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Production of L-lysine from L-lysine monohydrochloride by bipolar membrane electrodialysis

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ABSTRACT

Bipolar membrane electrodialysis consisting of one bipolar membrane, one anion-exchange membrane and one cation-exchange membrane was performed to achieve the production of L-lysine from L-lysine (L-Lys) monohydrochloride. Several experimental parameters including the operation voltage, the initial L-Lys·HCl concentration and operation temperature were compared and discussed. The Cl^- removal ratio, the electric conductivity, the pH in various compartments, the current efficiency (CE) and the energy consumption (EC) were presented and analyzed respectively. When the initial concentration of L-Lys·HCl is 0.6 mol/L and the constant voltage is 40 V, the removal ratio of Cl^- reached 86.6%, the CE 24% and the EC 28.2 kW h/kg. Elevating the operation temperature can promote the electrodialysis process slightly.

Keywords: Bipolar membrane; Electrodialysis; L-Lysine; L-Lysine monohydrochloride

1. Introduction

L-Lysine (L-Lys) is an essential amino acid generally considered as a fundamental substance of human body, which has a wide range of application in pharmaceuticals, food and feed industry [1]. L-Lys is necessary for physical growth and skeletal development especially for children [2]. Concretely, it is beneficial to absorb calcium, keep nitrogen balance and ensure lean body mass [3]. Commercial L-Lys is usually produced as L-lysine monohydrochloride (L-Lys·HCl), since L-Lys is difficult to preserve and easy to absorb moisture as a natural protein. However, L-Lys·HCl is not commonly suitable for eating directly, and Cl^- needs to be removed in order to gain the L-Lys of high medical and edible value [4].

In our earlier work, Cl^- was removed by traditional ion exchange membrane electrodialysis [5], where the final Cl^- removal ratio reached about 73.1%—a little lower for practical application. Then ion substitution electrodialysis was tried for production of L-Lys, and results shown a much higher Cl^- removal ratio, 95.6% [6]. However, in order to achieve the highest Cl^- removal ratio, a little amount of buffer solution was added to the feed liquid in ion substitution electrodialysis, which may lead to a slight impurity of the as-produced L-Lys. Therefore, another novel electrodialysis method, namely, bipolar membrane electrodialysis, is expected to be developed to achieve purer L-Lys from L-Lys·HCl.

Bipolar membrane is a special type of ion-exchange membrane and its well-known function is to split water into OH^- and H^+ in a direct current field [7–9]. Bipolar membrane electrodialysis has been used

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in several fields and many literature papers have been devoted to it [10,11], such as treating waste water containing ammonium nitrate [12–14], desalination of fruit juice [15,16], production of *citric* acid [17,18], lactic acid [19], salicylic acid [20], and so on. As for its application in amino acid field, Yu et al. [21] have reported a bipolar membrane electrodialysis process to regenerate sodium *p*-toluenesulfonate (or sodium 4-methylbenzenesulfonate) in the production of D-(−)-*p*-hydroxyphenylglycine (or R-α-amino-4-hydroxy-benzeneacetic acid). The conversion of pure sodium *p*-toluenesulfonate solution and actual feed solution that contains a small amount of R-α-amino-4-hydroxy-benzeneacetic acid besides sodium *p*-toluenesulfonate was compared and a new processing route was proposed. The removal ratio of Na^+ was typically around 80% with an average current efficiency (CE) of 20–50%. In addition, Zhang et al. [22] also reported the recovery of glutamic acid by two electrodialysis processes: two-compartment bipolar membrane electrodialysis and modified traditional electrodialysis. Eliseeva et al. [23] presented the recovery and concentration of basic amino acids by electrodialysis with bipolar membranes. However, till now, few literature papers have reported the production of L-Lys from L-Lys·HCl by bipolar membrane electrodialysis.

Consequently, the production of L-Lys from L-Lys·HCl by bipolar membrane electrodialysis was evaluated in this work. Effects of several parameters including the operation voltage, initial L-Lys·HCl concentration and the operation temperature are presented one by one. The process performance parameters of the whole operation system, such as the electric conductivity, pH in various compartments, removal ratio of Cl^-, the current efficiency (CE) and the energy consumption (EC) are also studied.

2. Experimental

2.1. Materials

A bipolar membrane (BPM, Neosepta BP-1, Tokuyama Soda Inc., Japan), a heterogeneous cation-exchange membrane (CEM) and an anion-exchange membrane (AEM) (Shanghai Shanghua Water Treatment Material Co. Ltd., China) were used during all experiments, and their main characteristics are listed in Tables 1 and 2. In order to avoid the perturbation of the impurities in membranes, the purchased membranes (BPM, CEM and AEM) were pretreated with 1 mol/L HCl and NaOH for 4 h alternately and then washed with deionized water before electrodialysis.

Table 1
Main characteristics of the ion-exchange membrane

Membrane	Cation-exchange membrane	Anion-exchange membrane
Water content (%)	35–55	30–45
Exchange capacity (mol/kg)	$\geqslant 2.0$	$\geqslant 1.8$
Resistance ($\Omega\,cm^2$)	11	12
Thickness (mm)	0.42	0.42
Transport number (%)	90	89

L-Lysine monohydrochloride, sodium hydroxide, sodium nitrate and sodium sulphate, etc. of AR grade were commercially obtained and used without further purification. Deionized water was used thoroughly.

2.2. Methods

In bipolar membrane electrodialysis, the laboratory-scale electrodialyzer consisted of one anode, one cathode, one piece of BPM, one piece of AEM as well as CEM inserted between them with an inter-membrane distance of 8 mm. There were four compartments with a volume of 25 mL from left to right: compartment 1 containing 0.5 mol/L of Na_2SO_4 solution, compartment 2 containing 0.3 mol/L of $NaNO_3$ solution, compartment 3 comprised of L-Lys·HCl solution of certain concentration, and compartment 4 comprised of 0.5 mol/L Na_2SO_4 solution. All the above compartments were connected to a separate external 500 mL beaker, allowing for continuous recirculation by four submerged pumps (AT-301, ATMAN) with a flow rate of 15 L/h. The size of ion-exchange membrane was 5 cm × 5 cm with an effective area of 7.06 cm^2. DC power (DF1731SLL3A, Zhongce Electronics Co. Ltd., China) was used to apply constant potential across the electrodes. The pH value of the solution was regularly monitored by an acidity meter (PHS-2C, Shanghai Hongyi Instrumentation Co. Ltd., China). The schematic diagram of the experimental apparatus is shown in Fig. 1, among which the bipolar membrane electrodialysis stack is illustrated in Fig. 2.

The whole electrodialysis process mainly involves the following four stages: (i) water was split at the interface layer of the BPM and dissociated into OH^- and H^+, and then they transfer across the anion exchange layer and cation exchange layer of the BPM, getting into compartments 3 and 4 respectively; (ii) substitution of Cl^- by OH^- in compartment 3; (iii) migration of Cl^- from compartment 3 to 2 through AEM; (iv) generation of H^+ by water splitting at the

Table 2
Main characteristics of the bipolar membrane

Membrane	Parameter	Value
Bipolar membrane (Neosepta, BPM-1)	Type, group	Composition of the cationic and anionic membranes
	Thickness (mm)	0.2
	Voltage drop (V)	1.2–2.2
	Water slitting efficiency	>0.98
	Burst strength (M Pa)	0.4–0.7

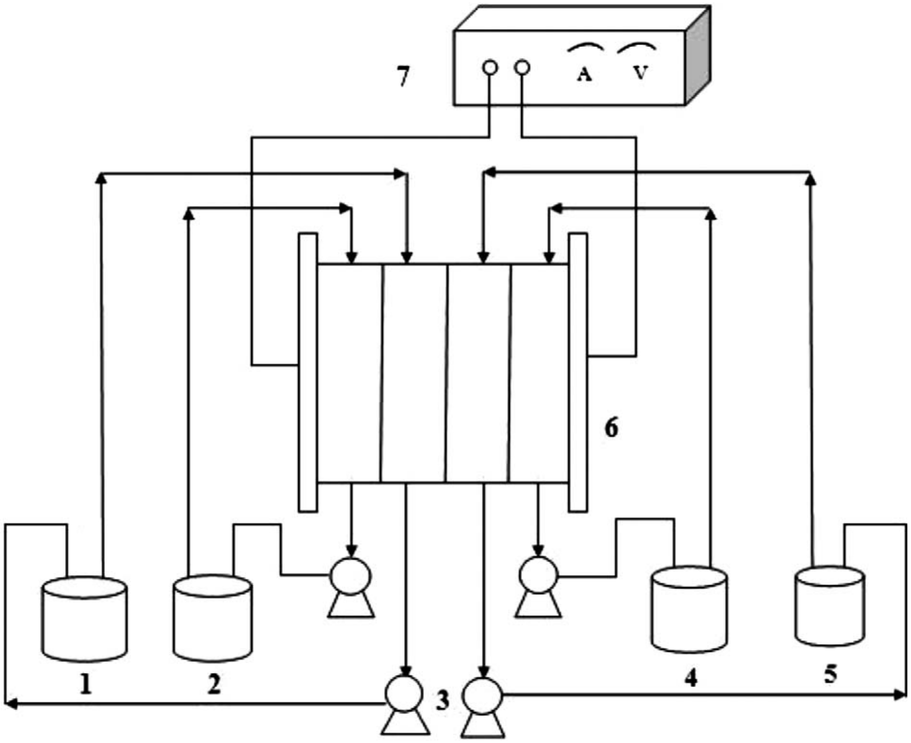

Fig. 1. Schematic diagram of the experimental apparatus (1, tank for concentrating recirculation; 2, tank for anolyte recirculation; 3, pump for recirculation; 4, tank for catholyte recirculation; 5, tank for desalting recirculation; 6, bipolar membrane electrodialysis stack; 7, DC power).

anode and its migration through CEM and subsequent formation of HCl in compartment 2.

All experiments were repeated three times and their mean value was taken as the final result. The estimated error is about ±5%.

2.3. Calculations

The removal ratio of Cl^- is one of the most important parameters to examine the practical feasibility in this work. In bipolar membrane electrodialysis process, almost all Cl^- ions transfer across the AEM and concentrate in compartment 2, and no Cl_2 can be measured in compartment 1. Thus the removal ratio of Cl^- can be calculated as R_m in Eq. (1):

$$R_m(\%) = \frac{n(t)}{n_S(0)} \times 100 \tag{1}$$

Where $n(t)$ is the mole amount of Cl^- titrated by $AgNO_3$ with K_2CrO_3 as an indicator in compartment 2 (mol); $n_s(0)$ the mole amount of initial L-Lys·HCl (mol) in compartment 3.

EC and CE are also important parameters of any electrochemical process for their practical application. The EC (kW h/kg of L-Lys recovered) is defined as Eq. (2):

$$\mathrm{EC(kWh/kg)} = \frac{\int_0^t IUdt}{m} \tag{2}$$

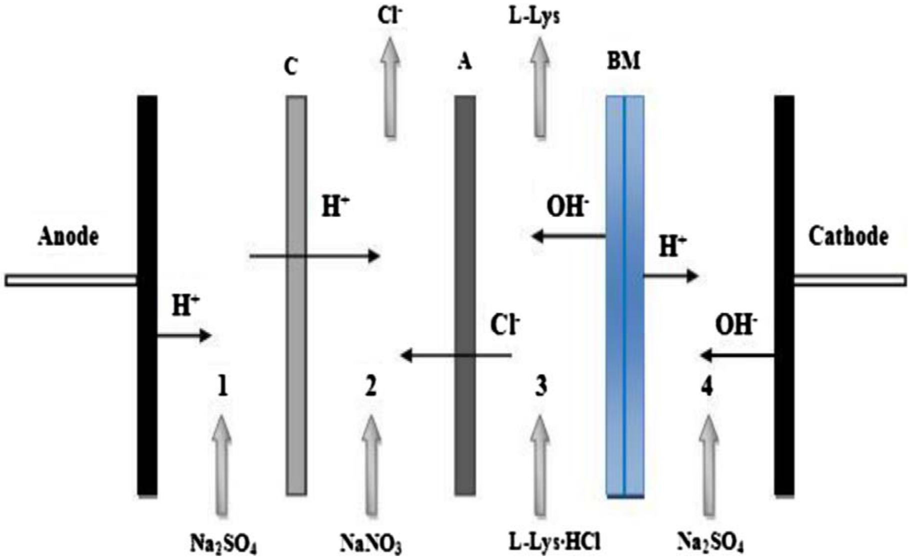

Fig. 2. Schematic of bipolar membrane electrodialysis stack.

Where m is the weight of produced L-Lys (kg); I the current (A); t the time (h) and U the voltage (V).

The overall CE is defined as Eq. (3):

$$\mathrm{CE}\ (\%) = \frac{zF\Delta n}{\int_t^0 I dt} \times 100 \qquad (3)$$

where Δn is the mole amount of recovered L-Lys (mol); z the ionic valence and F the Faraday constant (96,500 C/mol).

3. Results and discussion

3.1. Effect of operation voltage

Experiments for production of L-Lys by bipolar membrane electrodialysis were carried out at different applied potentials ranging between 20 and 50 V with an initial L-Lys·HCl concentration of 0.2 mol/L. The results of the operation process including conductivity, removal ratio of Cl^-, pH in various compartments, CE and EC are shown in Figs. 3–8. In order to make the figures in this work seen more clearly, plot curves for approximation of experimental data on Figs. 3–11 have been considered. The approximation plots not only approximate every experimentally obtained dot, but also depict basic law's on how experimental data change.

3.1.1. Electric conductivity and removal ratio of Cl^-

As mentioned above, when an electric field is applied, OH^- ion dissociated by bipolar membrane substitutes Cl^- in compartment 3, and then Cl^- ion migrates from compartment 3 to 2 through the AEM, thus the free L-Lys forms in compartment 3. Variations of electric conductivity in compartment 3 with time during such an ion substitution process at various operation voltages were depicted in Fig. 3.

At the beginning of bipolar membrane electrodialysis, water dissociation occurs at the interface of the bipolar membrane and current is sustained by the migration of H^+ and OH^- ions to the respective com-

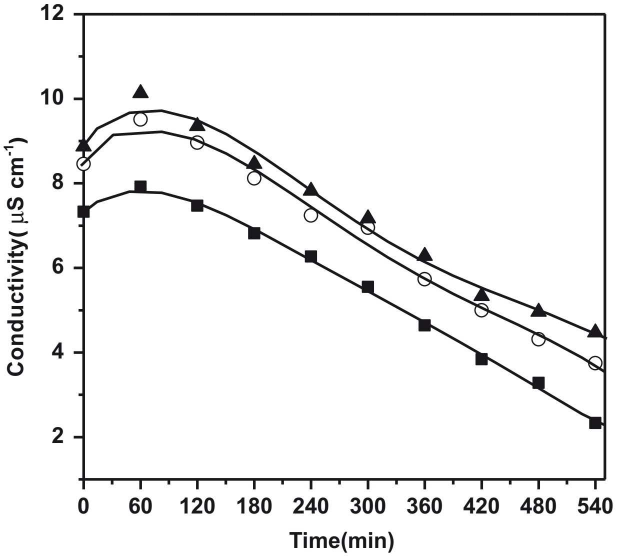

Fig. 3. Variation of electric conductivity in compartment 3 with time at different voltages (–■–, 20 V; –○–, 40 V; –▲– 50 V).

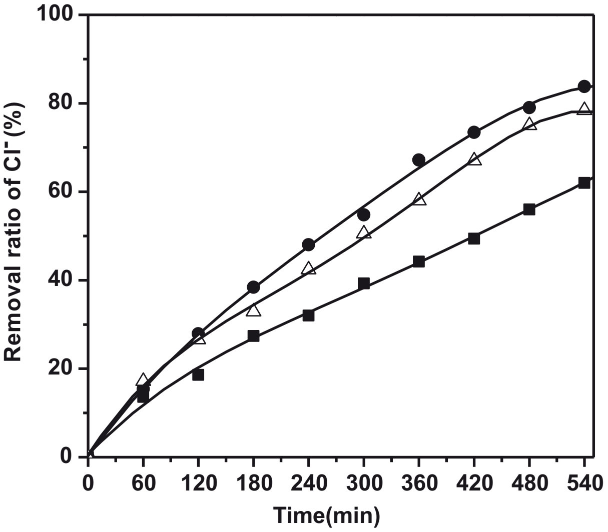

Fig. 4. Variation of removal ratio of Cl^- with time at different voltages (–■–, 20 V; –●–, 40 V; –△–, 50 V).

partment under the drive of the electric field force. As shown in Fig. 3, initial electric conductivity is slightly low and then it increases to some extent. After attaining the maximum value, it decreases with time gradually. During the electrochemical process, the applied voltage is responsible for the formation or transportation of OH^-, H^+ and Cl^-. The H^+ and OH^- produced by water dissociation raise the ionic amount in compartment 3 and 4, and the transportation of Cl^- to compartment 2 also increases the ion concentration. Therefore, the increase in ionic concentration in compartment 2, 3 and 4 leads to the rising electric conductivity inevitably. However, after reaching the maximum conductivity, the electrolyte (L-Lys·HCl) concentration in compartment 3 is lowered, which causes an increase in the overall electrical resistance, and thus the conductivity decreased gradually. In fact, such an experimental trend can be observed during all electrodialysis procedures, which is also in accordance with the results reported by Kumar et al. [24].

Removal ratios of Cl^- at different applied voltages are presented in Fig. 4. Obviously, the removal ratio of Cl^- first sharply increases with time and then rises a little slowly. When the voltage increases from 20 to 40 V, the final removal ratio of Cl^- also increases from 62.0% to 83.8%. However, this tendency does not exist anymore when a higher voltage of 50 V is applied. And it can be seen from Fig. 4 that the removal ratio of Cl^- at 50 V is 78.4%, which is lower than that at 40 V. The transference of Cl^- ions is probably limited because of its competitive transfer with OH^- at much higher voltage. Therefore, it is not favorable for choosing too high voltage for this bipolar membrane electrodialysis.

In addition, the pH value of the solution in each compartment also changes with the electrodialysis time as shown in Fig. 5, with an applied voltage of 40 V and an initial L-Lys·HCl concentration of 0.2 mol/L respectively. When the electrodialysis process begins, the electrode reaction occurs as Eqs. (4) and (5) shown below:

$$\text{Anode reaction: } 2OH^- - 2e \rightarrow H_2O + \frac{1}{2}O_2 \uparrow \qquad (4)$$

$$\text{Cathode reaction: } 2H^+ + 2e \rightarrow H_2 \uparrow \qquad (5)$$

Initially, the anolyte in compartment 1 offers nearly pH 7.4 because Na_2SO_4 solution is neutral, and afterwards it decreases sharply due to formation of H^+ by water splitting. The pH of the catholyte in compartment 4 is initially 6.8 and then it goes up slightly. But after a time of electrodialysis, the pH of catholyte sustains around 7.0. The reason may be considered that the H^+ splitted by bipolar membrane is neutralized by the electrolyzed OH^- in the cathode compartment. The pH in compartment 3 is firstly 6.7 and finally 10.2, close to be 9.74. When the electrodialysis process begins, OH^- is produced by the bipolar membrane in compartment 3 and the pH is raised. The OH^- combines with L-Lys^+, forming the free L-Lys accordingly. The isoelectric point of free L-Lys is 9.74, the pH in compartment 3 approaches such a value as

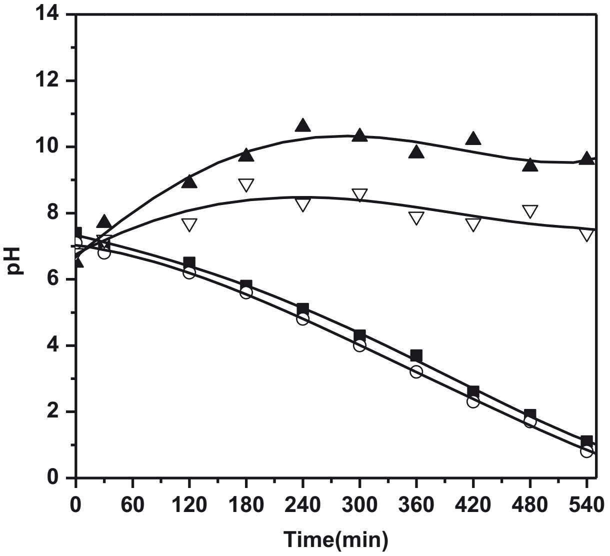

Fig. 5. Variation of pH value in different compartments with time (–■–, compartment 1; –○–, compartment 2; –▲–, compartment 3; –▽–, compartment 4).

a result. Besides, the pH in compartment 2 decreases with time on account of migration of H^+ from compartment 1. As a matter of fact, a similar relationship between pH in each compartment and electrodialysis time can be always observed during all of our experiments, and explanations are also analogous.

3.1.2. EC and CE

As shown in Figs. 6 and 7, as a whole, the CE decreases with the removal ratio of Cl^- and the EC increases with the removal ratio of Cl^- at a fixed voltage, which conforms to the typical trends reported by Xu et al. [18]. However, for the CE at the same removal ratio of Cl^-, its order follows: 40 > 50 > 20 V; while the order of EC at different voltages is 50 > 40 > 20 V. It can be explained from such aspects: On one hand, just as seen from Eq. (2) and Fig. 7, decreasing the voltage to 20 V can be of benefit to EC. Nevertheless, when the applied voltage is lowered (20 V), the electronic field force is weakened and the water dissociation at the interface of the bipolar membrane is much less, which induces the decrease in H^+ and OH^- ion concentrations. Thus, ionic transportation is lowered and the CE decreases incontrovertibly. On the other hand, water dissociation in the intermediate layer of the BPM always is enhanced with the increase in applied voltage, and electric current is due to the splitted H^+ and OH^-, which results in a higher CE. However, the following four reasons may lead to the decrease in CE and the increase in EC at the highest voltage (50 V): firstly, the transference of Cl^- ions is limited because of its competitive transfer with OH^- at too high voltage, leading to a lower CE. Secondly, when the applied voltage is promoted to a higher level as 50 V, the amount of H^+ and OH^- ions which transport out of BPM is larger than that into BPM and the speed of rehydrate is limited. And the CE decreases and the EC increases accordingly. It is well known that the current density is about 103 A/m^2 when above phenomenon occurs. As a matter of fact, the current density at 50 V is exactly about 5.19×10^3 A/m^2 based on our experimental data, which verifies our deduction. Thirdly, the water dissociation at the surface of CEM and AEM leads to the quick increase in membrane stack resistance [22]. Finally, heat emission transformed from part of the electric energy may occur at a higher voltage [25], causing the CE and EC decrease and increase respectively. Therefore, whether the CE/EC is high/low or low/high is dependent on which one is preponderant among the advantageous and disadvantageous aspects. As for the voltage of 50 V, the disadvantageous aspects apparently gain the upper hand, so the EC is higher and the CE is lower than 20 V. In conclusion, a lower or higher applied voltage is both disadvantageous to the electrodialysis process considering three parameters removal ratio of Cl^-, CE and EC.

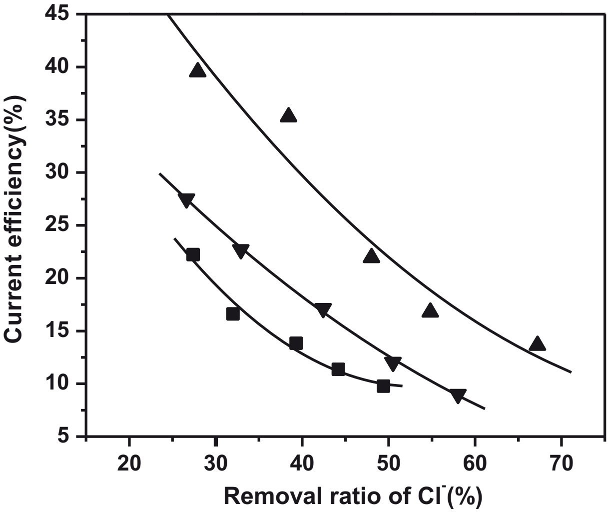

Fig. 6. Variation of CE with removal ratio of Cl^- at different voltages (–■–, 20 V; –▲–, 40 V; –▼– 50 V).

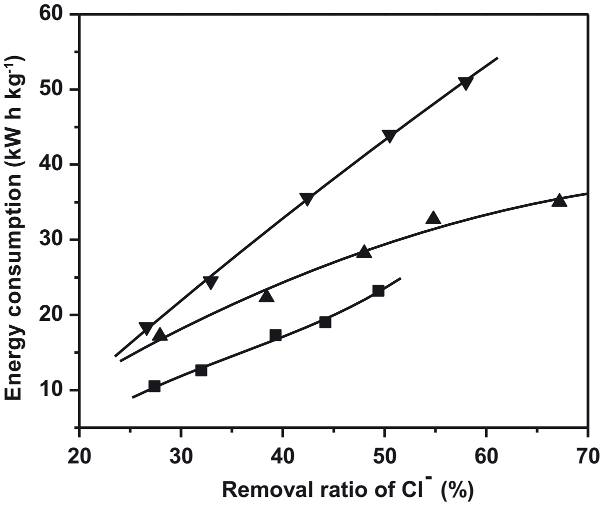

Fig. 7. Variation of EC with removal ratio of Cl^- at different voltages (–■–, 20 V; –▲–, 40 V; –▼– 50 V).

As for the low CE in production of L-lysine, according to Zhang et al. [22], their CE during recovery of glutamic acid from isoelectric supernatant using eletrodialysis with Neosepta BP-1 also reached as low as about 25%. As a result, our experimental results are in agreement with literature data. However, the used bipolar membrane (Neosepta BP-1) is well known for its high CE even in salt solutions of 1–4 mol/L. As a consequence, a discrepancy between the as-imagined high efficiency and the actual low value seems puzzling. In fact, such a discrepancy may be explained as follows: in order to obtain the highest Cl^- removal ratio as far as possible, the electrodialysis time in our work was elongated to be 540 min on account of L-lysine dissociation equilibrium, leading to a sharp lower CE undoubtedly. Whereas, for general inorganic salt solutions separation, the ionic transfer is simpler than that during L-lysine production process. Therefore, the electrodialysis time is much shorter, and the CE is higher for inorganic salt system as a result.

3.2. Effect of initial L-Lys·HCl concentration

In this part, the effect of initial L-Lys·HCl concentration was investigated with an applied voltage of 40 V. Various electrodialysis experiments were carried out at the L-Lys·HCl concentrations of 0.4, 0.6 and 0.8 mol/L. The obtained removal ratio of Cl^-, electric conductivity, EC and CE were shown in Figs. 8–11.

The initial concentration of L-Lys·HCl also affects electric conductivity and removal ratio of Cl^- during ionic substitution. Figs. 8 and 9 present the variation of conductivity and removal ratio of Cl^- with time respectively. At a first glance, the experimental trends of conductivity and Cl^- removal ratio with time are similar to those at different voltages. That is to say, conductivity first increases with electrodialysis time, then reaches a maximum and finally falls to some extent at different initial L-Lys·HCl concentrations. But there is an apparent distinction for conductivity value between Figs. 3 and 8, that is, the conductivity in the latter figure is generally larger than that in the former one. The reason is as below: the most remarkable influence factor of conductivity is ionic amount. In Fig. 8, the solution concentrations in compartment 3 are 0.4, 0.6 and 0.8 mol/L respectively, which are all larger than that (0.2 mol/L) in Fig. 3, resulting in the larger conductivity surely.

As for the removal ratio of Cl^-, it increases with time, reaches a maximum and then keeps almost unchanged at a fixed initial concentration of L-Lys·HCl. The final removal ratio at 0.4 mol/L is lower than that at 0.6 mol/L. The reason considered is that lower concentration implies fewer amounts of ions which can migrate across the ion-exchange membrane, leading to a lower final Cl^- removal ratio. Lack of ion causes increase in electrical resistance undoubtedly, which leads to the increase in EC and reduction in CE as shown in Figs. 10 and 11. However, when the initial concentration of L-Lys·HCl is high (e.g. 0.8 mol/L), the Cl^- removal ratio decreases contrarily. It can be also found from Figs. 10 and 11 that when the initial concentration increases from 0.6 to 0.8 mol/L, the EC and CE increases and falls, respectively. The reasons are considered as below: First, higher concentration of electrolyte means a higher osmotic pressure, which results in a decline in water supply in the mesosphere of the bipolar membrane, and so water splitting is weakened; second, the time taken to reach the same Cl^- removal ratio for a higher concentration is longer than that for a lower one, thus, too high initial concen-

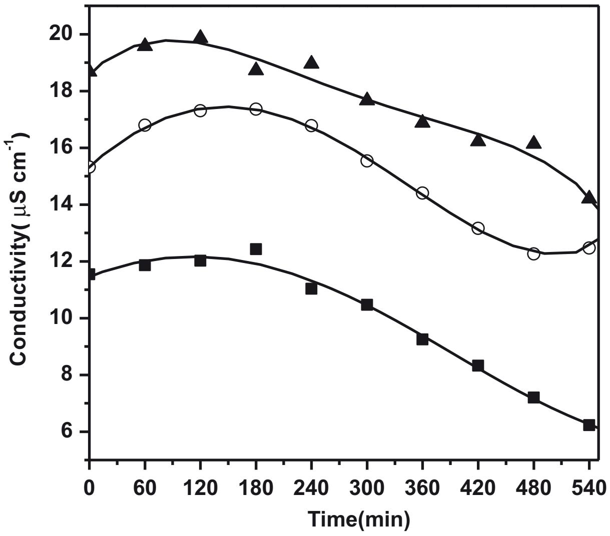

Fig. 8. Variation of conductivity with time at different initial L–Lys·HCl concentrations (–■–, 0.4 mol/L; –○–, 0.6 mol/L; –▲–, 0.8 mol/L).

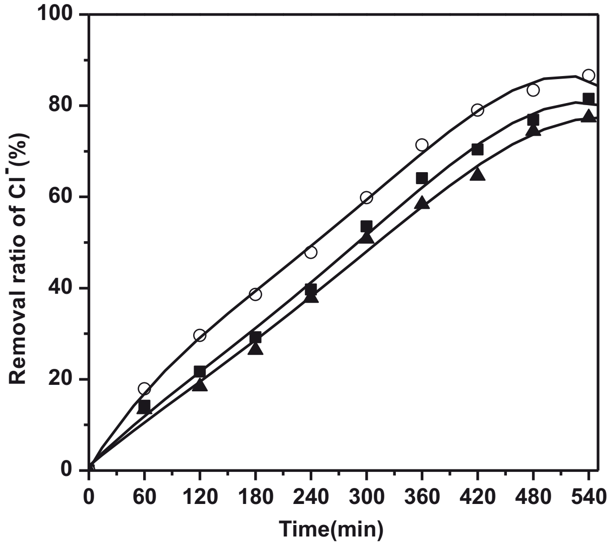

Fig. 9. Variation of removal ratio of Cl^- with time at different initial L-Lys·HCl concentrations (–■–, 0.4 mol/L; –○–, 0.6 mol/L; –▲–, 0.8 mol/L).

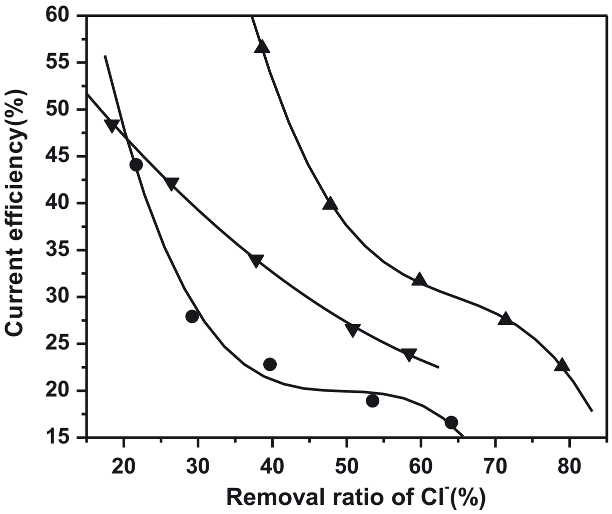

Fig. 10. Variation of CE with removal ratio of Cl$^-$ at different initial L-Lys·HCl concentrations (–●– 0.4 mol/L; –▲–, 0.6 mol/L; –▼–, 0.8 mol/L).

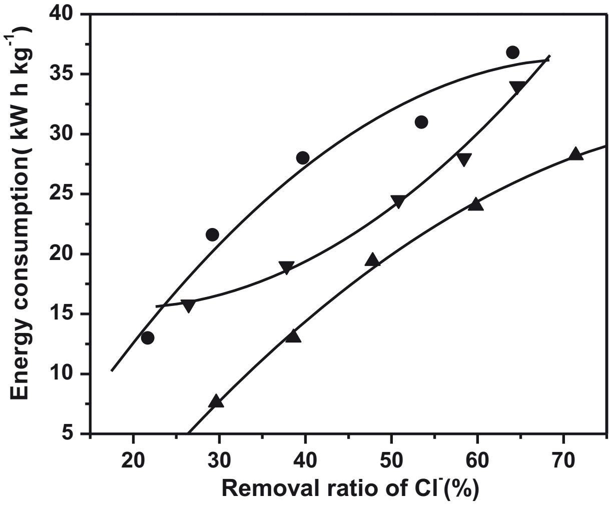

Fig. 11. Variation of EC with removal ratio of Cl$^-$ at different initial L-Lys·HCl concentrations (–●–, 0.4 mol/L; –▲–, 0.6 mol/L; –▼–, 0.8 mol/L).

tration is also disadvantageous to electrodialysis. All the above factors result in lower CE and higher EC at the highest initial L-Lys·HCl concentration. Thus, a suitable initial L-Lys·HCl concentration should be chosen for such a BPM electrodialysis.

3.4. Effect of operation temperature

Ionic transfer is affected by the operation temperature during BPM electrodialysis. However, there is little attention paid to such an aspect up to now. In order to study the effect of operation temperature, several experiments were carried out at 20, 40 and 60 °C, respectively. Here, the initial concentration of L-Lys·HCl was 0.6 mol/L and the applied voltage was 40 V. The comparisons of operation process performance parameters were also listed in Table 3.

Obviously, the process of BPM electrodialysis can be slightly affected by the operation temperature as indicated in Table 3. A higher operation temperature can improve the BPM electrodialysis process, such as increasing the Cl$^-$ removal ratio, consuming less energy and obtaining higher CE. The reason for these results is that the migration of ions through the ion-exchange membranes and BPM is faster at a higher temperature, which gives rise to the decrease in the electrical resistance and then brings out a slightly higher CE and lower EC.

4. Conclusions

A bipolar membrane electrodialysis process with four compartments was proved to be successful in the production of L-Lys from L-Lys·HCl. Effects of such parameters, such as the applied voltage, initial L-Lys·HCl concentration and the temperature on the operation process performance were studied. The following conclusions were drawn:

- The operation voltage can largely affect the Cl$^-$ removal ratio, CE and EC. When the applied voltage is 40 V with a constant initial L-Lys·HCl concentration of 0.2 mol/L, the removal ratio of Cl$^-$ is the highest, 83.8%. A lower or higher applied voltage is both disadvantageous to the electrodialysis process.
- At a constant operation voltage 40 V, the initial L-Lys·HCl concentration can also affect the electrodialysis process. When the initial L-Lys·HCl concen-

Table 3
Comparison of operation process parameters at different temperatures

Temperature (°C)	Time (h)	Cl$^-$ removal ratio (%)	Current efficiency (%)	Energy consumption (kw h/kg)
20	9	86.6	24.0	28.2
40	9	88.2	26.2	27.2
60	9	89.8	27.4	25.3

tration is 0.6 mol/L, the Cl^- removal ratio is the highest (86.6%), the CE is 24% and the EC is 28.2 kW h/kg.

- Increasing the operation temperature can promote the electrodialysis process slightly, such as increasing the Cl^- removal ratio, consuming less energy and obtaining higher CE.

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