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Recovery of organics from aspartame effluents with electrodialysis

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ABSTRACT

In the process for producing aspartame, a great deal of effluents containing sodium chloride and organic acids were discharged. It not only made serious pollution but decreased the yield of aspartame. Recovery of organics from aspartame effluents by removing salt with electrodialysis (ED) is proposed in this paper. The desalinated solution containing few organic reactants could be reused into the synthesis unit. A laboratory-scale electrodialyzer of two compartments with 10 cell pairs was employed. The content of organics in the effluents was determined with total organic carbon (TOC) analyzer. The influences of voltage and flow rate on operation time, energy consumption, current efficiency and organic mass loss were investigated. The ED proved to be an effective method to remove salts from aspartame effluents. The experimental results showed that the optimal operation condition was at 12 V and 20 L/h for the electrodialyzer. During 65 min, the recovery of organics and the salt removal rate were 78 and 98%, respectively. The most efficient time for the laboratory-scale ED process is about 40 min from 10 to 50 min. The diffusion of small organics through ion exchange membranes could be the major reason for the low organics recovery. The current efficiency was 69.9% and the energy consumption was 0.786 kWh/kg. These basic data might be helpful for the large-scale application of ED in treatment of aspartame effluents.

Keywords: Electrodialysis; Aspartame; Effluent; Current efficiency

1. Introduction

As a kind of noncariogenic sweetener [1], aspartame has been used in food, beverage, pharmaceutical, and tabletop sweetener in several thousands of different products and consumed by hundreds of millions of people all over the world. Due to the high sweetness (160–200 times sweeter than sucrose), without bitter or metallic aftertaste, aspartame was used more and more widely. Generally, aspartame could be synthesized in chemical methods or enzymatic methods and the former was easy to be industrialized. In the process for producing aspartame with the chemical method, lots of effluents containing sodium chloride and aspartame were discharged after crystallization. Not only the yield of aspartame would be decreased, but also pollution was caused. Therefore, the effluent should be treated in some suitable technologies. Organic wastewater is often treated by biotechnologies [2,3]. However, highly saline effluents are recalcitrant to biological treatment. Thus, pretreatment technologies including evaporation [4], electrochemical technology [5], and ion exchange were used to remove

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salts. These pretreatment methods couldn't solve all the problems caused by the high salinity of solution due to the high cost of treatment.

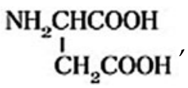
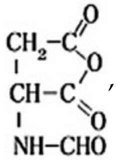
Electrodialysis (ED) could separate ions from an aqueous solution by the ions transport through selective membranes under the influence of an electrical field [6–8]. An electrodialyzer generally consisted of several pairs of cation exchange membranes (CEM) and anion exchange membranes (AEM) arranged between an anode and cathode. It is an important method for desalination from natural water, brine water, seawater [9–11], and organic solutions [12,13]. As for the aspartame effluents containing some organics and salts, if ED was used to separate salts from the effluents, the desalinated effluents could be reused into the former reaction unit. Therefore, the effluents of aspartame were suitably resolved with ED.

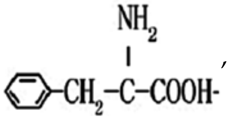
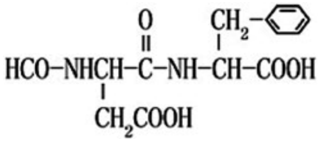
In this work, ED was proposed to separate salts from the aspartame effluents. The operating parameters for ED of aspartame were optimized, which was expected to be helpful for industrialization of ED for treating the effluents.

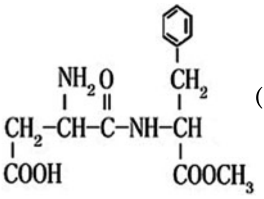
2. Experimental

2.1. Materials

The deionized water was produced by Milli-Q (Millipore, 18.2 M). The aspartame effluents were from a plant of Jiangsu, China. The content of sodium chloride in the effluents was 10%. The content of total organic carbon (TOC) in the effluents was 50 g/L. The organics in aspartame effluents are mainly consist of

HCOOH, CH_3OH, 
$NH_2CHCOOH(CH_2COOH)$, ,

,  and

 (aspartame).

2.2. ED setup

A laboratory-scale electrodialyzer with two compartments shown in Fig. 1 was employed in the research. As shown, the stack consisted of 10 cell pairs and each cell pair contained a CEM and AEM. The flow-pass length and width were 18 and 12 cm, respectively. The ion exchange membranes were from Shandong Tianwei Co. of China and the specific parameters were listed in Table 1. The distance between two membranes was 1 mm and the effluent flow was parallel to the membrane. A direct current power source was supplied to provide electrical energy with maximum output ability of 30 V and 10 A.

The ED was run at batch mode. The aspartame effluent of 500 ml and deionized water of 500 ml were pumped into desalination chamber and concentrate chamber, respectively. The electrode solutions were 800 ml of 0.06 mol/L Na_2SO_4. The ED tests were investigated under the voltage of 6 V (cell voltage 0.6 V) to 12 V (cell voltage 1.2 V) and flow rate of 10 L/h (velocity 0.35 cm/s) to 30 L/h (velocity 1.04 cm/s) at the room temperature (20°C). The experiment stopped, when the electric conductivity of the feed reduced to 2 mS/cm. Generally, the electrolytes in the effluent feed were removed by about 98% from the desalination chamber.

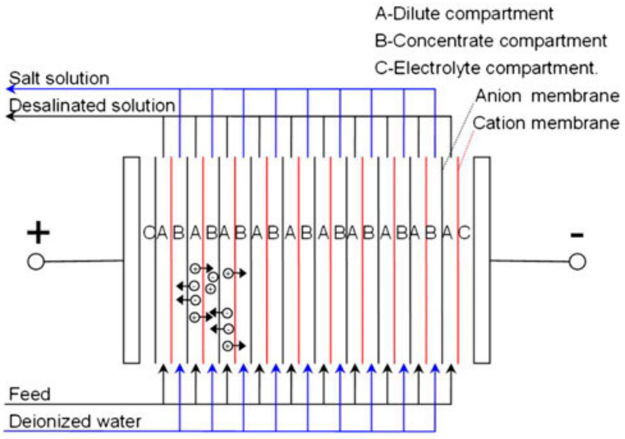


Fig. 1. Schematic representation of experimental electrodialyzer.

Table 1
The specific parameters of ion exchange membranes

Membrane properties	CEM	AEM
Water content (%)	55	65
Ion exchange capacity (mol/kg, dry)	1.4	1.6
Electric resistance (Ω/cm^2)	4	4
Transport number (–)	0.95	0.97
Thickness (mm)	0.25	0.3
Mechanical strength (MPa)	0.9	0.9

2.3. Analysis method

A TOC analyzer (TOC-VCPH, Shimadzu, Japan) was used to determine the content of organics in the effluent. All samples were neutralized to neutrality and diluted to a carbon concentration of less than 300 mg/L, with an automatic sampler; all the results can be obtained automatically.

The current efficiency (η) means the overall efficiency of current utilized for transport salts from the desalination compartment to the concentrate compartment, and can be calculated by Eq. (1) [14]

$$\eta = \frac{ZF\Delta n}{N \int I\, dt} \tag{1}$$

where Z is ionic valence number, F is Faraday's constant (96485 C/mol), Δn is mole number of salt removed from desalination compartment to concentrate compartment, I is the electric current (A), t is the operation time (s), and N is the number of cell pair.

The energy consumption (p) is defined as Eq. (2) [6]

$$p = \frac{\int EI\, dt}{\Delta m} \tag{2}$$

where E is the operation voltage (V) and Δm is the mass transferred during the ED run.

3. Results and discussion

3.1. Electric current variation during the ED

The electric current variation with time at the operation condition of 12 V, 20 L/h is presented in Fig. 2. At the beginning, the electric current raised rapidly from 0.7 to 1.2 A in 10 min and changed slightly in the following 40 min. After 50 min, the electric current declined sharply from 1.2 to 0.4 A till the experiment stop (total 65 min). The initial value of electric current was low because the solute in concentrate compartment was deionized water of low conductivity. And then, ions migrated from dilute compartment to concentrate compartment, which resulted in that the resistance in concentrate compartment continuously decreased. When the salts in dilute compartment were depleted, the total resistance increased rapidly and the current declined quickly. A reverse electric current was produced due to the salts diffusion from concentrate compartment to dilute compartment [15]. Thus, the current declined sharply. The experiment was terminated when the electric conductivity of the feed was below 2 ms/cm. The salts were removed from dilute compartment by over 98%.

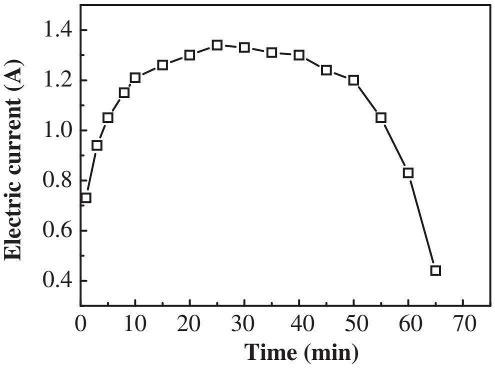


Fig. 2. The electric current variation with time at the flow rate of 20 L/h and voltage of 12 V.

As shown, in each batch experiment the current efficiency varied with time and the most efficient time for the ED process is about 40 min from 10 to 50 min.

3.2. The influence of voltage on ED process

The influence of voltage on energy consumption and operating time at the flow rate of 15 L/h is shown in Fig. 3. During the voltage range from 6 to 14 V, the energy consumption increased rapidly from 0.47 to 0.93 kWh/kg with the increase of voltage while the operating time decreased. Clearly, for the higher voltage, the energy consumption was higher but the operating time was shortened. As shown, the operation time under 6 V was 235 min, but it was shortened to 60 min under 12 V. It was because the low operate voltage made the low mass transfer force for the electromigration of ions in the ED process.

Fig. 4 shows the influence of voltage on current efficiency and organic mass loss at the flow rate of 15 L/h. With the increase of voltage, the current efficiency increased at the range of 6 to10 V and then decreased. The current efficiency had the maximum in

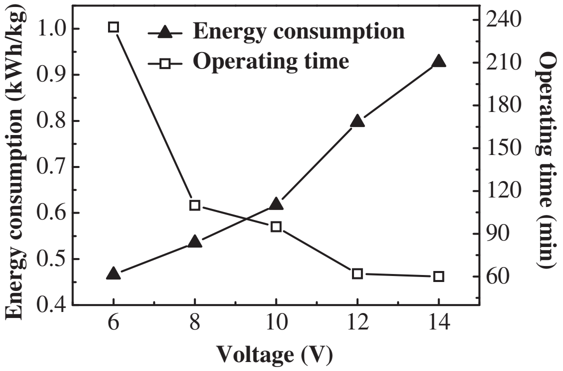


Fig. 3. The influence of voltage on energy consumption and operating time at the flow rate of 15 L/h.

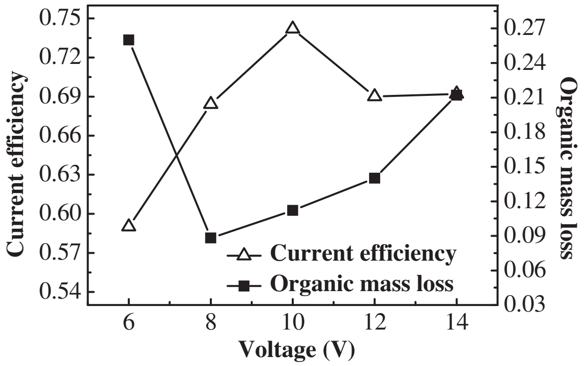


Fig. 4. The influence of voltage on current efficiency and organic mass loss at the flow rate of 15 L/h.

the range of 6–14 V where the ratio of energy used for electromigration arrived at the largest. The organic mass loss arrived at the maximum value at 6 V due to the diffusion effect of the longest operation time (about 2–4 times than others), and then decreased to the minimum value at 8 V. After 8 V it increased till 14 V because there was some charged organic mass. It's important to choose a appropriate operating condition to decrease organic loss.

3.3. The influence of flow rate on ED process

The influence of flow rate on energy consumption, operating time, current efficiency, and organic loss in the process of ED is presented in Figs. 5 and 6 at the voltage of 10 V. When the flow rate increased from 10 to 30 L/h, the energy consumption decreased from 0.75 to 0.57 kWh/kg and the operating time decreased from 110 to 80 min. Current efficiency and organic mass loss increased with the increasing flow rate. In general, an increase of flow rate could make the uniform distribution of the feed velocity and intensify the turbulence, which reduced the thickness of laminar boundary layer on the surface of ion exchange membrane. So, the concentration difference of mass between the bulk phase and the membrane surface became smaller, which resulted in that the limiting current density could become larger and the concentration polarization was weakened at the later stage of ED procedure. The mass transfer resistance became smaller and the velocity of electromigration increased. It made the energy consumption and the operation time decreased and the current efficiency increased. But the increase of flow rate could strengthen the diffusion and convection of organics, therefore, the organic loss increased. Diffusion of small organics through membrane is also investigated in this work.

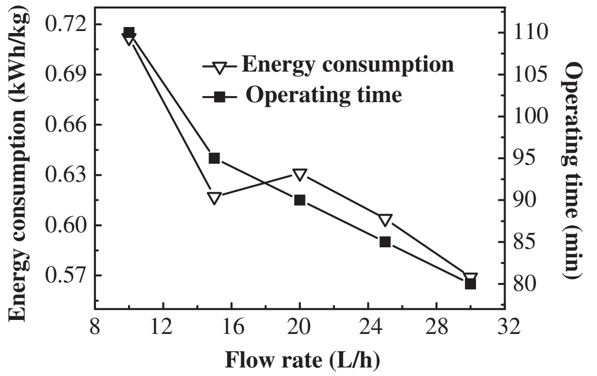


Fig. 5. The influence of flow rate on operating time and current efficiency at the voltage of 10 V.

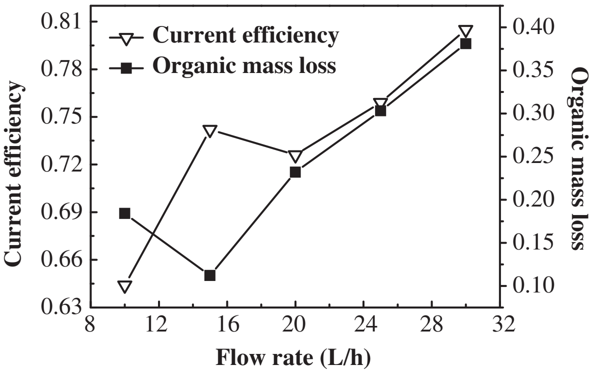
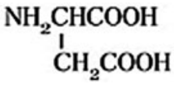
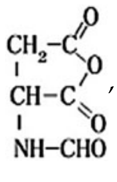
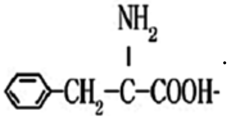
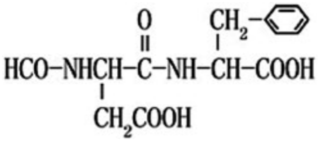
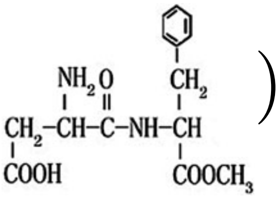


Fig. 6. The influence of flow rate on energy consumption and organic mass loss at 10 V.

3.4. Diffusion of small organics through membrane

Considering the concentration difference of organics between the dilute compartment and the concentrate compartment, diffusion of small organics could be an important reason for organic mass loss. Generally, ion exchange membranes can reject most organic substances of molecular weight larger than 250 Da. Thus, a diffusion experiment was carried out with the same apparatus without electric field. The organic mass loss ratios in 30 min were 7.6, 11.5, 15.5, and 24.3%, respectively when the flow rates were 10, 20, 25, and 30 L/h. These small organics mainly included HCOOH, CH_3OH,  $\begin{array}{l} NH_2CHCOOH \\ \;\;\;\;\;| \\ \;\;\;CH_2COOH \end{array}$, , . However, during ED process, part of the organic acids of high molecular weight more than 250 ( and ) could be ionized and transfer through the membranes, which led to a larger mass loss of organics than that of the former diffusion experiment without electric field. Therefore, diffusion of small organics and ionization of the part of organics might be a major reason for the mass loss of organics in ED of the aspartame effluents.

3.5. Optimization of voltage and flow rate

In order to obtain the suitable operating parameters, the ED tests with different voltages and flow rates were investigated. The influences of voltage and flow rate on operation time, current efficiency, energy consumption, and organic mass loss are shown in Figs. 7–10. At the voltages of 8, 10, 12, and 14 V, the cell voltages were 0.8, 1.0, 1.2, and 1.4 V, respectively. For the flow rates of 10, 15, 20, and 25 L/h, the velocity values of feed flows were 0.35, 0.52, 0.69, and 0.87 cm/s, respectively. As shown in Fig. 7, the operation time decreased with voltage at different flow rates and became shortened at the larger flow rate. Fig. 9 shows that the energy consumption increased with voltage but became low at high flow rate. From Figs. 8 and 10, the influences of voltage on current efficiency and organic mass loss were complicated. Based on the effects of voltage on four parameters, a suitable operation condition of 12 V and 20 L/h was selected. Under this operation condition, the current efficiency was 69.9%, the energy consumption was 0.786 kWh/kg, and the recovery of organic mass was 78% in a relative short operation time of 65 min.

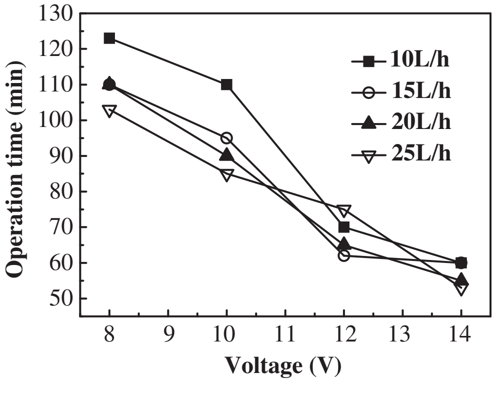


Fig. 7. The influence of voltage on operation time at different flow rate.

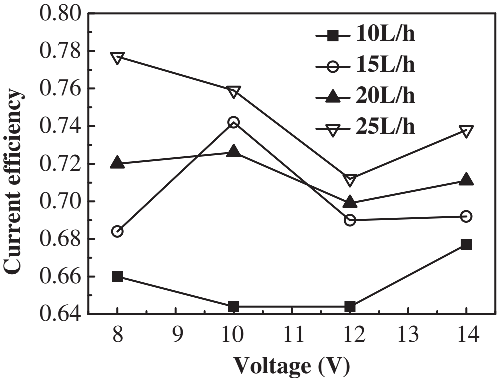


Fig. 8. The influence of voltage on current efficiency at different flow rate.

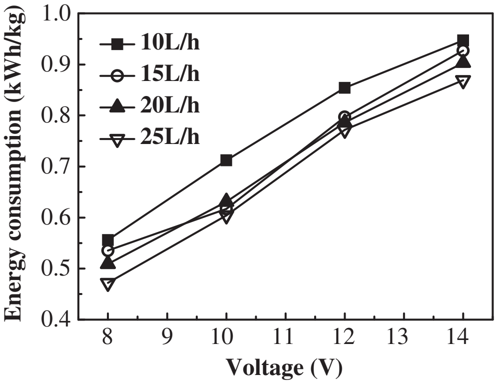


Fig. 9. The influence of voltage on energy consumption at different flow rate.

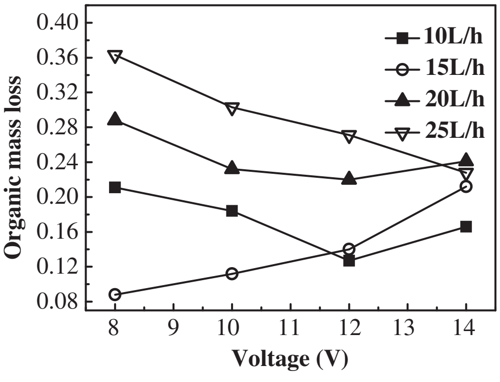


Fig. 10. The influence of voltage on organic mass loss at different flow rate.

4. Conclusions

Salt removal with ED from aspartame effluents was investigated. The influences of voltage and flow rate on operating time, current efficiency, energy consumption, and organic mass loss were studied. ED proved to be an effective method for removing the electrolytes by over 98%. With the increase of voltage, the operation time decreased but the energy consumption increased. With the increase of flow rate, the operation time and the energy consumption decreased but the organic mass loss increased due to diffusion of some small organics through ion exchange membranes. An optimum operating condition of 12 V and 20 L/h for the test electrodialyzer was obtained. Under this condition, the current efficiency was 69.9%, the energy consumption was 0.786 kWh/kg and the recovery of organics was 78% in 65 min. During the test cycle, the most efficient time for the ED process was about 40 min. The experimental results showed that the ED process was effective for dealing with aspartame effluents.

Acknowledgments

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