Application of Electrodialysis for Lactic Acid Recovery

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Abstract

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The paper deals with the possibility of using two-stage electrodialysis for recovery of lactic acid from model solutions and from fermentation broth. In the first step lactate was concentrated with desalting electrodialysis using ion exchange membranes Ralex (Mega, Czech Republic). The highest final concentration of 111 g/l was reached in the concentrate, it means an increase more than 2.5-times in comparison with the initial concentration. At the most 2 g of lactate per litre remained in the feed. The second step was the electroconversion of sodium lactate to lactic acid by water-splitting electrodialysis with the bipolar membranes Neosepta (Tokuyama Corp., Japan). The final lactic acid concentration of 157 g/l was reached in the diluate. Total required energy in both electrodialysis processes consisting of the energy consumption for lactate transfer and for its electroconversion to lactic acid was 142 Wh/mol. The fermentation broth was decolourised before electrodialysis experiments. The best decolourisation capacity was shown by granulated active charcoal filled in the column operated by a slow flow of broth.

Keywords: lactic acid recovery; electrodialysis; desalting electrodialysis; water-splitting electrodialysis; decolourisation

Lactic acid is one of the organic acids having wide use in many fields, e.g. food industry, beverage industry, pharmaceutical industry, chemical industry, medicine (VICK ROY 1985). Today, two thirds of the world production of lactic acid are manufactured by the fermentation method (KAŠČÁK *et al.*1996). The conventional fermentation process produces calcium lactate precipitate, which must be concentrated by evaporation and reacidified with strong acid (BUCHTA *et al.* 1983). The disadvantages of the conventional fermentation process are low reaction rate, elaborate product recovery, large amount of by-products and thereby negative impacts on the environment. There are other possibilities for lactic acid recovery, but solvent extraction, direct distillation, adsorption and other relatively simple methods have some limitations that obstruct their wider use (LEE *et al.* 1998; HERIBAN *et al.* 1993). Electrodialysis is one of the very promising and perspective methods conditioned by rapid development of the membrane processes, especially the membranes in the 80's and 90's (ASENJO 1990).

Electrodialysis is a process where ion exchange membranes are used for removing ions from an aqueous solution under the driving force of electrical potential. Electrodialysis is applied to remove salts from solutions or to concentrate ionic substances. A large number of existing applications of electrodialysis was described in the literature (BLEHA *et al.* 1988; KRAVJAROVÁ 1991; MATEUS *et al.* 1993). A special type of electrodialysis is water-splitting electrodialysis. Instead of anion exchange membranes in desalting, bipolar membranes are used in water-splitting electrodialysis. Bipolar membranes are composed of anion and cation exchange layers. Water is split to H^+ and OH^- ions in the interlayer, which is formed by water film. Ions pass through the corresponding layer of the membrane. Water-splitting electrodialysis is applied to electroconversion of salts to the corresponding acids (MULDER *et al.* 1991).

There are two different methods for recovery of lactic acid. It is a two-stage electrodialysis method in the first case and electrodialysis with double exchange reaction in the second case. The two-stage electrodialysis method was described by GLASSNER and DATTO (1990). In the first step of desalting, sodium lactate is recovered, purified and concentrated, in the water-splitting or acidification step, lactic acid is regenerated from sodium lactate, and sodium hydroxide is recovered and purified. In the

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first step sodium lactate is pumped through the diluate circuit. When a direct current is applied, the positively charged sodium ions migrate to the cathode and pass through cation exchange membranes to the concentrate being accumulated there. The negatively charged lactate ions migrate to the anode and pass through anion exchange membranes to the concentrate. They are accumulated in the concentrate, too. The concentration of sodium lactate in the concentrate gradually increases. In the second step, the concentrated sodium lactate solution is pumped into the diluate cells, where it is converted to lactic acid by H^+ ions arising by water-splitting. Na^+ ions are pushed out through the cation exchange membranes to the concentrate, where they combine with OH^- ions to sodium hydroxide. LEE *et al.* (1998) studied the two-stage electrodialysis method for lactic acid recovery. In the desalting electrodialysis, 115 g lactate per litre was obtained in the concentrate, only 1g lactate per litre remained in the diluate, the current efficiency was about 90% and the energy consumption for lactate transfer from diluate to concentrate was 0.25 kWh/kg. In the second step, 88–93% of the total amount of lactate was converted to lactic acid, the current efficiency was about 80%. The total energy consumption for lactic acid recovery was in the range from 0.78 to 0.97 kWh/kg.

Electrodialysis with double exchange reaction represents the second method. It is a one-step process. The processed lactate solution and the mineral acid solution (e.g. H_3PO_4) are pumped into the diluate streams. There are two outlets, the first for the salt of mineral acid and the second for lactic acid. HERIBAN *et al.* (1993) reached 4-times higher lactic acid concentration in the continuous mode compared to the lactic acid concentration in the processed solution. 236.8 g lactic acid per litre was obtained in the course of experiment with model solutions, the energy consumption was in the range from 1.3 to 2.3 kWh/kg.

An advantage of the electrodialysis is a possibility to be coupled directly with fermentation. HONGO *et al.* (1986) proposed to use electrodialysis for *in situ* lactate recovery to reduce the product inhibition. The obtained productivity was three times higher than in non-pH controlled fermentation. However, the fouling of anion-exchange membranes by cells was observed in electrodialysis fermentation. To solve this problem, NOMURA *et al.* (1987) used immobilized growing cells entrapped in calcium alginate. The amount of lactic acid produced by semicontinuous electrodialysis fermentation using immobilized cells was 8-times higher than that produced by non-pH controlled fermentation, but some free cells were determined in the solution. CZYTKO *et al.* (1987) found out that the electrodialysis unit could only be operated with cell free solutions in order to prevent deposition of bacteria on the membrane surface and creation of bacteria clusters in the spacer between the membranes. To increase the productivity of the lactic acid fermentation and to reduce the amounts of effluents, BOYAVAL *et al.* (1987) chose for the lactic acid fermentation the bioreactor with total cell recycling, which was coupled with an ultrafiltration module and an electrodialysis unit, the outlet concentration of lactate was stabilized at 85 g/l. A similar system was studied by YAO and *TODA* (1990), H_2SO_4 was used as a donor of protons and lactic acid was the final product, its concentration reached 90 g/l.

MATERIAL AND METHODS

Chemicals: Sodium lactate (p. a. purity) was from Sigma, the other chemicals were chemically pure products of Lachema (Brno, Czech Republic). Demineralised water was prepared from distilled water in a device Millipore – Q gradient 18.2 MΩ.cm (Molsheim, France). Real fermentation broth was obtained from the continuous lactic acid fermentation with recycle using *Lactobacillus plantarum* L10 as a producer strain (SVOBODOVÁ 1999). Lactate concentration ranged from 17 to 22 g/l. Granulated active charcoal and non-ionogenic sorbent Amberlite XAD 4 (Supelco, Switzerland) were used as agents for decolourisation of fermentation broth.

Analytical Methods: Lactate, citrate, acetate and glucose were determined by HPLC (Laboratorní přístroje Prague, Czech Republic); column – Ostion LG KS in H-cycle; refractometer detector RIDK 101; mobile phase – H_2SO_4 ($c = 0.005$ mol/l), flow rate of mobile phase 0.5 ml/min; column temperature 85° C. The concentration of lactic acid and the concentration of NaOH were titrated by standard solution of NaOH ($c = 0.025$ mol/l), resp. HCl ($c = 1$ mol/l), using phenolphthalein as indicator. The colour of fermentation broth was measured by spectrophotometer at wavelength of 400 nm relative to water.

Electrodialysis Equipment: Electrodialysis laboratory unit BEL-500 (Berghof, Germany) consisted of control unit (adjustable outputs of voltage from 0 to 50 V and current from 0 to 3.9 A), measuring device (conductivity and voltage) and 3 independent circulation circuits with pumps and storage containers (for the diluate, the concentrate and the electrode solution). The membrane stack Type 500 (Berghof, Germany) and membrane stack ED 0 (Mega, Czech Republic) with 5 and/or 20 pairs of ion-exchange membranes Ralex CMH and Ralex AMH were used for the desalting electrodialysis experiments, their effective membrane areas were 58 cm^2 (Type 500) and 180 cm^2 (ED 0). Stack Type 500 with 4 bipolar membranes Neosepta BP-1 and 5 cation exchange membranes Neosepta CMB (Tokuyama Corp., Japan) were used for water-splitting electrodialysis.

Limiting Current Measurements: To maintain the constant lactate concentration, the diluate and concentrate circuits were connected. The relationship between voltage and current was measured for different concentrations of sodium lactate. The limiting current for each lactate concentration was identified from a graph.

Operating Conditions – Desalting Electrodialysis: Electrodialysis experiments were carried out in a batch mode. The electrode solution (Na_2SO_4 – 25 g/l), the concentrate (sodium lactate – initial concentration 4 g/l) and the diluate (sodium lactate – initial concentration from 15 to 42 g/l) were circulated through the corresponding compartments of the desalting stack with flow 60–70 l/h. For the constant current period, voltage 1.5 V per one pair and current 1.4 A were used for both stacks. For the constant voltage period, voltage 12V and 16 V were used for stack Type 500 and stack ED 0, respectively. The experiments were terminated when the lactate concentration in the diluate dropped to 1–2 g/l.

Operating Conditions – Water-splitting Electrodialysis: The following solutions were used: NaOH – 20 g/l (electrode solution), NaOH – 1g/l (concentrate), sodium lactate 43 and 178 g/l (diluate). Current 3.9 A and voltage 12 V were applied. When the conductivity in the diluate reached the value 5 mS/cm, the experiment was terminated.

Decolourisation: Decolourisation was carried out in the glass column filled with the above-mentioned material (bed volume 40 ml), the flow rate of the fermentation broth through the column was from 40 to 300 ml/h. The fermentation broth was filtrated through the microfilter before decolourising.

Calculations: The calculation equations were taken from LEE *et al.* (1998) and JANDOVÁ (1996).

RESULTS AND DISCUSSION

Desalting Electrodialysis

The initial measurements were focused on determination of the conditions for electrodialysis experiments, first of all on limiting current measurements for both membrane stacks. The limiting current is the maximum current for a given bulk lactate concentration, further current increase does not promote the ion transport rate any more, only the electrical resistance increases rapidly. The excessive energy is also dissipated for splitting water and the current efficiency significantly decreases. If the operating current is beyond the limiting value, the membranes can irreversibly be damaged. The method of limiting current determination is evident from Fig. 1. It has been found that the limiting current depends on the lactate concentration, limiting current increases with the increasing lactate concentration. The maximum limiting currents 1.51 A (for stack Type 500) and 1.58 A (for stack ED 0) were found with the lactate concentration 16 g/l and 6.5 g/l, respectively (Fig. 2). The limiting current was not found at higher lactate concentrations than those mentioned above. The working currents were a bit lower than the determined maximal limiting currents to prevent the operation beyond the limiting current. For the constant-current period, the current 1.4 A was used for both stacks. When the lactate concentration decreased to the limiting value, the operating mode was switched from the constant-current mode to the constant-voltage mode and voltages of 16 V and 12 V were adjusted for stack ED 0 and Type 500, respectively. The characteristic course of the electrodialysis experiment is illustrated in Fig. 3. The results of the performed experiments are shown in Table 1. The experiments DS 1–DS 3 were carried out with the aim of considering the influence of the applied current on the course of electrodialysis. It was found that the current value significantly influenced the transport rate of lactate ions through the membrane. A decrease in current from 1.4 A to1.0 A resulted in the prolongation of constant-current period by 90 min, that means by 60%, and in the significantly decreased rate of the lactate transport (Table 1 – experiments DS 3 and DS 1), therefore all other experiments were performed at current 1.4 A. The value of the lactate concentration in the concentrate is very important for the next recovery step, i.e. for electroconversion. The degree of concentration (the ratio of final concentrate concentration to initial diluate concentration) can be influenced by a change in the ratio of initial diluate volume to initial concentrate volume (Fig.4). However, the

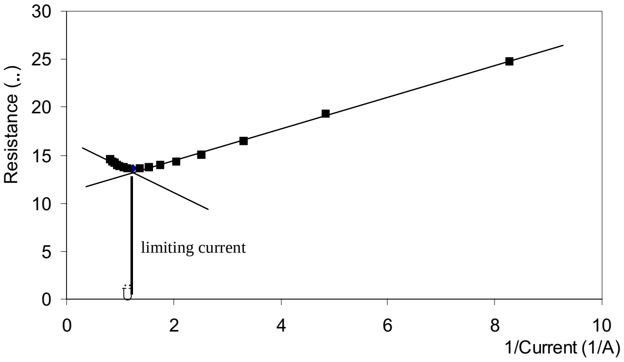

Fig. 1. Identification of the limiting current (membrane stack Type 500, lactate concentration = 7 g/l, limiting current = 0.84 A)

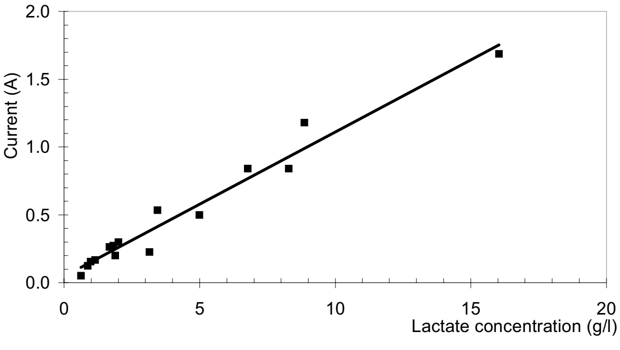

Fig. 2. The dependence of the limiting current on the lactate concentration (membrane stack Type 500)

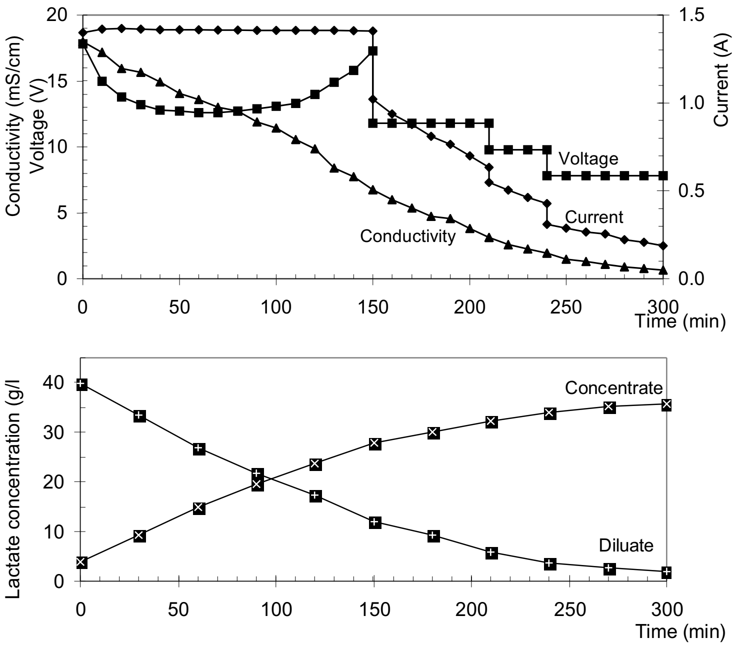

Fig. 3. The time course of the desalting electrodialysis experiment (membrane stack Type 500, experiment DS 3)

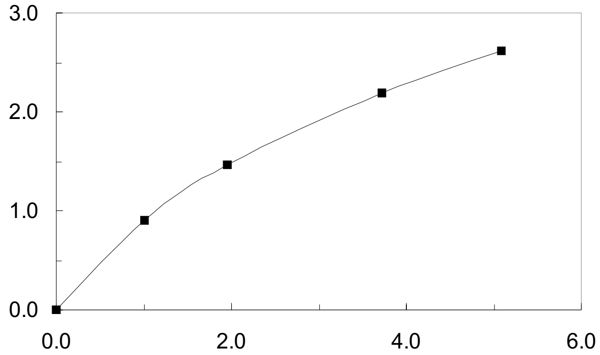

Fig. 4. Ratio of the final concentrate concentration to the initial diluate concentration (axis x) vs. ratio of the initial diluate volume to the initial concentrate volume (axis y)

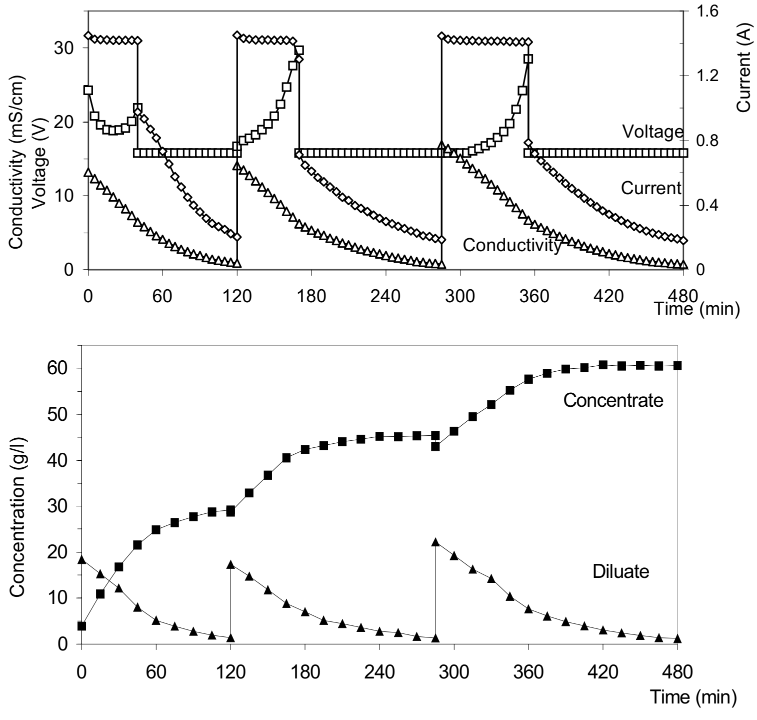

Fig. 5. The time course of the three-level desalting electrodialysis experiment (membrane stack ED 0, fermentation broth)

Table 1. Desalting electrodialysis experiments

Experiment	Operating time	Switching time	Concentration			V_{D0}/V_{K0}	Water transport index
			initial diluate	final diluate	final concentrate		
	(min)	(min)	(g/l)	(g/l)	(g/l)		(ml/g)
DS 1 [a c f]	360	240	41.2	1.6	37.5	1.00	4.25
DS 2 [a c g]	330	180	39.8	1.7	36.4	1.00	4.50
DS 3 [a c]	300	150	39.8	1.9	35.8	1.00	4.53
DS 4 [a c]	600	290	37.4	2.0	54.9	1.95	5.06
DS 5 [a c]	690	270	34.9	2.3	76.6	3.73	5.54
DS 6 [a c]	1020	480	42.5	2.1	111.2	5.09	4.74
DS 7 [b c]	80	50	43.7	0.8	34.1	1.00	7.08
DS 8 [b c]	160	100	39.6	0.8	52.6	1.87	5.27
DS 9 [b c h]	150	100	42.4	1.2	61.5	1.87	4.71
DS 10 [b c k]	100	100	40.4	1.2	55.9	1.87	5.20
DS 11 [b c]	75	15	15.3	0.7	26.9	1.87	4.78
DS 12 [b d]	120	40	18.4	1.3	29.2	1.87	6.97
DS 13 [b d]	165	50	17.3	1.3	45.4	1.87	9.04
DS 14 [b d]	195	70	22.2	1.2	60.6	1.87	9.10

	Lactate transport	Rate of lactate transport	Current efficiency	Energy consumption	Constant-current period		
					rate of lactate transport	current efficiency	energy consumption
	(g)	(g/h)	(%)	(kWh/kg)	(g/h)	(%)	(kWh/kg)
DS 1	82.4	13.7	80	0.61	17.2	81	0.60
DS 2	80.0	14.6	79	0.73	21.1	83	0.71
DS 3	79.5	15.9	78	0.79	23.8	80	0.82
DS 4	144.3	14.4	73	0.81	22.2	75	0.83
DS 5	130.9	11.4	65	0.90	21.2	71	0.87
DS 6	221.4	13.0	68	0.84	21.6	73	0.81
DS 7	83.3	62.5	68	0.34	79.6	67	0.35
DS 8	161.2	60.4	66	0.31	77.7	65	0.32
DS 9	180.3	72.1	74	0.27	91.1	77	0.26
DS 10	167.3	60.8	60	0.32	67.0	56	0.34
DS 11	60.7	48.6	76	0.29	80.8	68	0.40
DS 12	73.2	36.6	54	0.41	58.6	53	0.45
DS 13	67.5	24.6	42	0.54	53.1	45	0.56
DS 14	90.1	27.7	44	0.48	53.4	45	0.48

[a]stack Type 500; [b]stack ED 0; [c]model solutions of lactate; [d]fermentation broth; [f]current 1 A; [g]current 1.2 A; [h]addition of glucose to the diluate (9 g/l); [k]addition of glucose (9 g/l) and salts (K_2HPO_4, $MgSO_4$, $MnSO_4$, sodium acetate, ammonium citrate) to the diluate; V_{D0}/V_{K0} ratio of the initial diluate volume to the concentrate volume

transport rate decreased with a higher ratio of initial volumes. In the course of electrodialysis experiments the concentrate and diluate volumes changed due to water passage by electroosmosis through the membranes simultaneously with lactate ions. Water transport index (amount of passed water to amount of transported lactate) was in the range from 4.3 to 5.5 ml/g (stack Type 500) and from 4.8 to 7.1 ml/g (stack ED 0) in our experiments.

The influence of other components (glucose and salts, which are commonly present in the fermentation broth) on electrodialysis (see the legend to Table 1) was studied. While the addition of glucose to the diluate did not show any effect, the addition of salts resulted in a decrease in current efficiency. The current efficiency was in the range from 60% to 80% during the electrodialysis experiments with the model solutions and the energy consumption

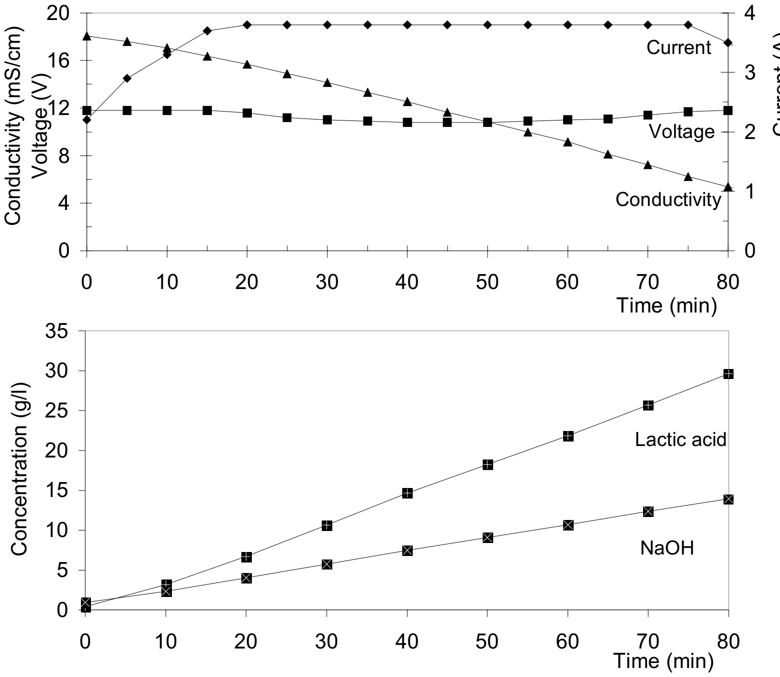

Fig. 6. The time course of the water-splitting electrodialysis experiment (WS 1)

was from 0.6 to 0.9 kWh/kg (stack Type 500) and 0.3 kWh/kg (stack ED 0). The electrodialysis experiments with the pre-treatment of fermentation broth were carried out. The fermentation broth pre-treatment was carried out to prevent a decrease in electrodialysis efficiency due to the colour fixing on the membrane surface (HERIBAN 1993). The comparison of the lactate recovery from the model solutions (DS 11) and from the fermentation broth (DS 12) has shown that in the case fermentation broth the transport rate decreased by 25%. The energy consumption increased and the current efficiency decreased. The other components that are present in the fermentation broth were probably transported through the membranes together with lactate. Fig. 4 shows the time course of the three-level electrodialysis with fermentation broth when 231 g lactate was transported from the diluate to the concentrate. About 94% of the total amount of lactate present in the diluate was recovered. About 1 g lactate per litre remained in the diluate after each level. The concentration of lactate obtained during the three-level electrodialysis was about 3 times higher than the lactate concentration in the fermentation broth.

Water-splitting Electrodialysis

Water-splitting electrodialysis was the second recovery step, when sodium lactate was converted to lactic acid. Fig. 6 shows an example of water-splitting experiment (WS 1). It is obvious that the experiments were carried out at constant fixed current (3.9 A), which was higher than that during desalting electrodialysis. The results of two water-splitting experiments (Table 2) indicate that the amount of converted sodium lactate is proportional to

Table 2. Water-splitting experiments

Experiment	Operating time (min)	Initial diluate concentration (g/l)	Final diluate concentration (g/l)	Final concentrate concentration (g/l)	Na^+ transport (g)
WS 1	82.5	43.3	29.7	14	15.9
WS 2	406.0	178.3	156.8	58	70.8

Experiment	Water transport index (ml/g)	Rate of Na^+ transport (g/h)	Rate of lactic acid conversion (g/h)	Current efficiency (%)	Energy consumption (kWh/kg)
WS 1	0.0038	11.6	45.3	92	0.91
WS 2	0.0030	10.5	41.0	78	0.84

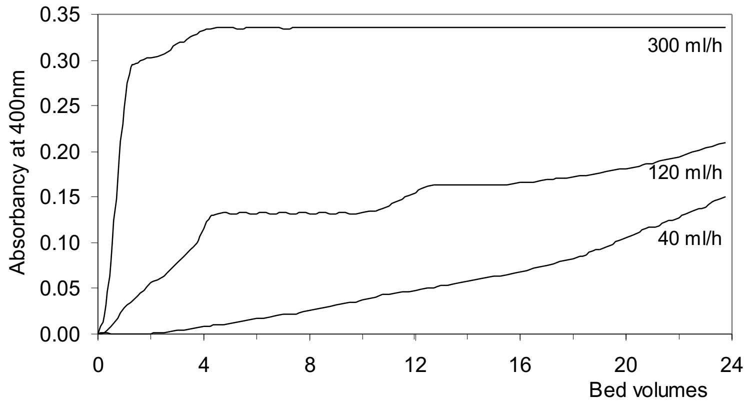

Fig. 7. Decolourisation of fermentation broth at different flow rates

the amount of supplied charge. High current efficiency 80–90% was achieved. The energy consumption of water-splitting electrodialysis has increased in comparison with desalting electrodialysis, from 63 to 79 Wh/mol. The results confirmed the possibility of sodium lactate conversion to lactic acid with the use of water-splitting electrodialysis with bipolar membranes.

Decolourisation

Due to high demands of the electrodialysis membranes, esp. water-splitting ones, for the quality of solutions used in electrodialysis processes, the fermentation broth had to be decolourised. Two decolourising agents were tested. Granulated active charcoal showed the higher decolourisation capacity than Amberlite resin. Further experiments were carried out with active charcoal only. The flow of the fermentation broth through the column significantly influenced the decolourisation degree. The course of decolourisation experiments at different flow rates is shown in Fig.7. When the flow rate was only 40 ml/h, the amount of fermentation broth corresponding to the 10-fold bed volume was decolourised from 90%. When the decolourisation was carried out in a batch mode, decolourisation degree was equal to that mentioned above, but more decolourising agents were consumed.

CONCLUSION

The obtained results confirm that two-stage electrodialysis is a suitable and efficient technique for recovery of lactate ions from the pre-treated fermentation broth and subsequent conversion into lactic acid with respect to environmental aspects.

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Souhrn

HÁBOVÁ V., MELZOCH K., RYCHTERA M., PŘIBYL L., MEJTA V. (2001): **Využití elektrodialýzy pro izolaci kyseliny mléčné.** Czech J. Food Sci., **19**: 73–80.

Práce se zabývá využitím dvoustupňové elektrodialýzy pro izolaci kyseliny mléčné z modelových roztoků a fermentačního média. V prvním stupni byl laktát sodný nakoncentrován pomocí elektrodialýzy s iontoměničovými membránami Ralex.V 1 l koncentrátu bylo získáno až 111g laktátu sodného, tzn., že koncentrace se ve srovnání s původní hodnotou zvýšila více než 2,5krát, v diluátu zůstávalo nejvýše 2 g laktátu v 1 litru. Ve druhém stupni byl laktát sodný pomocí elektrodialýzy s bipolárními membránami Neosepta konvertován na kyselinu mléčnou – v 1 l diluátu bylo 157 g kyseliny mléčné. Na izolaci (tj. na koncentrování a na elektrokonverzi) kyseliny mléčné bylo zapotřebí celkem 142 kWh/mol. Reálná fermentační média byla před elektrodialyzačními pokusy odbarvována. Nejlepších výsledků bylo dosaženo s granulovaným aktivním uhlím v koloně s nízkým průtokem.

Klíčová slova: izolace kyseliny mléčné; elektrodialýza; odsolovací elektrodialýza; elektrodialýza s bipolárními membránami; odbarvování

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