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Lactic acid production by electrodialysis
Part I: Experimental tests

N. BONIARDI, R. ROTA*, G. NANO, B. MAZZA

Dipartimento di Chimica Fisica Applicata, Politecnico di Milano, via Mancinelli 7, 20131 Milano, Italy

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Lactic acid, the production of which from cheap raw materials is attractive due to its great potential in the production of biodegradable polymers, can be obtained from fermentation broths using electrodialysis. This work discusses the feasibility of this process using commercial membranes, together with the influence of several operating parameters on the electrodialysis stack performance. It was found that it is possible to operate with high current efficiency values, while the overall recovery of sodium lactate depends on the current density. Electroosmotic water transport limits the maximum concentration value achievable using this technique.

1. Introduction

For almost a quarter of a century electrodialysis was considered as a desalination process to be applied to such solutions as brackish water and sea-water. The wider scope of this technology was only gradually recognized in the recent past, leading to several applications that have considerable economic significance [1–3]. In particular, electrodialysis can be used as an alternative to the usual end-of-pipe treatment processes to recover valuable products from several industrial effluents [1, 4–6]. For instance, the waste water produced in large amount (about 130 million tons in 1987) during cheese-making, can be fermented to produce lactic acid which is usually recovered from the fermentation broth as sodium salt [7, 8]. Interest in producing lactic acid is growing due to its great potential in the manufacture of biodegradable packaging and films [9]. However, the main drawback is that fermentation derived sodium lactate is difficult and expensive to purify [10]. Among the other processes, electrodialysis is promising for the downstream processing of lactic acid from fermentation broth [10–14].

The main goal of this work was to investigate a single-step isolation and purification process of free lactic acid from sodium lactate solutions through electrodialysis. The feasibility of the use of a four-compartment cell stack equipped with commercial membranes for the downstream processing of lactic acid was tested using a laboratory scale apparatus with standard solutions. In particular, the main phenomena which affect the process were investigated, and the influence of several operating parameters on the overall performance of the electrodialysis stack was discussed.

2. Materials and methods

The experimental equipment used is a laboratory-scale four-compartment electrodialysis stack (Eurodia TS-EUR2-5 D). The specification of the main parts constituting such a stack is given in Table 1 [15].

The process involves a double decomposition reaction, in which ion combination of two types of salt, provided by sodium lactate and hydrochloric acid solutions, produces sodium chloride and lactic acid (see Fig.1). The double decomposition reaction was achieved in a four-compartment electrodialyser. Each compartment was alternately separated by cation- and anion-selective membranes. The path of the solutions through the cells was regulated by spacer gaskets which ensure a sheet flow (i.e., a flow parallel to the membranes) distribution.

The electrodialysis unit was provided with ten anion-exchange commercial membranes (Neosepta AMX, Tokuyama Soda) and twelve cation-exchange membranes (Neosepta CMX, Tokuyama Soda), arranged to give five cells. Manufacturer's data on membrane properties are shown in Table 2 [15]. Stack cells were placed between two electrode chambers. The anolyte compartment included a platinum-plated titanium anode, while the cathode was stainless steel.

The general process flow sheet is shown in Fig. 2. It involved five independent streams: the electrode solution was supplied by the same stream line to both electrode chambers, while the remaining four distribution circuits were connected to the solution compartments. Starting from the holding tank, each solution passed through a pump, a rotameter, the electrodialysis stack, and then returned to the tank until the required percentage of lactate recovery was achieved (batch recirculation operation mode).

Cylindrical holding tanks (15 cm diameter) were graduated to determine the volume variations of the solutions; the scale resolution (1 mm) permitted

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* Author to whom all correspondence should be addressed.

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Table 1. List of the main components of the electrodialysis stack

Stack assembly	
Number of cells	5
Number of compartments	4
Compartments	
Membrane overall size	0.137×0.297 m^2
Membrane useful size	0.1×0.2 m^2
Total membrane area (22 membranes)	0.44 m^2
Spacers	
Solution path	sheet flow
Material	polyethylene resin (net)
Thickness	0.75 mm
Electrode chambers	
Anode	platinum plated titanium
Cathode	AISI 316 Type stainless steel
Electrode chamber solution	0.5 M NaCl
Electrode chamber material	polyvinyl chloride resin
Press plate	carbon steel

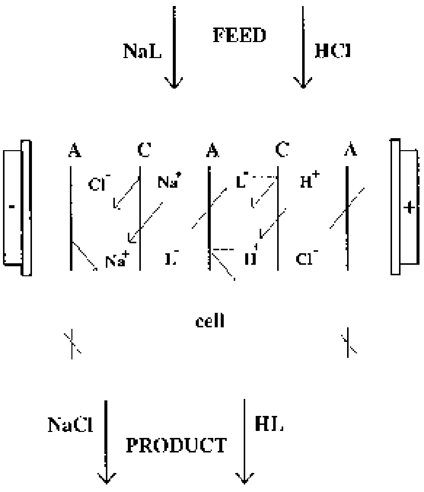

Fig. 1. Sketch of a four-compartment electrodialysis cell for recovering lactic acid (HL) from sodium lactate (NaL) solutions by a double decomposition reaction. A and C indicate anion- and cation-selective membranes, respectively.

a ±10 cm^3 accuracy. Each tank was thermostated at 21 ± 1 °C by means of cooling coils. Feed solution flow rate for each compartment was 0.3 dm^3 min^{-1}.

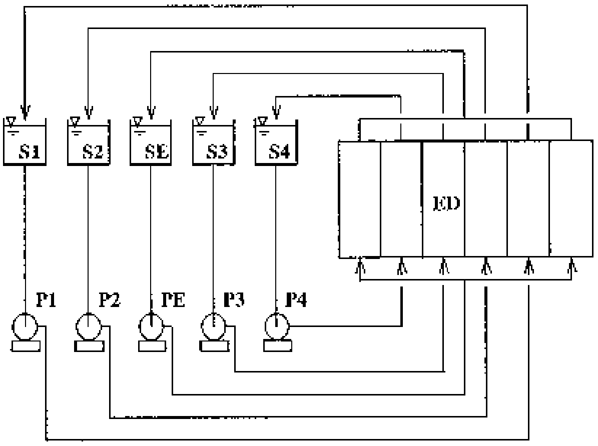

Fig. 2. Sketch of the batch recirculation operation mode for a four-compartment cell: ED indicates the electrodialysis stack, S1 to S4 the reservoirs and P1 to P4 the pumps. Electrode stream lines are also represented: SE indicates the tank of the electrode solution and PE the pump.

Electric power was supplied to the electrodialysis stack by a power unit (Nemic Lambda PWS 350-35). This instrument independently set either the voltage in the range 0-35 V, or the current in the range 0-10 A. All tests were performed supplying a constant current during the run.

After a few tests, membranes were cleaned-in-place by recirculating for 20 min an acid solution (0.05 M HCl), followed by an alkaline one (0.05 M NaOH), and then acid solution again. Finally, process lines were flushed with demineralized water for 10 min.

Solutions of lactic acid, sodium lactate, sodium chloride, and hydrochloric acid were used. They were prepared by diluting the reactants (analytical grade, purity > 99%) with distilled water until the required concentration was reached. Different values of initial concentration of sodium lactate and lactic acid were used, while the initial concentration values of hydrochloric acid and sodium chloride solutions were set at 9 and 25 g dm^{-3}, respectively, for all the runs. Finally, an aqueous solution of sodium chloride (0.5 M) was used to feed the electrode compartments.

The analysis of hydrochloric acid was performed by titration with sodium hydroxide solution. Sodium

Table 2. Manufacturer's data on membrane properties

Membrane	*Neosepta CMX*	*Neosepta AMX*
Type	high mechanical stability	high mechanical stability
Material	styrene-divinyl benzene copolymer	styrene-divinyl benzene copolymer
Reinforce	polyvinyl chloride	polyvinyl chloride
Thickness	0.17–0.19 mm	0.16–0.18 mm
Temperature range allowance	0–40 °C	0–40 °C
pH range allowance	0–10	0–10
Burst strength	500–600 kPa	450–550 kPa
Ion-exchange capacity	1.5–1.8 meq g^{-1}	1.4–1.7 meq g^{-1}
Electric resistance (0.5 M NaCl)	2.5–3.5 Ω cm^2	2.5–3.5 Ω cm^2
Water transport (10 g dm^{-3} NaCl)	~6 mol F^{-1}	~6 mol F^{-1}

chloride determination was carried out by a Mohr titration procedure [16].

Lactic acid and sodium lactate concentrations were measured by gas-chromatography. The GC apparatus (Hewlett-Packard 5890) was equipped with a flame ionization detector and a Carbopack B-DA-4% Carbowax 20M (2 m × 2 mm i.d.) glass column (Supelco). The analyses were run isothermally at 175 °C, with inlet and detector chambers at 200 °C. The carrier gas (helium) flow rate was set at 9.6 $\mathrm{cm^3\ min^{-1}}$. Concentration values were computed by comparison to standards: 1 $\mathrm{mm^3}$ of standard solutions of lactic acid (0.9091 $\mathrm{g\ dm^{-3}}$) and sodium lactate (1.244 $\mathrm{g\ dm^{-3}}$) were used. Moreover, a solution of oxalic acid (0.03 M) was used to condition the column, and was also added for proper analysis in all lactic acid standards and samples. Sodium lactate solutions were prepared by adding an oxalic acid solution until the conversion of the lactate salt into lactic acid was almost complete.

3. Results and discussion

The influence of the initial concentration of the lactic acid and sodium lactate in the feed solutions, as well as that of the applied current density on the process performances was assessed according to the experimental plan summarized in Table 3. The main reasons for this plan are that the final concentration of lactic acid is very important for use in many applications [13, 17, 18], while the initial concentration of sodium lactate may affect the performance of the electrodialysis apparatus, where lactic acid solution is produced [10]. Moreover, the current density is expected to be the most important parameter affecting the energy-related performance indices of the process. The values of all these variables were selected close to those usually found in real situations [10, 12, 19]. The influence of the initial concentration of sodium lactate was investigated in three runs (4, 6, and 7) performed at similar lactic acid concentration, while that of the lactic acid was studied in four tests (1, 2, 3, and 4) with the same sodium lactate concentration. The influence of current density was evaluated by comparing runs 5 and 8, carried out at 110 $\mathrm{A\ m^{-2}}$, with runs 4 and 7, performed using similar solutions but with a current density of 300 $\mathrm{A\ m^{-2}}$.

Table 3. Scheme of the experimental runs[*]

$[NaL]$/ $\mathrm{g\ dm^{-3}}$	$[HL]$/$\mathrm{g\ dm^{-3}}$			
	400	*150*	*80*	*50*
30			7 8	
45	1	3	4 4R 5	2
125			6	

[*] All the runs were carried out at a current density of 300 $\mathrm{A\ m^{-2}}$, apart from runs 5 and 8 for which the current density was 110 $\mathrm{A\ m^{-2}}$. Initial concentration values are approximate: see Table 5 for the exact values.

All tests were carried out until stack voltage values exceeding 35 V (i.e., the maximum value supplied by the power unit) were reached to sustain the given current value. Stack voltage increased during the run due to the decrease in the concentration of sodium lactate. The amount of hydrochloric acid was always larger than the stoichiometric value, except for run 6, performed at higher sodium lactate concentration. In this case, the dilution in the hydrochloric acid stream caused the stack voltage to rise.

One run was repeated to determine the experimental uncertainty (see runs 4 and 4R in Table 3). The variations of the number of mols in the lactic acid tank and the stack voltage values, reported in Fig. 3 as a function of run time, show the good reproducibility of the operating procedure. This conclusion is also supported by the overall mass balances: the mol variations of each solute, determined on the basis of independent analytical measures in the four different solutions, are similar,

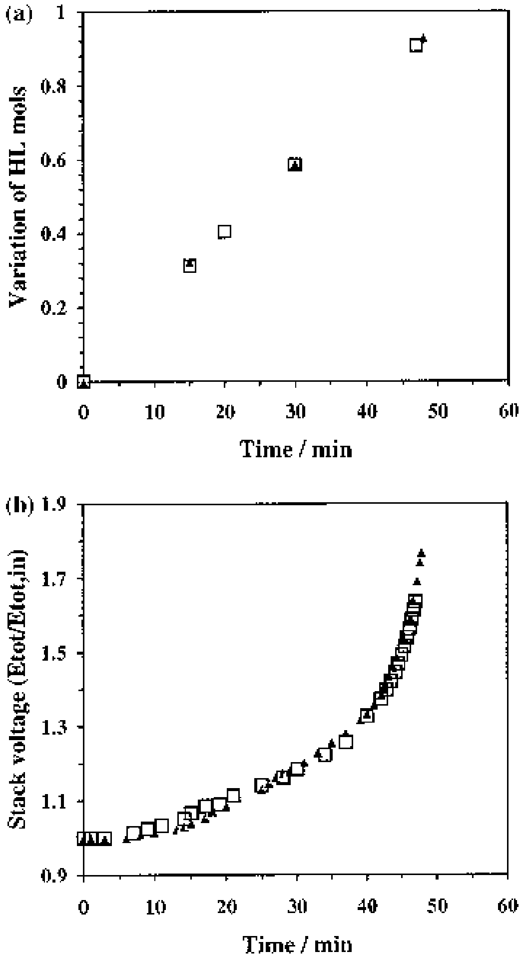

Fig. 3. Comparison between experimental runs 4 (▲) and 4R(□). (a) Variation of the number of mols in the lactic acid tank as a function of time. (b) Overall stack voltage (made dimensionless dividing by the initial value) against time.

giving a maximum average percentage error of less than 5%.

Experimental results are reported in Tables 4 and 5 in terms of volume and concentration of the solutions in each reservoir, and overall stack voltage. Figures 4 and 5 show the variation of lactic acid mols versus time for the runs performed both at similar sodium lactate or lactic acid initial concentration. It can be seen from both figures that there is no influence of the initial concentration of sodium lactate and lactic acid on the rate of the lactic acid recovery. However, higher initial concentration values of sodium lactate may cause longer run time. The effect of the current density is presented in Fig. 6, where the runs performed at 300 A m^{-2} are compared, in terms of variation of number of lactic acid mols versus time, with the tests carried out at lower current density (110 A m^{-2}). As expected, lower current density values correspond to longer run times.

The permeation through the membranes of the ionic species under the influence of an electric field takes place along with the movement of water mo-

Table 4. Tank volume and overall stack voltage values measured in all the tests

Run	*Time* /min	*HCl volume* /dm^3	*NaCl volume* /dm^3	*NaL volume* /dm^3	*HL volume* /dm^3	*Voltage* /V
1	0	5.18	2.59	2.81	2.55	20.0
	15	5.14	2.64	2.73	2.63	20.4
	30	5.11	2.69	2.66	2.70	23.0
	45	5.05	2.73	2.56	2.75	29.0
	48	5.03	2.76	2.53	2.78	35.0
2	0	5.12	2.55	2.61	2.70	21.1
	15	5.10	2.61	2.49	2.75	22.5
	20	5.09	2.64	2.48	2.78	23.2
	30	5.05	2.66	2.42	2.82	25.0
	48	5.02	2.72	2.34	2.88	35.0
3	0	4.89	2.62	2.71	2.63	19.0
	15	4.85	2.70	2.62	2.69	19.5
	30	4.81	2.74	2.56	2.75	22.0
	40	–	–	2.47	2.77	25.0
	51	4.75	2.81	2.42	2.82	35.0
4	0	5.10	2.59	2.58	2.62	19.5
	15	5.05	2.63	2.49	2.68	19.5
	20	5.02	2.67	2.47	2.70	19.8
	30	5.00	2.68	2.39	2.74	21.5
	47	4.95	2.76	2.31	2.81	35.0
4R	0	5.43	2.58	2.70	2.67	19.3
	15	5.39	2.67	2.63	2.78	20.3
	30	5.37	2.71	2.54	2.82	23.0
	48	5.32	2.77	2.45	2.89	35.0
5	0	5.12	2.57	2.77	2.78	8.4
	30	5.10	2.62	2.70	2.84	8.5
	60	5.08	2.67	2.64	2.88	9.1
	90	5.04	2.70	2.55	2.92	9.7
	120	5.01	2.74	2.50	2.95	12.7
	149	5.00	2.77	2.42	2.99	35.0
6	0	5.20	2.55	2.73	2.66	17.6
	15	5.16	2.60	2.66	2.75	17.8
	20	5.15	2.63	2.63	2.77	17.8
	30	5.13	2.65	2.59	2.81	17.8
	58	5.05	2.74	2.44	2.92	35.0
7	0	5.18	2.63	2.54	2.64	18.0
	15	5.15	2.68	2.46	2.72	20.0
	20	5.15	2.72	2.42	2.73	22.0
	32	5.11	2.74	2.38	2.79	35.0
8	0	5.17	2.62	2.71	2.63	7.0
	20	5.16	2.66	2.67	2.67	7.0
	40	5.13	2.68	2.62	2.69	7.0
	60	5.11	2.71	2.57	2.71	7.6
	80	5.08	2.73	2.54	2.75	8.4
	118	5.04	2.79	2.45	2.80	35.0

Table 5. Tank concentration values measured in all the tests

Run	*Time* /min	*[HCl]* /g dm^{-3}	*[NaCl]* /g dm^{-3}	*[NaL]* /g dm^{-3}	*[HL]* g dm^{-3}
1	0	9.3	27.3	45.4	411.9
	30	4.9	40.8	19.0	–
	48	2.5	45.7	5.9	409.3
2	0	9.1	30.0	47.7	47.1
	15	6.9	36.6	–	–
	20	–	–	27.8	59.6
	30	4.9	42.4	–	–
	48	2.5	49.0	7.6	73.6
3	0	9.5	22.4	45.9	152.9
	15	7.2	28.3	–	–
	30	4.5	35.2	–	–
	40	–	–	14.2	171.4
	51	1.9	41.9	6.2	176.2
4	0	8.8	22.6	44.6	78.1
	15	6.6	29.3	–	–
	20	–	–	27.9	89.6
	30	4.5	34.3	–	–
	47	2.4	40.3	5.2	103.7
4R	0	8.1	22.8	46.1	79.1
	15	5.9	29.2	–	–
	30	4.2	34.6	–	–
	48	1.8	40.4	7.3	102.9
5	0	8.6	23.8	45.0	76.0
	30	6.8	27.3	–	–
	60	5.1	32.1	24.2	89.7
	90	3.8	34.3	–	–
	120	2.4	38.2	10.1	98.9
	149	1.2	44.9	1.8	102.2
6	0	7.8	26.2	126.7	85.7
	15	6.1	31.0	–	–
	20	–	–	116.6	–
	30	3.9	37.4	–	–
	58	0.6	46.4	65.4	108.8
7	0	9.1	22.3	30.4	77.3
	15	7.2	28.1	–	–
	20	–	–	13.6	87.8
	32	5.0	34.3	4.6	93.6
8	0	9.4	24.5	36.1	85.4
	20	8.5	27.9	–	–
	40	7.4	30.6	–	–
	60	–	–	18.7	98.6
	80	5.2	35.5	–	–
	118	3.4	40.7	1.1	108.0

lecules associated with the ions. This phenomenon is called electroosmosis and generates an increase in the volume of the solutions circulating in concentrated streams, and a simultaneous decrease in that of the diluted streams. The extent of this phenomenon is shown in Fig. 7 for all the process streams, in terms of tank volume variations versus the electric charge passed through the stack, that is, the product of time by current. It is evident that the water transport is linearly dependent on the amount of electric charge. This confirms that water transport through the membranes is mainly due to electroosmosis. Moreover, it should be noted that the initial concentration of the solutions has no influence on the water flow rate. The slopes of the straight lines in Fig. 7, obtained from linear regression on the four sets of experimental data, represent the average water transport rates and are equal to -3.1×10^{-6} dm^3 C^{-1} for sodium lactate, 2.3×10^{-6} dm^3 C^{-1} for lactic acid, 2.0×10^{-6} dm^3 C^{-1} for sodium chloride, and -1.4×10^{-6} dm^3 C^{-1} for hydrochloric acid. As expected, considering the water transport number for all the solutions, their algebrical sum is approximately equal to zero. This is an indirect confirmation of the reliability of the experimental procedure used.

Representing all the experimental results in terms of experimental mols variation versus the number of theoretical mols required for the process, it is noted

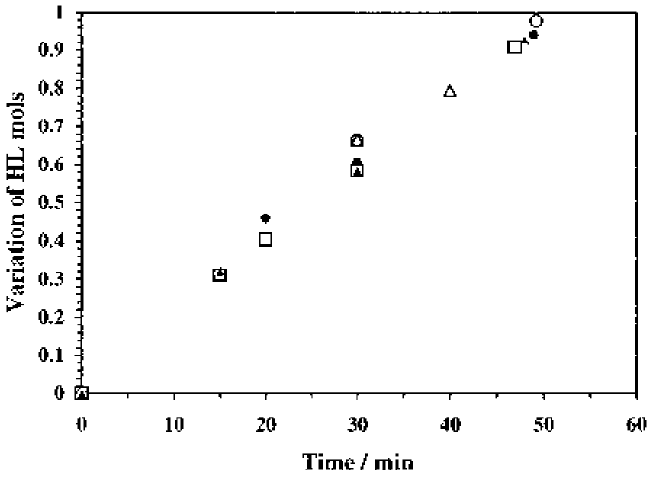

Fig. 4. Variation of mol number of lactic acid as a function of time for the runs carried out at the same initial sodium lactate concentration and current density value. Runs: (○)1, (●) 2, (△) 3, (▲) 4 and (□) 4R.

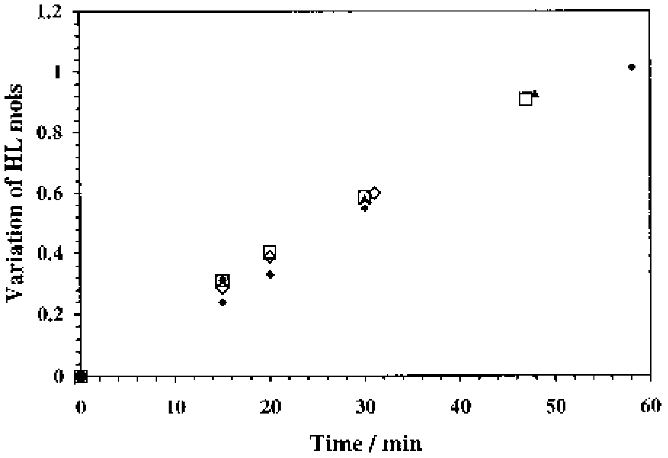

Fig. 5. Variation of mol number of lactic acid as a function of time for the runs carried out at the same initial lactic acid concentration and current density value. Runs: (▲) 4, (□) 4R, (◆) 6 and (◇) 7.

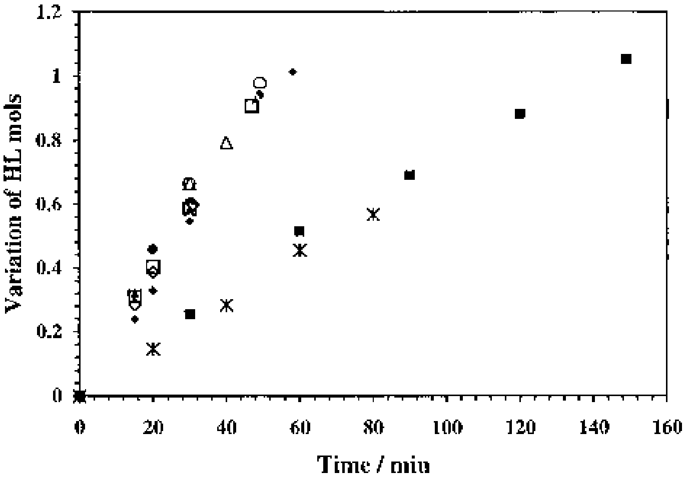

Fig. 6. Variation of mol number of lactic acid as a function of time for the runs performed at different current density values (300 A m^{-2} and 110 A m^{-2}). Runs: (○) 1, (●) 2, (△) 3, (▲) 4, (□) 4R, (■) 5, (◆) 6, (◇) 7 and (✳) 8.

that they lay around a straight line with slope of 45 °, as shown in Fig. 8. This indicates ideal membrane behaviour. To confirm this two additional tests were carried out in order to evaluate both the solute and the solvent permeation due to the concentration gradient across the membranes. In the first run, cells were fed with solutions of concentration equal to the typical values found at the end of each of the previous runs, while the second run was carried out using solutions of initial concentration similar to the average

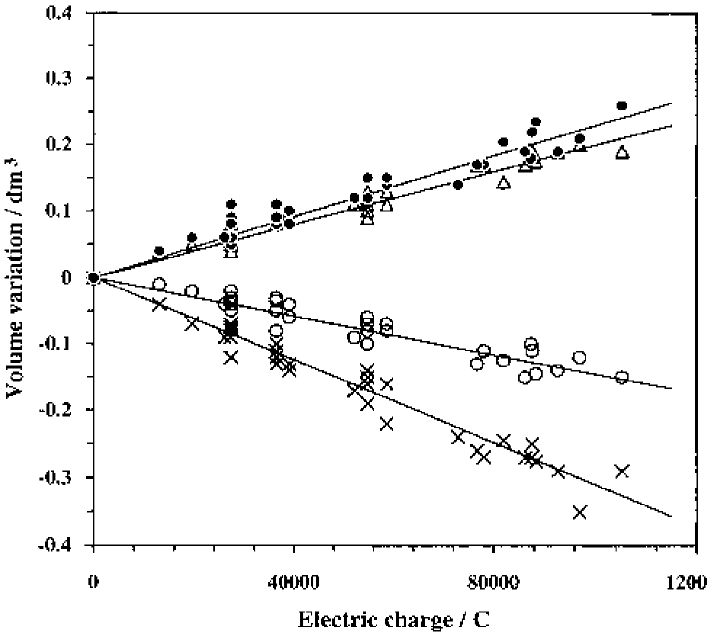

Fig. 7. Experimental volume variations (symbols) as a function of electric charge and linear regression straight lines for the four reservoirs. Key: (△) NaCl, (×) NaL, (○) HCl and (●) HL.

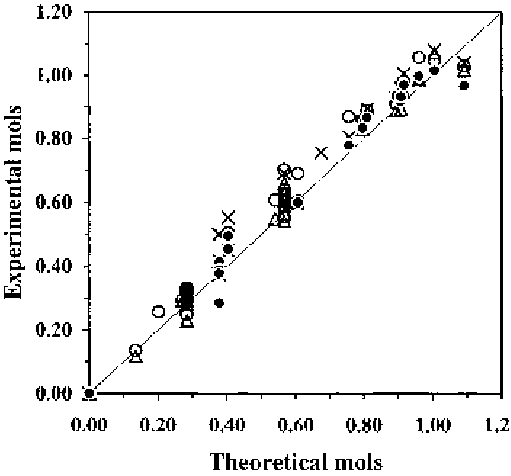

Fig. 8. Experimental against theoretical mols for all the tests. Key: (△) NaCl, (×) NaL, (○) HCl and (●) HL.

initial values recorded in the previous tests. Solutions were circulated in the apparatus for 180 min, without turning on the power. The results clearly show that both the concentration and volume variations are within the range of the experimental uncertainty. Therefore, solute and solvent permeation phenomena promoted by concentration gradients rising between adjacent compartments containing different solutions are negligible, in comparison with transport due to the electric field.

The process performances of the electrodialysis stack were analysed by comparing all the experimental results in terms of four overall process indices, namely:
sodium lactate recovery

$$\Gamma = \frac{\left(m_{\mathrm{NaL,in}} - m_{\mathrm{NaL,fin}}\right)}{m_{\mathrm{NaL,in}}} \times 100 \qquad (1)$$

current efficiency

$$\Theta = \frac{\Delta \mathrm{mol}_{\mathrm{exp}}}{\Delta \mathrm{mol}_{\mathrm{th}}} = \frac{\left(\frac{m_{\mathrm{HL,fin}} - m_{\mathrm{HL,in}}}{MW}\right)}{\frac{(In_c t_{\mathrm{fin}})}{F}} \times 100 \qquad (2)$$

specific energy requirement

$$\Omega = \frac{\int_0^{t_{\mathrm{fin}}} (IE_{\mathrm{tot}})\mathrm{d}t}{m_{\mathrm{HL,fin}} - m_{\mathrm{HL,in}}} \qquad (3)$$

average power requirement

$$\Psi = \frac{\int_0^{t_{\mathrm{fin}}} (IE_{\mathrm{tot}})\mathrm{d}t}{t_{\mathrm{fin}}} \qquad (4)$$

Here $m_{\mathrm{NaL,in}}$ and $m_{\mathrm{NaL,fin}}$ represent, respectively, theinitial and final amount of sodium lactate, $m_{\mathrm{HL,in}}$ and $m_{\mathrm{HL,fin}}$ the initial and final amount of lactic acid in their own reservoirs, $\Delta\mathrm{mol}_{\mathrm{exp}}$ and $\Delta\mathrm{mol}_{\mathrm{th}}$ the experimental and theoretical variation of mols in the lactic acid stream, MW the molecular weight, t_{fin} the run duration time, I the current, E_{tot} the stack voltage, F the Faraday constant, and n_c the number of cells.

From Figs 9 to 11 it is noted that it is always possible to recover more than 85% of the sodium lactate; for the runs performed at lower current densities this index rises to about 95%. The only exception is run 6, where the low value of lactate recovery may be ascribed to the amount of hydrochloric acid initially present in the system; this was less than the stoichiometric value. In this case the recovery of sodium lactate was limited by the amount of hydrochloric acid available for the double decomposition reaction. Current efficiency is near 100% in all the tests. This indicates the good permselectivity of the membranes and confirms their ideal behaviour. Higher current density values correspond to an in-

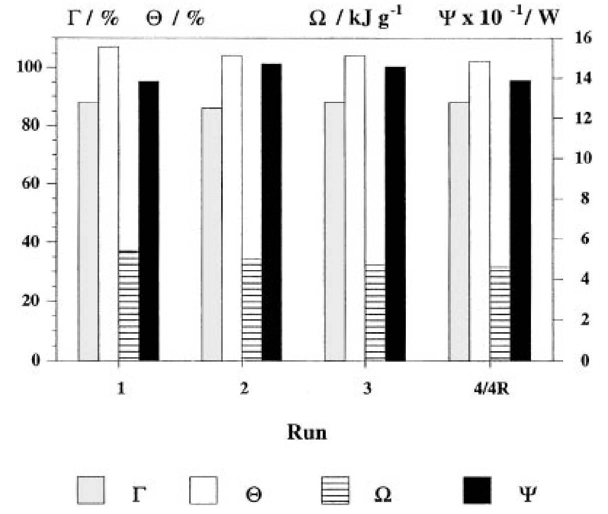

Fig. 9. Process performance indices for runs carried out at the same initial sodium lactate concentration and current density value.

crease in specific energy and power requirements. The initial concentrations of both lactic acid and sodium lactate solutions do not influence any performance index. Thus, a preliminary concentration process by a classical two-compartment electrodialysis stack would not be advantageous if the lactic acid recovery step is performed by electrodialysis.

Finally, the possibility of concentrating lactic acid solutions up to a value suitable for polymer production was also investigated. Typical specialty and commodity chemical uses of lactic acid require about 50% by weight solutions, while polymer manufacture requires a concentration of approximately 90% by weight [17, 18]. Unfortunately, the electric conductivity of a 90 wt % lactic acid solution is too low to permit the enrichment of the solution via electrodialysis. Reasonable conductivity values were found for concentration values of about 400 g dm^{-3}, which is the initial concentration of lactic acid solution involved in run 1. However, the results reported in Table 5 for this run show that it is not possible to concentrate the solution over 40 wt %: despite almost all the sodium lactate being recovered, the concentration of the lactic acid stream did not rise. This means that water transport due to electroosmosis limits the degree of lactic acid concentration achievable. Assuming no water transport through the membranes, the final lactic acid concentration in run 1 should be about 45 wt %.

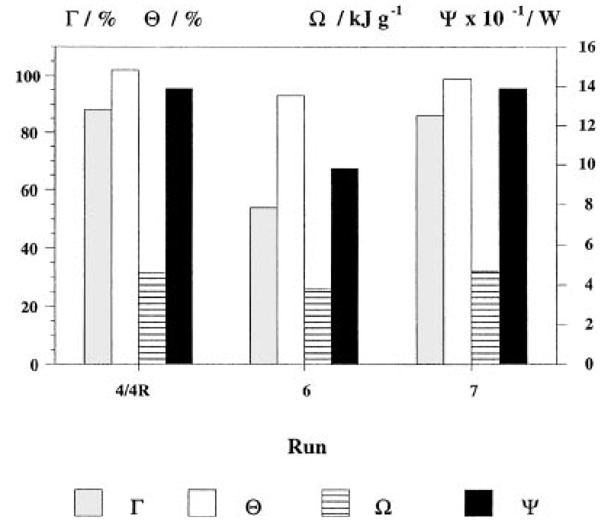

Fig. 10. Process performance indices for runs carried out at the same initial lactic acid concentration and current density value.

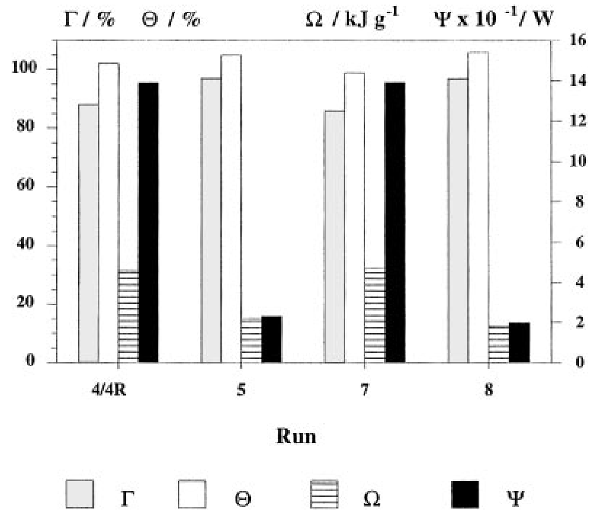

Fig. 11. Process performance indices for runs 4/4R, 5, 7 and 8 carried out in similar conditions but with different current density values (300 A m^{-2} and 110 A m^{-2}).

4. Conclusions

The technical feasibility of an efficient separation and concentration process of lactic acid from sodium lactate solutions, using a four-compartment electrodialysis stack, was investigated. In particular, this work has shown that:

(i) Commercial anion and cation-exchange membranes (Neosepta AMX and CMX, Tokuyama Soda) are able to perform near-ideally, resulting in current efficiency values close to one.
(ii) For a system operating in the batch recirculation operation mode, different initial concentrations of sodium lactate and lactic acid do not affect the performances of the electrodialysis process. This applies for lactic acid concentration values up to about 400 g dm^{-3}.
(iii) Increasing the current density reduces the process duration time without significant changes in current efficiency. The percentage of sodium lactate recovery drops slightly from about 95% to 85%, operating at 300 A m^{-2} instead of 110 A m^{-2}.
(iv) Energy-related performance indices (Ω and Ψ) do not depend on the initial concentration of the solutions, but rise dramatically with current density. However, this also results in a lower process time, thus requiring an economic analysis to find the best operating conditions.

(v) Water transport limits the maximum lactic acid concentration achievable; this can be calculated from the ratio of the flux through the membranes of lactate ions to that of the water.

In conclusion, electrodialysis appears to have a good potential in the downstream processing of lactic acid production. However, the main drawback is represented by the degree of concentration of lactic acid solution. A possible alternative to reduce the water transport may involve highly cross-linked membranes. These restrict swelling to produce low water transport, but greatly increase membrane electric resistance.

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