation without the pH adjustment of a broth has been designed by Nomura et al. (9, 10). It consists of a fermenter joined directly to an electrodialyzer working for the continuous removal of acid from fermenting broth. In this system, operating with the continuously supplied nutrient, the production of acetic acid proceeded for 30 days instead for 2 days only, with the acid in a fermenter.

In our laboratory the new system for the continuous recovery of acids from broths consisting of a dialysis unit, applied for the extraction of acid, integrated with the periodically operating electrodialysis unit has been worked out. In the preceding papers details of the extraction of lactic acid by the facilitated membrane extraction technique (FME) and the conversion of sodium lactate to the acid (model solution and broth) were presented (11–13). The later done experiments proved the FME technique to be effective for the separation and extraction of acetic and propionic acids either. Both acids are weaker than lactic by one order ($pK_{lactic} = 3.86$, $pK_{acetic} = 4.75$, $pK_{propionic} = 4.87$). The resistance of weak carboxylic acids solutions, which exceeds the resistance of membranes, produces problems. The low concentration of ions in ED compartments limits the current density drastically and strengthens the polarization phenomena. In paper (13) we proved that the limiting current density can be much increased by filling the acid compartment with a conducting turbulence promoter. That way, $I_{lim}$ for the conversion of sodium lactate to lactic acid increased from a few up to 18–20 mA/cm$^2$. In this paper, the same technique has been applied for studying the conversion of sodium acetate to acetic acid and sodium propionate to propionic acid. Accounting for the high resistance of acetic and propionic acid solutions, the efficiency of standard electrodialytic conversion without a filler was found to be very low.

Apart from high resistance and concentration polarization effects, two phenomena are known to limit the yield of conversion. These are the back diffusion and the osmotic dilution of an acid in ED unit. Both effects intervene in getting the high concentration of acids in the final products. These phenomena were examined for three acids: acetic (AA), propionic (PA), and lactic (LA) using three anion exchange membranes differing in ohmic resistance and permeabilities. The results are described.

## EXPERIMENTAL

### Electrodialysis Unit

Experiments were carried out using a small-scale four-compartment electrodialyzer designed in this laboratory (13). Equipped with eight platinum wire electrodes (two terminal + six probe electrodes), it enabled the detail examination of the current/voltage characteristics for the whole unit and for each one of the membrane solution elements. The voltage drops were measured in one passage for the whole unit and for three constituting elements by using four multimeters (Metex M4650, Poland). Current was supplied by a ZT-980-4M power unit (Unitra, Poland). The solutions filling each tank/compartment loop were circulated individually using a MasterFlex pump (USA). The rate of circulation was 100 mL/min. The surface of each membrane was 12.56 $cm^2$. Experiments were carried out at 293 K.

Having these results the limiting current densities for the whole electrodialyzer and for each of constituting elements were determined by Covan method. The method for determining $I_{lim}$ and the polarization effects were discussed (13).

Sodium acetate and sodium propionate 0.25M and 0.5M were applied as model solutions of a feed. The starting molarity of acids in ED compartments ($c_t = 0$) was 0.1. Sodium hydroxide 0.2M and the bicomponent solution of sulfuric acid and sodium sulphate of 0.1 ± 0.05 M (all POCh Poland) were used as the cathode and anode solutions, respectively.

### Determination of the Diffusional Leakage of Acids and Salts

Experiments for diffusional permeation of acids and salts (single solutions) were carried out using an automatic setup developed in our laboratory (11,14,15). The conductivity for each dialysate versus time was stored and processed automatically. The permeability coefficients were calculated on the basis of the Fick's law adopted to the experimental findings. The relation: concentration—conductivity of a solute was predetermined for each compound in the study.

### Osmotic Transport of Water

Accounting for molarities of carboxylates in broths and aiming at getting high molarity acids after conversion the osmotic flow was measured in the systems:

a) 0.25 mol/L sodium carboxylates | anion exchange membrane | 0.75 mol/L carboxylic acid
b) 0.25 mol/L sodium carboxylates | anion exchange membrane | 1.0 mol/L carboxylic acid.

The osmotic volume flow was measured in a standard two-compartment cell equipped with a capillary attached horizontally to the acid solution side (16,17).

Volumes of the compartments were 130 $cm^3$ each, the diameter of a capillary 0.6 mm.

Both the diffusional leakage and osmotic flow were determined with three anion exchange membranes differing in ohmic resistance and permeation.

## Membranes

The cation exchange membrane Neosepta CM-2 and three anion exchange membranes Neosepta AM-1, AMX, and ACM (Tokuyama Corp., Japan) were taken for experiments. The cation exchange membrane was the same as used in Narębska and Kurantowicz (13). All the anion exchange membranes are strongly basic. The AMX membrane is known as mechanically stable, AM-1 is the low-resistance membrane, whereas the ACM is the low $H^+$ transport membrane (producer data and (18)). The membranes differ in ohmic resistance, exchange capacity, water content, and thickness. The characteristics of membranes as determined by standard methods (19) are as follows:

| Membrane | Exchange Capacity [mmol/g] | Water Content [g/g] | Ohmic Resistance [$\Omega$ $cm^2$] | Thickness [cm] |
|---|---|---|---|---|
| AM-1 | 1.74 | 0.24 | 1.22 | 0.012 |
| AMX | 1.40 | 0.19 | 3.12 | 0.016 |
| ACM | 1.44 | 0.18 | 4.05 | 0.011 |

Before being put into ED unit, the cation exchange membrane was equilibrated repeatedly with sulfuric acid solution. The anion exchange membranes were prepared in acetate and propionate forms by treating with sodium acetate and sodium propionate respectively. After washing out with distilled water the membranes were put into the solutions planned for conversions and stored there until taken for experiments.

## Analytics

The concentration of carboxylic acids and sodium carboxylates were determined by capillary electrophoresis (CS Capillary Electrophoresis Analizer EA 100, Villa Labeco, Slovakia) (12). The accuracy in determining acids and salts was ±4%.

To estimate the rate of conversion the concentration of acids produced in the 5 h and the long-time experiments was determined after every 60 and 120 min correspondingly.

## RESULTS AND DISCUSSION

### Limiting Current Density for the Conversion of Sodium Carboxylates to Carboxylic Acids

In large-scale electrodialysis the working current density normally amounts to 75–80% of the limiting current density. The increased temperature, the high fluid velocities, and the spacers promoting turbulent flow make the stagnant layers on membrane surfaces thinner, increasing thus the current. Yet, the nature and resistance of the solutions in compartments are fundamental.

Within the four compartment unit used here one compartment is for the formation of acetic or propionic acid (Fig. 1). The formation of acids proceeds through the migration of carboxylic anions from a feed across the anion exchange membrane. To the same compartment hydrogen ions migrate from the anode solution crossing the cation exchange membrane. Thus, the acid forms in this compartment. The conductivities of acetic and propionic acids of the molarity 0.1 are as low as 0.06 S/m and 0.05 S/m and do not change drastically when the molarity increases. In order to increase conductivity, the weak acid compartments were filled with the suspension of granular beads of the cation exchange resin Amberlite IR 120PLUS in hydrogen form. By composition this resin is a crosslinked strong acid able to conduct current by hydrogen ions. In Table 1 the limiting current densities found for electrodialyzer with the acidic compartments filled with acid only and with the acid and a suspension are collected. The method for establishing limiting current densities, used here, was the same as in our earlier paper (13). The low-current densities for both acids without the conducting filler were confirmed. With the filler in a compartment the limiting current densities were found 19.5 ± 2 $\mathrm{mA/cm^2}$ for the conversion of sodium acetate to acetic acid and 22.0 ± 2 $\mathrm{mA/cm^2}$ for the conversion of sodium propionate to propionic acid. In

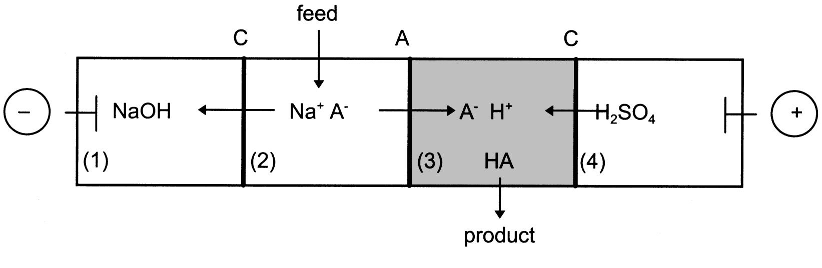

***Figure 1.*** Scheme for the conversion of sodium carboxylate to carboxylic acid in four compartment ED unit.

NaA—sodium carboxylate
HA—carboxylic acid
C, A—cation and anion exchange membrane

***Table 1.*** Limiting Current Densities for the Conversion of Sodium Carboxylates to Carboxylic Acids With and Without the Resin Amberlite IR 120PLUS, $H^+$ (A Conducting Filler)

| Limiting Current Density [mA/cm$^2$] (whole unit)* | Sodium Acetate/Acetic Acid | Sodium Propionate/Propionic Acid | Sodium Lactate/Lactic Acid** |
|---|---|---|---|
| Without the filler | 0.75 ± 0.3 | 0.70 ± 0.3 | 2.5 ± 0.3 |
| With the filler | 19.1 ± 2.0 | 21.5 ± 2.0 | 17.7 ± 2.0 |

* Membrane in the unit (see Fig. 1):
C: Neosepta CM-2
A: Neosepta AM-1
** Taken from (13)

the same table the data for the conversion of sodium lactate to lactic acid taken from Narębska and Kurantowicz (13) are included. Intensification factors, which denote the ratio of the limiting currents with and without the resin in a unit, were found within 30–35. For the conversion of sodium lactate to lactic acid the intensification factor is ~7.

Certainly, the use of a sheet type conducting spacers, made of the same or comparable conducting polymers, would be handy.

### Current Efficiency and Energy Consumption for the Conversion

For short-time conversions (up to 5 h) the production of acids is linear in time (Fig. 2). Having received these data with each of three anion exchange membrane in the unit, the current efficiency ($\eta$) and energy consumption ($\eta_{EC}$) were calculated (Table 2).

$$\eta = \frac{(c_0 - c_t)vF}{It} \cdot 100\% \tag{1}$$

$$\eta_{EC} = \frac{tI\,\Delta V}{m} \tag{2}$$

The symbols denote: $c_0$—concentration of acid at $t = 0$ (mol/L), $c_t$—concentration of acid in time $t$ (mol/L), $I$—current (A), $F$—Faraday constant (mol/C), $v$—volume of a solution (L), $m$—mass of acid (kg), $\Delta V$—potential difference (V), and $t$—time (s).

Despite conversions being carried out at the same molarity of a feed and the limiting current densities being established individually for each acid, the energy consumed per the production of one mole of acetic and propionic acids was found to be higher than for lactic acid.

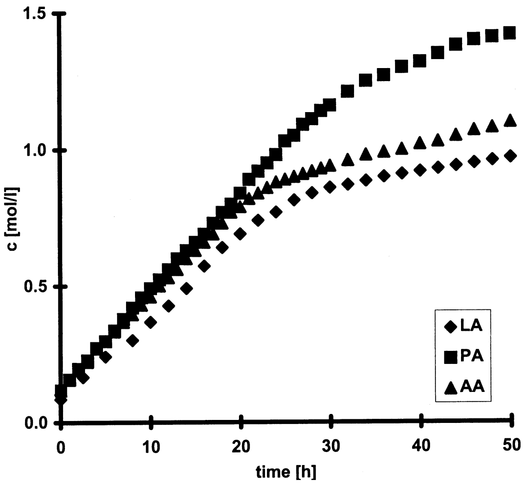

*Figure 2.* Conversion of sodium carboxylates to carboxylic acids with Neosepta AM-1 membrane. Accumulation of acids in time: AA—acetic, PA—propionic and LA—lactic.

*Table 2.* Current Efficiency and Energy Consumption for the Conversion of Sodium Carboxylates to Carboxylic Acids With Different Anion Exchange Membranes (Short-time Experiments)

| Membrane | Limiting Current Density [mA/cm$^2$] | $\Delta c$ [mol/dm$^3$] | Rate of Conversion [mol/m$^2$h] | $\eta$ [%] | $\eta_{EC}$ [kWh/kg] | [kWh/mol] |
|---|---|---|---|---|---|---|
| | | | *Acetic Acid* | | | |
| AM-1 | 19.0 | 0.18 | 7.0 | 99 | 8.1 | 0.49 |
| ACM | 13.0 | 0.12 | 4.8 | 98 | 7.2 | 0.43 |
| AMX | 15.5 | 0.15 | 5.6 | 99 | 6.6 | 0.40 |
| | | | *Propionic Acid* | | | |
| AM-1 | 21.0 | 0.18 | 7.1 | 98 | 6.4 | 0.47 |
| ACM | 13.5 | 0.12 | 4.9 | 98 | 6.6 | 0.49 |
| AMX | 16.0 | 0.15 | 5.9 | 98 | 6.1 | 0.45 |
| | | | *Lactic Acid* | | | |
| AM-1 | 17.5 | 0.16 | 6.5 | 96 | 3.1 | 0.28 |
| ACM | 14.3 | 0.13 | 5.2 | 95 | 3.4 | 0.35 |
| AMX | 22.0 | 0.22 | 8.2 | 97 | 3.0 | 0.27 |

The long-time conversions revealed nonlinearity in formation of acids (Fig. 2). For propionic acid at $c > 1$ mol/L and for acetic acid above 0.7–0.8 mol/L the rate of processes declined in time, the current efficiencies decreased, and the energy consumptions increased drastically (Table 3). Because the concentration of feed was the same in all experiments, the results suggested some additional phenomena deteriorating the process. The back diffusion and osmosis were supposed to affect the yield and rate of the conversions.

## Back Diffusion of Acids

Figure 3 presents the diffusional permeation of acetic, propionic, and lactic acids across three anion exchange membranes tested. Here the diffusional permeation was measured with water on the receiving side (at $t = 0$). With solutions on both membrane side as in the real system, that is, with a membrane separating a feed solution and dialysate (instead of water), the effects should not be as drastic as those displayed here. Yet, the results of Fig. 3 seem to substantiate the differences in $\eta$ and $\eta_{EC}$ (Tables 2 and 3) and should not be neglected in the large-scale electrodialyzers. Irrespective of the anion exchange membrane, acetic acid is the

***Table 3.*** Current Efficiency and Energy Consumption for the Conversion of Sodium Carboxylates to Carboxylic Acids in Short- and Long-Time Experiments (30 to 50 hours)

| Time [h] | $\Delta c$ [mol/dm$^3$] | Rate of Conversion [mol/m$^2$h] | $\eta$ [%] | $\eta_{EC}$ [kWh/kg] | $\eta_{EC}$ [kWh/mol] |
|---|---|---|---|---|---|
| *Acetic Acid* ($i_{lim} = 19$ mA/cm$^2$) | | | | | |
| 5 | 0.18 | 7.0 | 99 | 8.1 | 0.49 |
| 30 | 0.82 | 6.3 | 92 | 9.9 | 0.59 |
| 50 | 0.98 | 4.0 | 85 | 12.6 | 0.76 |
| *Propionic Acid* ($i_{lim} = 21$ mA/cm$^2$) | | | | | |
| 5 | 0.18 | 7.1 | 96 | 6.4 | 0.47 |
| 30 | 1.04 | 7.0 | 91 | 8.7 | 0.64 |
| 50 | 1.30 | 5.6 | 87 | 10.9 | 0.81 |
| *Lactic Acid* ($i_{lim} = 17.5$ mA/cm$^2$) | | | | | |
| 5 | 0.16 | 6.5 | 96 | 3.1 | 0.28 |
| 30 | 0.78 | 5.4 | 83 | 3.9 | 0.35 |
| 50 | 0.90 | 5.2 | 80 | 5.3 | 0.48 |

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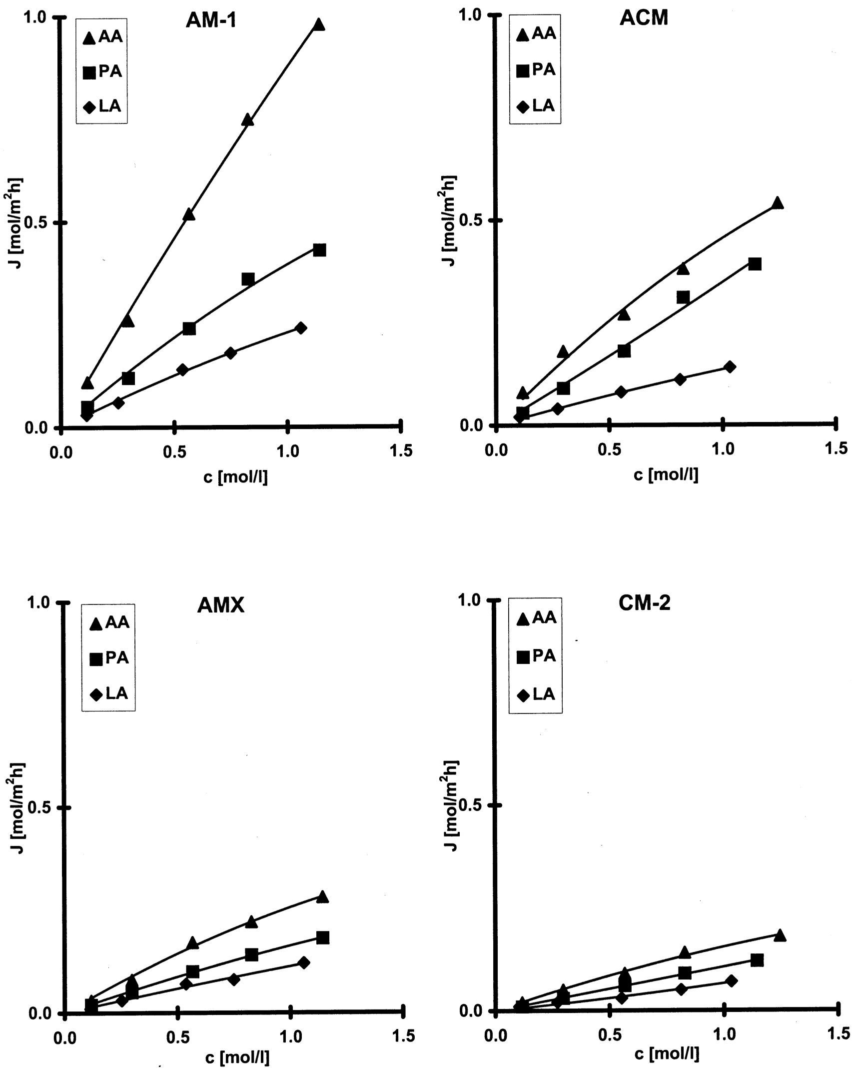

***Figure 3.*** Diffusional permeability of acetic (AA), propionic (PA), and lactic (LA) acids across Neosepta anion exchange membranes AM-1, ACM, and AMX and the cation exchange membrane Neosepta CM-2.

most permeable one whereas the least permeable is lactic acid. The much different permeations of acids demands for some comment.

The strong-base anion exchange membranes are known to exclude the strong electrolytes, both acids and salts. It is different, however, for weak acids.

Examining the permeability of acetic, propionic and lactic acid across the strong base Neosepta AFN-7 membrane (Tokuyama Co., Japan) and the Selemion DSV membrane (Asahi Glass Co., Japan), we have found that these membranes strongly absorb the weak acids (molecular sorption).

For being exact the partition coefficients $k = \bar{c}/c$ ($\bar{c}$, $c$ are concentrations of an acid within a membrane and in an equilibrated solution) are as follows (11).

| | Acids<br>Acetic | <br>Propionic | <br>Lactic |
|---|---|---|---|
| $k$ (dimensionless) | 2.00 | 1.83 | 1.43 |
| $\bar{D} \times 10^{11}$ m$^2$s$^{-1}$ | 7.50 | 4.70 | 4.03 |

$\bar{D}$ denotes apparent diffusion coefficient for the acid in a membrane (Neosepta AFN-7).

We then suppose that the range of diffusional permeations found here, that is $J_{AA} > J_{PA} > J_{PA}$, reflect the same high sorption and different diffusion rates of acids in membranes examined here.

Comparing membranes, it appears that Neosepta AM-1 membrane is the most leaky whereas the mechanically stable Neosepta AMX is fairly resistive against diffusional permeation. The cation exchange membrane Neosepta CM-2 is less-permeable for the acids than the anion exchange membranes.

Looking at the results, the Neosepta AMX membrane turns out to be the most effective for the conversion.

*Table 4.* Osmotic Fluxes across Neosepta Anion Exchange Membranes

| | Flux of Water [mol/m$^2$h] | | |
|---|---|---|---|
| The System* | AM-1 | ACM | AMX |
| Lactic acid** | | | |
| 0.75 mol/l | 3.0 | 2.9 | 2.4 |
| 1.0 mol/l | 6.2 | 5.4 | 4.6 |
| Propionic acid: | | | |
| 0.75 mol/l | 6.3 | 3.9 | 2.2 |
| 1.0 mol/l | 12.1 | 8.3 | 5.2 |
| Acetic acid: | | | |
| 0.75 mol/l | 5.6 | 3.1 | 2.0 |
| 1.0 mol/l | 10.8 | 5.9 | 4.3 |

| * I | | II |
|---|---|---|
| Sodium Carboxylate<br>c = 0.25 mol/l | Anion Exchange<br>Membrane | Carboxylic Acid<br>c = 0.75 mol/l (or c = 1.0 mol/l) |

** Concentration of acid in II.

### Osmotic Dilution of Acids

Osmotic fluxes of water toward solutions of acetic, propionic and lactic acids are collected in Table 4. Conclusions are very much alike as for the back diffusion. The fluxes of water differ and the Neosepta AMX membrane appears the most resistive against the osmotic permeation.

One can expect the osmotic fluxes in an electrodialyzer to be lower. It appears, however, that because of osmotic dilution it will be rather difficult to produce acids of high concentrations or the process will consume more energy.

## CONCLUSIONS

Experiments performed for the conversions of sodium acetate and sodium propionate and our early results for the conversion of sodium lactate to corresponding acids let us draw the following conclusions.

The conversion of carboxylates to acids by electrodialysis can be made effective by incorporating into the acid compartments the conducting turbulence promoter (e.g., cation exchange resin Amberlite IR 120PLUS, $H^+$, or a sheet spacer made of conducting polymer). With the conducting filler the limiting current density, normally low: 0.7–3 mA/cm$^2$, increases up to 18–22 mA/cm$^2$, making the technique effective.

With these current densities the current efficiencies are high for all acids examined. The energy consumption for the conversion of lactate to lactic acid is in a range of 0.27–0.35 kWh/mol whereas for the other acids it is 0.43–0.49 kWh/mol.

The osmotic dilution and the back diffusion of acids (mainly across anion exchange membrane) were found to depress the efficiency of conversion. The experiments done for establishing diffusional permeation of acids indicated the rather strong leakage of acetic acid. For propionic acid the back diffusion is lower and the lowest is for lactic acid.

Of three Neosepta anion exchange membranes examined—AM-1, ACM, and AMX—the one diminishing these effects effectively is AMX membrane.

Due to osmotic dilution the economically reasonable concentration of the produced acids was found within the limit ~0.7–1.0 mol/L. At higher acid concentration a consumption of energy would increase.

## ACKNOWLEDGMENT

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