

Conversion of Sodium Lactate to Lactic Acid with Water-Splitting Electrodialysis

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Abstract

The conversion of sodium lactate to lactic acid with water-splitting electrodialysis was investigated. One way of reducing the power consumption is to add a conductive layer to the acid compartment. Doing this reduced the power consumption by almost 50% in a two-compartment cell, whereas the electric current efficiency was not affected at all. Three different solutions were treated in the electrodialysis unit: a model solution with 70 g/L of sodium lactate and a fermentation broth that had been prefiltered two different ways. The fermentation broth was either filtered in an open ultrafiltration membrane (cut-off of 100,000 Dalton) in order to remove the microorganisms or first filtered in the open ultrafiltration membrane and then in an ultrafiltration membrane with a cut-off of 2000 Dalton to remove most of the proteins. The concentration of sodium lactate in the fermentation broth was 70 g/L, as well. Organic molecules present in the broth (peptides and similar organic material) fouled the membranes and, therefore, increased power consumption. Power consumption increased more when permeate from the more open ultrafiltration membrane was treated in the electrodialysis unit than when permeate from the membrane with the lower cut-off was treated, since there was a higher amount of foulants in the former permeate. However, the electrodialysis membranes could be cleaned efficiently with a 0.1 M sodium hydroxide solution.

Index Entries: Lactic acid; electrodialysis; bipolar membranes; fermentation broth; wheat.

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Introduction

Lactic acid is a well-known organic acid that can be polymerized to polylactic acid, which is biodegradable and therefore used in the pharmaceutical industry for the production of sutures and matrices for slow-release drugs (1). Lactic acid can be produced by both organic synthesis and fermentation (2). Organic synthesis is most widely used for the production of lactic acid in the pharmaceutical industry, but as the cost of petroleum has increased, fermentation has become more interesting (3). Fermentative production of lactic acid is inexpensive, especially when waste products such as whey are used as substrate. On the other hand, the subsequent downstream processing is difficult owing to the complexity of the media and the byproducts, such as proteins and organic acids, formed during fermentation. In addition, lactic acid has a high boiling point and polymerizes at elevated temperatures, and, thus, traditional processes, such as distillation and conventional evaporation, cannot be used for the recovery (4). Because lactic acid-producing bacteria often exhibit the highest production rate at pH values over the pK_a of lactic acid, the acid is present as a salt (e.g., sodium lactate) in the fermentor. Many different processes have been suggested for the downstream processing of lactic acid/lactate, including extraction and ion-exchange chromatography (5,6). Another method of recovery is to precipitate the lactic acid with calcium hydroxide, refine the calcium lactate, and convert it to lactic acid by acidification with sulfuric acid (7). These processes all have disadvantages in that they are unspecific and expensive, produce waste streams, and are difficult to scale up (4,7–9). Water-splitting electrodialysis is a membrane process that has been suggested as a competitive alternative for the conversion of lactate to lactic acid because the process does not produce waste streams.

Research has been conducted on electrodialysis for the desalination of seawater. However, electrodialysis has been found to be a more realistic alternative when used for desalination of brackish water (10–12). It is also used to remove salts from process water in power plants and to remove radioactive substances from water in nuclear power plants (13). Recently, electrodialysis has attracted interest in other areas for the recovery or removal of chemicals in process streams such as those in the pulp and paper and the iron and steel industries (14,15). In the food industry, electrodialysis has been suggested for various processes such as desalination of whey, prevention of precipitation of tartrates in wine, and for purification of proteins (16). In the biotechnologic industry, electrodialysis can be used for the refinement of organic acids from fermentation broths (17,18). Much interest has been focused on recovery of the organic acid lactic acid from fermentation broth using electrodialysis to use the acid for the production of the biodegradable and environmentally friendly polymer polylactic acid (19–22). Several mathematical models for the conversion of sodium lactate to lactic acid have also been presented (23–25).

The use of bipolar membranes for the conversion of lactate to lactic acid has recently attracted interest (24,26). In the bipolar membrane, water molecules are split into hydrogen and hydroxide ions. These ions can be combined with the ions in the salt, which are assumed to be converted into an acid and a base, without adding any extra chemicals, thus preventing the production of waste streams. The streams leaving the electrodialysis unit are the product (lactic acid in this case) and the base (sodium hydroxide), which can be reused as a pH adjuster in the fermentor.

The present study is part of a project on process development for the production and refinement of lactic acid from starchy raw material, mainly wheat flour (27–31). The total process comprises prehydrolysis and saccharification of starch, fermentation, and separation and concentration of the product. In this article, we describe the conversion of sodium lactate to lactic acid using electrodialysis with bipolar membranes. Experiments were performed with a conventional three-compartment cell and for energy-saving reasons; a two-compartment cell was also investigated. In both cases, a model solution of sodium lactate and one or two fermentation broths containing sodium lactate were used.

Materials and Methods

Preparation and Fermentation of Medium

The lactic acid-producing bacterium *Lactococcus lactis* spp. *lactis* ATCC 19 435 was grown on a medium consisting of wheat flour hydrolysate and yeast extract. The wheat flour hydrolysate was prepared by passing the flour (4800 g) through a sieve, mesh 0.4 mm, to remove the bran. This flour was then mixed with water (20 L) and heated to 50°C, and 67 µL/L of the enzyme α -amylase (Termamyl 120 L; Novo Nordisk, Bagsvaerd, Denmark) was added. The mixture was heated to 95°C and maintained at this temperature for 50 min, while liquefaction occurred. The medium was cooled to 30°C and diluted to 50% of the original concentration using water. The medium (10 L) was then transferred to the fermentor, where 1200 µL/L of the enzyme mixture SAN Super 240 L (α -amylase and amyloglucosidase) (Novo Nordisk), 5 g/L of yeast extract, and the inoculum were added (27). Fermentation was performed as simultaneous saccharification and fermentation. Owing to product inhibition of growth and thus product formation, resulting from the formation of sodium lactate, the glucose concentration had to be <73 g/L (30).

The inoculum was prepared by taking a colony of *L. lactis* grown on M17 agar in a Petri dish and transferring it to a test tube with 5 mL of M17 broth (Merck, Darmstadt, Germany). This was incubated overnight at 30°C, and the mixture was then transferred to a 1-L Erlenmeyer flask containing 500 mL of M17. The flask was incubated for 12 h, and the cells were then transferred to the fermentor.

Lactic acid was produced in a 22- or a 16-L fermentor (model NLF22 and NLF16; Bioengineering, Wald, Switzerland) with baffles. The tempera-

ture was 30°C, the stirring speed 300 rpm, and the pH was maintained at 6.0 by adding 30% (w/v) NaOH. Fermentation was allowed to proceed for 3 d.

Bacterial Cell and Protein Separation

To avoid fouling in the electrodialysis unit, the flour particles, bacterial cells, and proteins had to be separated. The bacterial cells and the flour particles were separated from the broth in a shear-enhanced, crossflow, laboratory-scale ultrafiltration module (CR filter from Flootek, Malmö, Sweden) with recirculation of the retentate (29). The module was equipped with two membranes: one above and one below the rotor blade. The area of each membrane was 0.05 m². A polymeric ultrafiltration membrane PS100 (Hoechst, Wiesbaden, Germany) with a nominal cut-off of 100,000 Dalton was used. This membrane was chosen because previous experiments with ceramic microfiltration (MF) membranes showed that MF membranes have a higher tendency toward irreversible fouling than membranes with somewhat smaller pores such as open ultrafiltration membranes (results not shown). Proteins were separated in the same module, but a membrane with a cut-off of 2000 Dalton was used (PS2; Hoechst). In the electrodialysis experiments, recovery of lactic acid from both permeate from the open ultrafiltration membrane and permeate from the ultrafiltration membrane with a lower cutoff was investigated.

Electrodialysis

In the electrodialysis unit, the sodium lactate is converted to lactic acid and sodium hydroxide. Two different membrane configurations were used: a three-compartment cell and a two-compartment cell.

Three-Compartment Cell

The membrane module used had a membrane area of 33 cm² (constructed at the Department of Chemical Engineering; DTU, Lyngby, Denmark). The module was equipped with four membranes, two bipolar membranes, a cation-exchange and an anion-exchange membrane, fitted between 0.6-mm-thick tortuous path spacers. The configuration is shown in Fig. 1. The three-compartment cell was used only in batch separation mode.

Two-Compartment Cell

Two different modules were used. One had a membrane area of 10 cm² (Micro Flow Cell; Electrocell AB, Täby, Sweden), and the other was the one used in the experiments with the three-compartment cell, adapted to a two-compartment module (membrane area of 33 cm²). The membrane configuration (two bipolar membranes and an anion-exchange membrane) was the same for both modules (Fig. 2). Limiting current trials showed that there was only a very small difference between the two modules, so the results from the two modules are comparable. Both batch separation mode and continuous separation mode were investigated with the two-compartment cell configuration.

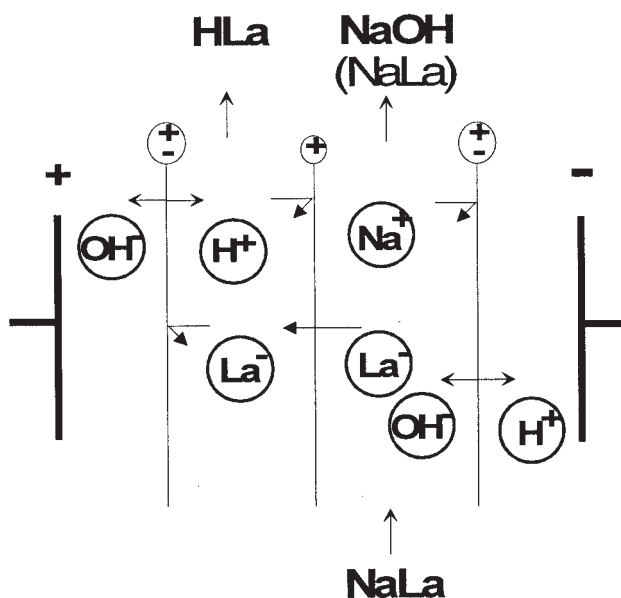


Fig. 1. Membrane configuration in the three-compartment cell. (+) anion-exchange membrane; (-) cation-exchange membrane; (±) bipolar membrane.

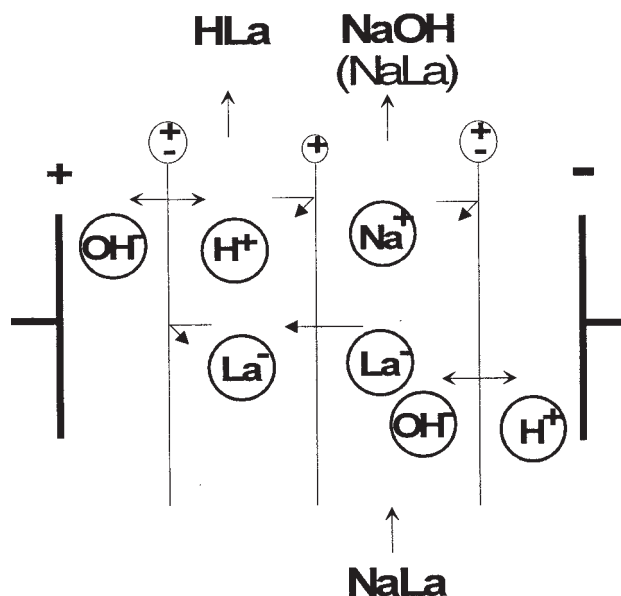


Fig. 2. Membrane configuration in the two-compartment cell. Symbols are the same as in Fig. 1.

Membranes and Experimental Conditions

The bipolar membranes used were BP-1 (Tokuyama, Tokyo, Japan). The anion-selective membrane used was Selemion AMV membrane (Asahi

Glass, Tokyo, Japan). The cation-selective membrane used in the three-compartment cell was a Neosepta CMB (Tokuyama).

In both the three-compartment and two-compartment cell experiments, the current density was 7 A/cm², and the crossflow velocity over the membranes was in the range of 0.05–0.07 m/s in the batch experiments and 0.0023 m/s in the continuous-mode experiments. The temperature was maintained at 25°C in all experiments. Three solutions were treated in the electrodialysis unit: one model solution with a concentration of 70 g/L of lactic acid (KEBO Lab A/S, Albertslund, Denmark) and two different ultrafilter permeates (fermentation broth with approx 70 g/L of sodium lactate). The first permeate was fermentation broth treated in an open ultrafiltration membrane (PS100) with a cut-off of 100,000 Dalton and the second permeate was broth first treated in the membrane with a cut-off of 100,000 Dalton and then in a membrane (PS2) with a cut-off of 2000 Dalton.

Analyses

The concentration of lactic acid was analyzed with high-performance liquid chromatography in an Aminex HPX-87H column (Bio-Rad, Richmond) as reported previously (29).

The protein concentration was measured with the Bradford Coomassie Brilliant Blue method (Coomassie Protein Assay Reagent 23200; Pierce, Rockford, IL). The concentration of inorganic ions was measured with ion chromatography using an AS4ASC column (Dionex, Sunnyvale, CA), and the eluting buffer was 1.80 mM Na₂CO₃/1.70 mM NaHCO₃.

Cleaning Procedure

After filtration, the ultrafiltration membranes were rinsed with deionized water until the retentate stream was clear, and the electrodialysis membranes were rinsed until the pH was neutral. The polymeric ultrafiltration membranes were cleaned with a 0.4% alkaline cleaning agent (Ultrasil 10; Henkel, Germany), which was recirculated in the system at 50°C for 20 min. The system was then rinsed with deionized water at 20°C (5 × 5 L). A solution of 0.1 M NaOH at 25°C was recirculated in the electrodialysis modules for 30 min, and then the membrane was rinsed with deionized water until the pH was neutral.

Results and Discussion

The fermentation broth used was well suited for the electrodialysis step since the concentrations of small inorganic ions that could compete with the lactate ions (625 mM) were low (2.25 mM chloride ions, 1.46 mM phosphate ions, and 0.20 mM sulfate ions).

The results from the experiments using electrodialysis are presented as current efficiency, CE (%), and power consumption, PC (kWh/kg).

$$CE = [n / (I \cdot F \cdot t)] \quad (1)$$

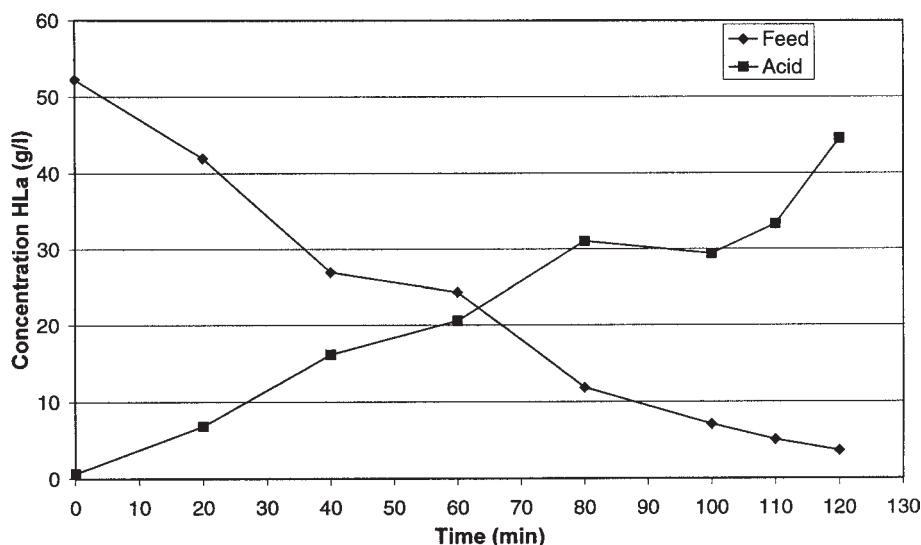


Fig. 3. Concentration in the feed and in the acid stream vs time in the three-compartment cell using a 70 g/L sodium lactate model solution.

$$PC = [U \cdot I \cdot t / (M \cdot 1000 \cdot 3600)] \quad (2)$$

in which n is the molar amount of lactate molecules transported over the membrane, I is the current (A), F is Faraday constant (As/mol), t is the time (s), U is the voltage (V), and M is the mass of the lactic acid formed (g). The current efficiency is given as the overall current efficiency, and the power consumption is based on the voltage over the whole membrane stack, including the electrode compartments.

Three-Compartment Cell

The most widely reported membrane configuration when converting lactate to lactic acid with bipolar membrane electrodialysis is the three-compartment cell. The change in concentration of lactate in the feed and in the concentrate stream can be seen in Fig. 3. In the experiment shown in Fig. 3, the model solution 70 g/L of sodium lactate at pH 6.0 was used. The overall current efficiency was 76%. The reasons for the current efficiency being <100% may be owing to a selectivity of the anion-exchange membrane <100% (i.e., cations are also transported through the membrane), back-diffusion of already converted lactic acid, or current leakage through manifolds and tubes used for the electrode rinsing solution. The overall current efficiency decreased with time (80% at 40 min, 78% at 80 min, and finally 76% at 120 min). The power consumption was 5.6 kWh/kg, calculated over the whole stack, including the electrode compartments. The resistance over the electrode compartments constitutes a significant fraction of the energy required for the process, and, thus, the energy consumption per kilogram of lactic acid can be reduced if the number of cells (per electrode pair) in the stack is increased.

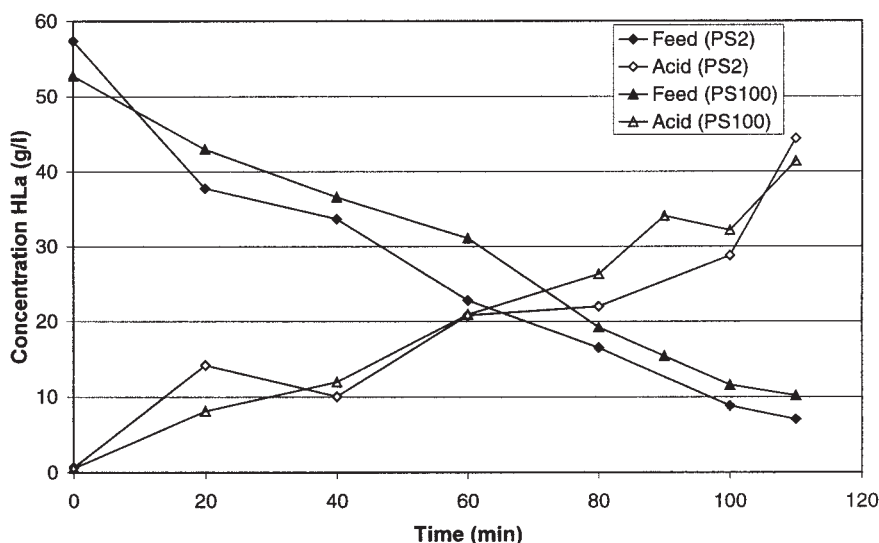


Fig. 4. Comparison of the concentration in the feed and in the acid stream vs time in a three-compartment cell using two fermentation broths treated in a PS100 and in a PS2 membrane.

Two experiments were conducted with fermentation broth: one using permeate from the PS100 membrane and one using permeate from the PS2 membrane (Fig. 4). The current efficiency for the two fermentation broths was about the same, 82% for the PS2 permeate and 77% for the PS100 permeate. However, the power consumption was higher for the PS100 permeate (7.3 kWh/kg) than for the PS2 permeate (6.5 kWh/kg). The increase in power consumption compared with the model solution was probably owing to fouling of the membranes by charged molecules (other than lactic acid), e.g., amino acids and polypeptides. The greater increase when permeate from the PS100 membrane was used is owing to the higher cut-off of the membrane, which leads to a higher concentration of foulants in the permeate. The concentration of proteins in the PS100 permeate was 0.20 mg/mL and in the PS2 permeate 0.05 mg/mL. When membrane fouling occurs, the membrane resistance increases, thus increasing the power consumption. However, the membrane properties were restored during cleaning.

The concentration of lactic acid in the acid stream was quite low (50 g/L); thus, if the concentration could be increased without extra power consumption, the gain could be considerable. To achieve this, lactate ions from a large volume (800 mL) of feed were transferred to a smaller volume of acid solution (200 mL). The results are shown in Fig. 5.

From simple mass balance calculations, the maximum theoretical concentration of lactic acid in the acid stream for this module is estimated to be about 280 g/L, which is the ratio between the total lactic acid flux and the total water flux into the acid compartment. In an investigation by Börgardts et al. (26), the highest concentration reached in the acid stream

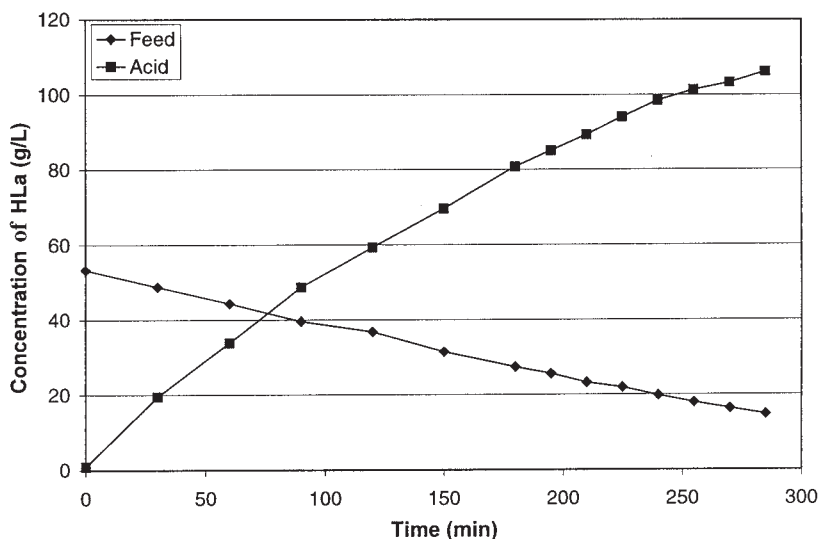


Fig. 5. Concentration in the feed and acid streams vs time in the three-compartment cell when increasing the concentration in the acid stream by transferring lactate ions from a larger volume of feed.

using the same membrane configuration was about 200 g/L. There could be several reasons for the difference between the higher estimated value in the present study and the value given in their study. The most important is that the 200 g/L is a “real” maximum value where back-diffusion of lactic acid and the osmotic water flux owing to higher ion concentration in the acid compartment is at its maximum. In the present experiment, the maximum lactic acid concentration was evaluated on the basis of the separation seen in Fig. 5 in which the concentration in the acid compartment only reached 106 g/L; that is, the process had not reached steady state. Therefore, the lower back-diffusion and lower osmotic water flux in the considered interval might lead to an overestimation of the maximum lactic acid concentration attainable. Other reasons for the difference in the two maximum values could be different concentrations of inorganic anions in the feed streams, facilitating extra electroosmotic water flux, and, of course, that different membranes might also have different water transport properties.

Two-Compartment Cell

To decrease the resistance in the module, the cation-exchange membrane was removed and the module was run with two compartments. To reduce the conductivity even further, the conductivity in the acid compartment was increased by the addition of salt. Lactic acid in a water solution has a low conductivity, even at high concentrations (Fig. 6). This low conductivity makes the voltage over the module very high, and, thus, the power consumption becomes high.

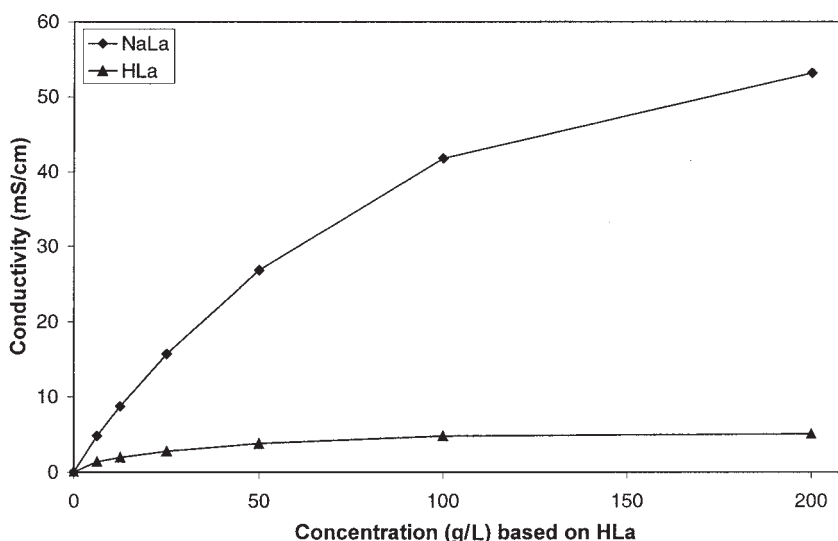


Fig. 6. Conductivity vs concentration of lactic acid and sodium lactate (based on lactic acid equivalents).

If a conductive layer is used in the acid compartment of the module, the resistance and thus the power consumption can be reduced (32,33). In the present study, the resistance in the acid compartment was reduced by using a 2% sodium chloride solution as acid stream. The disadvantage of sodium chloride is that it is difficult to separate it from the lactic acid after the electrodialysis step. A more convenient way of reducing the resistance in the acid compartment is to use beads of charged chromatography gel, because they can easily be removed by filtration after electrodialysis.

Four experiments were performed with the two-compartment cell with feed solutions at pH 6.0: two with the model solution (70 g/L of sodium lactate) and two with permeate from the PS2 membrane. Continuous and batch separation were employed with both solutions.

The final concentration did not differ between continuous and batch separation, being about 20 g/L of lactic acid in the acid compartment in all cases. Figure 7A shows the results from the batch and continuous separation experiments when the fermentation broth (permeate from the PS2 membrane) was treated in the module.

The current efficiency was 33% when the model solution was treated in the module (for both batch and continuous separation mode) and somewhat lower when the fermentation broth was treated (30% for batch separation and 28% for continuous separation). The power consumption was also about the same for the two separation methods, about 8 kWh/kg for the model solution and 10 kWh/kg for permeate from the PS2 membrane. However, the current efficiency decreased and the power consumption increased when the PS2 permeate was processed in the electrodialysis unit compared with model solution (Fig. 7B). This was owing to fouling of the

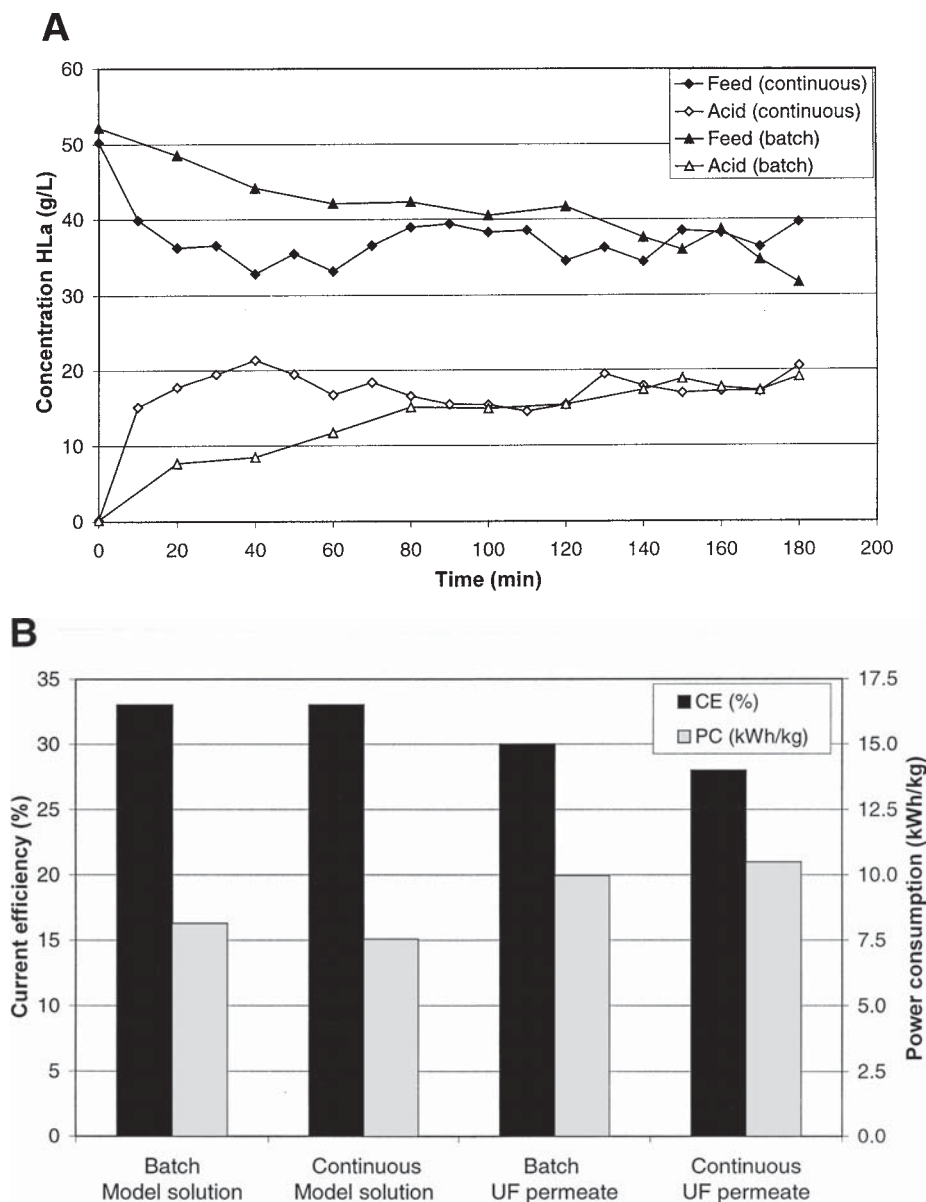


Fig. 7. **(A)** Concentration in the feed and acid compartment vs time for continuous and batch separation. PS2 permeate at pH 6.0 was used as the feed. **(B)** Current efficiency and the power consumption for batch and continuous separation when model solution and PS permeate were processed in the electrodialysis unit. UF, ultrafiltration; CE, current efficiency; PC, power consumption.

membrane, the same kind of fouling as was encountered in the three-compartment cell.

The power consumption of the two-compartment cell was higher in all cases than that of the three-compartment cell, although a conductive layer

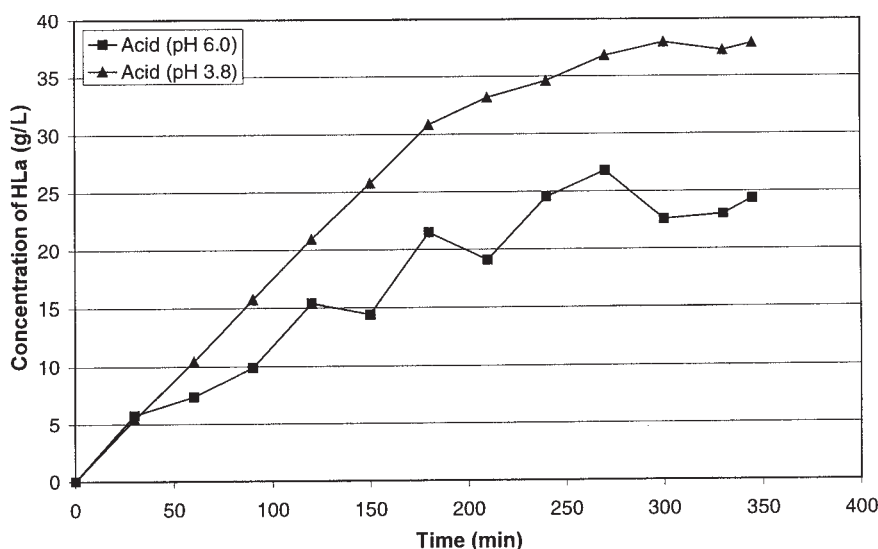


Fig. 8. Concentration of lactic acid in the acid stream vs time for a feed with low pH (3.8) and a feed with high pH (6.0). Model solutions with 70 g/L of sodium lactate molar equivalents were used.

was employed in the acid compartment in the two-compartment cell. For example, when the model solution was used in the batch experiment, the power consumption was 8.1 kWh/kg for the two-compartment cell, whereas it was 5.6 kWh/kg for the three-compartment cell. This was owing to the low current efficiency of 33% for the two-compartment cell, compared with 75% for the three-compartment cell.

This very low current efficiency was thought to be owing to competition between the lactate and hydroxide ions produced by the bipolar membrane. To elucidate this, a model solution of 70 g/L of sodium lactate equivalents at pH 3.8 was processed in the electrodialysis unit. The pK_a of lactic acid is at about pH 3.8, so 50% of the molecules are present in their acid form and 50% are present in their salt form. The first hydroxide ions produced by the bipolar membrane neutralize the molecules in the acid form, and, thus, the competition between the lactate and hydroxide ions can be avoided. It was found that the transport of lactate ions over the anion-exchange membrane increased when the pH in the feed stream decreased (Fig. 8). The current efficiency was 66% when the pH of the feed was 3.8 compared with 33% with a feed pH of 6.0. The power consumption was simultaneously reduced from 8.1 to 4.7 kWh/kg. Unfortunately, most of the bacteria used for the production of lactic acid do not have pH optima for the production of lactic acid at such a low pH as 3.8. Hence, lowering the pH is not a realistic alternative in a commercial plant for the production of lactic acid.

The final investigation was carried out to show that the conductive layer actually had a positive influence on the power consumption. The experiment with the low-pH feed was compared with an experiment under

similar conditions except that the solution in the acid compartment was pure water. When no salt was present in the acid compartment, the power consumption was doubled compared with the case in which sodium chloride was present in the acid compartment (8.3 and 4.7 kWh/kg, respectively), although the current efficiency was about the same for the two experiments, 66% with sodium chloride and 64% without sodium chloride in the acid compartment.

Conclusion

The ultrafiltered fermentation broth (lactic acid bacteria grown on hydrolyzed wheat starch) used in this investigation was well suited for use in the electrodialysis, because the concentration of inorganic ions is very low. It was found that the three-compartment cell exhibited higher current efficiency and lower power consumption than the two-compartment cell, since competition between hydroxide and lactate ions can be avoided in a three-compartment cell.

It was also shown that the power consumption could be reduced by almost 50% when a conductive layer was added in the acid compartment and that this did not effect the yield nor the current efficiency. This was found to be the case for the two-compartment cell but would probably also reduce the power consumption for the three-compartment cell.

It was also found that when fermentation broth was treated in the electrodialysis module, the power consumption increased owing to fouling by larger organic molecules present in the broth despite the fact that the broth had been pretreated with ultrafiltration. The fermentation broth treated in the membrane with a cut-off of 100,000 Dalton fouled the membranes more than the broth that also had been treated in a membrane with a cut-off of 2000 Dalton. The difference in power consumption was only about 1 kWh/kg, and because the membrane could be cleaned in both cases, it might be advantageous to omit the second ultrafiltration step, depending on the capital and operating cost of the extra ultrafiltration membrane unit. However, to be able to evaluate further the advantages or drawbacks with this process, an economical evaluation in which electrodialysis is compared with other process alternatives, such as ion-exchange chromatography or extraction, has to be made.

Appendix 1

Calculation of the maximal concentration of lactic acid is made as follows. The total mass of lactic acid in the system is calculated from the start concentrations in the acid and feed compartments:

$$M_{TOT} = C_{F0} \cdot V_{F0} + C_{A0} \cdot V_{A0} \quad A1.1$$

in which C_{A0} and C_{F0} are the start concentrations in the acid and feed streams, respectively, and V_{A0} and V_{F0} are the start volumes of acid and feed, respec-

tively. The total mass can also be determined using the final concentrations in the acid and feed compartments (C_A and C_F):

$$M_{TOT} = C_F \cdot (V_{F0} - X) + C_A (V_{A0} + X) \quad A1.2.$$

in which X is the volume (lactic acid + water) that has been transferred from the feed compartment to the acid compartment. The mass transferred from feed to acid stream is calculated from

$$M = C_A \cdot (V_{A0} + X) \quad A1.3.$$

The theoretical maximum concentration is calculated from

$$C_{MAX} = M/X \quad A1.4.$$

Appendix 2: Notation

C_A	= concentration of lactic acid in acid compartment (g/L)
C_{A0}	= start concentration of lactic acid in acid compartment (g/L)
C_F	= concentration of lactic acid in feed compartment (g/L)
C_{F0}	= start concentration of lactic acid in feed compartment (g/L)
F	= Faraday constant (As/mol)
I	= current (A)
M	= transferred mass of lactic acid (g)
M_{TOT}	= total mass of lactic acid (g)
n	= molar amount of molecules transported over membrane (mol)
PC	= power consumption (kWh/kg)
t	= time (s)
U	= voltage (V)
V_{A0}	= start volume of acid stream (L)
V_{F0}	= start volume of feed stream (L)
X	= volume of lactic acid and water transported over membrane (L)
η	= current efficiency

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