



The removal of aniline from wastewater by electrodialysis in the presence of hydrochloric acid

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ABSTRACT

A novel method has been proposed for the treatment of aniline wastewater using electrodialysis (ED) apparatus to remove the aniline ions formed in the presence of hydrochloric acid (HCl). The optimum operating parameters, including HCl concentration, initial aniline concentration, applied voltage, and flow rate were determined via a series of experiments considering energy consumption and the aniline removal efficiency. Under the selected conditions, about 99% aniline removal was obtained within 120 min, and the aniline concentration can be reduced from 500 to 4 mg/L, which suggest the city standards of pollution discharge (namely, 5 mg/L) can be met. Further, the limiting current density was determined and the total energy consumption was calculated. It was proved that the ED apparatus can be used to remove aniline from wastewater effectively at low cost.

Keywords: Aniline; Wastewater treatment; Electrodialysis; Heterogeneous ion-exchange membrane

1. Introduction

Aniline is one of the most important industrially produced amines and widely used for the manufacture of polyurethanes, rubber, dyestuffs, and pesticides [1]. Aniline wastewater is mainly formed from the manufacture of dyes, chemical industry, and pharmaceutical manufacturing industries. Currently, the amount of aniline produced is over 80,000 tons per year in China. Aniline wastewater, which is discharged into the pipes, could easily have chemical

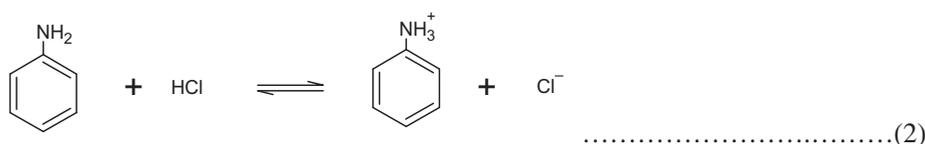
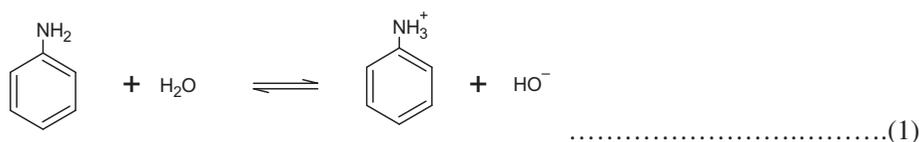
reactions with other substances, forming ammonia, which increases the amount of organic pollution. High concentrations of aniline contaminated water could poison other aquatic organisms; therefore, aniline should be removed from the wastewater before disposal.

Many techniques, including activated carbon adsorption [2,3], solvent extraction, distillation, reverse osmosis [4], catalytic wet oxidation [5], electrochemical oxidation, and biodegradation [6], have been adopted for the removal of aniline in the pollutants. However, these techniques are sometimes restricted because of economical or technical constraints. Activated carbon

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adsorption is very difficult to carry out because of the regeneration costs. Traditional solvent extraction usually produces secondary liquid organic waste, which may cause environmental problems. Distillation columns require a large amount of energy to vaporize the low-boiling material. Biodegradation is not suitable for the wastewater with high concentration of aniline. This process can decompose or remove aniline in wastewater to some extent, but aniline is very difficult to decompose completely. The other methods are not suitable to be applied to the practice due to the high costs and the fact that the pollutants cannot be

not effective in the absence of HCl. Aniline is a weak base and dissolves in water up to 3.5%. In neutral aqueous environments, aniline mainly presents as $C_6H_5-NH_2$, and partially as $C_6H_5-NH_3^+$, according to the dissociation reaction ($pK_b=9.40$, $K_b=3.98 \times 10^{-10}$) as shown in Eq. (1). It could have a reaction with acid. In this study, $C_6H_5-NH_3^+$ and Cl^- ions formed when the aniline solution in the presence of HCl as in Eq. (2). So it was presumed that aniline ions, which are formed in the presence of HCl, could possibly be diluted and concentrated using electro dialyzer.



completely degraded. Therefore, there is a need to develop a new method, which can be used to remove aniline economically and successfully.

Electrodialysis (ED) is a membrane separation process based on the selective migration of aqueous ions through ion-exchange membranes as a result of an electrical driving force [7]. This process has been used to deal with the problems in desalination of salted waters [8], wastewater minimization [9], separation of electrolytes and non-electrolytes, products of acids and alkalis from their salts [10], concentration of dilute solutions, and synthesis of ionic liquid [11].

Nicoletta Boniardi et al. [12] reported that the concentration process of sodium lactate solutions was analyzed using a two-compartment electro dialytic unit equipped with commercial membranes. The influence of several operating parameters on the overall process performance was investigated. For instance, it came out that at low current density values such membranes are able to operate a perfect separation of opposite charges, resulting in a good process performance. Moreover, water transport by electro-osmosis has to be considered, hence lowering the stack performance.

In our study, ED process is used in aniline wastewater treatment. It is noticed that aniline removal is

2. Experimental

2.1. ED setup and materials

The experimental electrically driven membrane separation apparatus and heterogeneous ion-exchange membrane are supplied by Zhejiang Qianqiu Group Co Ltd. The apparatus is comprised of an electro dialyzer, a DC power supply unit, pumps, rotameters, and tanks. The electrodes are made of high-quality

Table 1
Characteristics of applied membranes

Kind of ion-exchange membrane	Heterogeneous ion-exchange membrane	
	CEM	AEM
Resistance of membrane surface ($\Omega \text{ cm}^2$)	≤ 10	≤ 12
Thickness (mm)	0.6 ± 0.05	0.6 ± 0.05
Water content (%)	≤ 50	≤ 40
Exchange capacity (mol/kg) (dry)	≥ 2.0	≥ 1.8
Selective penetration (%)	≥ 92	≥ 94
Permeability of water (ml/h cm^2 MPa)	≤ 0.1	≤ 0.1

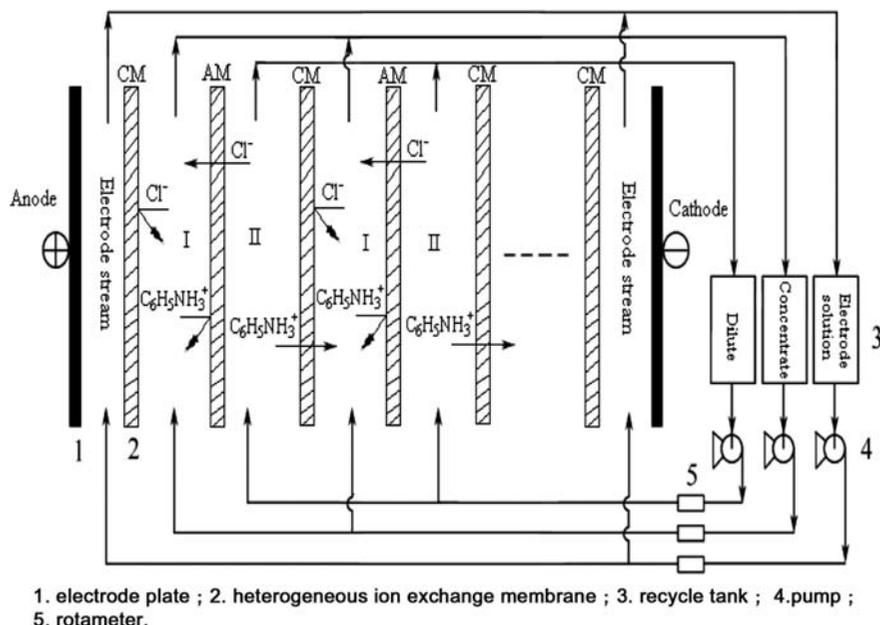


Fig. 1. Schematic illustration of the concentration and removal of aniline using electrodedialyzer.

titanium substrate; and the surface is coated with metal ruthenium, which has good electrochemical properties, corrosion resistance and some other characteristics, one on either side parallel to the membrane. The cation-exchange membranes (CEMs) and anion-exchange membranes (AEMs) were alternately arranged in the electrodedialyzer. The laboratory ED unit with 40 cell pairs was built for studies on the treatment of aniline-containing wastewaters in this work. The dimensional size of the clapboard is $200 \times 120 \times 1$ mm, and the effective membrane area of each membrane is $10,824 \text{ mm}^2$. Three tanks are used for the dilute solution, concentrate solution, and electrode solution, respectively. Table 1 shows the properties of the heterogeneous ion-exchange membrane.

A schematic diagram, which simply describes the principles of aniline desalination and concentration using electrodedialyzer, is shown in Fig. 1.

As shown in Fig. 1, the initial concentration solution (aniline and HCl at the molar ratio of 1:5) was added to compartment I and II, respectively. A voltage was applied to the electrodes to generate an electric field. It is well known that the AEM permits the passage of anions only, while the CEM permits the passage of cations only. When an electric potential is applied across the stack, the $\text{C}_6\text{H}_5\text{-NH}_3^+$ in solution migrate toward the cathode. They can pass through the CEM, while they are stopped by the AEM. Meanwhile, Cl^- migrates toward the anode. They can pass through the AEM but are stopped by the CEM. Consequently, $\text{C}_6\text{H}_5\text{-NH}_3^+$ and Cl^- ions entered into

compartment I, and in turn to concentrate aniline; At the same time, the aniline solution of compartment II could be diluted.

Aniline was bought from Tianjin Guangfu Fine Chemical Industry Institute. HCl solution (36%), anhydrous sodium sulfate, and sodium hydroxide were from Beijing Yili Fine Chemical Ltd.

2.2. Analytical methods

The aniline concentration was detected using high performance liquid chromatography (SCL-10Avp, Shimadzu). Shim-pack VP-ODS (4.6×150 mm, $5 \mu\text{m}$) C-18 was used. A mixture of methanol and pure water at the ratio of 40:60 was used as the mobile phase, and the flow was 0.8 ml/min . The detection wave length was 280 nm . The standard curve between aniline and $\text{C}_6\text{H}_5\text{-NH}_3^+$ varied within a narrow band. Thus, the concentration of aniline was detected instead of $\text{C}_6\text{H}_5\text{-NH}_3^+$ by adding sodium hydroxide (NaOH) to sample. This is due to the fact that the concentration of $\text{C}_6\text{H}_5\text{-NH}_3^+$ could only be detected within 200 mg/L by HPLC.

In this study, an initial solution at a concentration of 500 mg/L (aniline and HCl at molar ratio of 1:5) was added into concentrate and dilute compartment, respectively. Under the operating voltage of 32 V , the solutions in the compartments were circulated at the flow rate of 60 L/h using pumps, respectively. Sodium sulfate (Na_2SO_4) solution with a conductivity of around $2,000 \mu\text{S/cm}$ was used as electrode solution. After operation for 120 min , the electrodedialyzer was

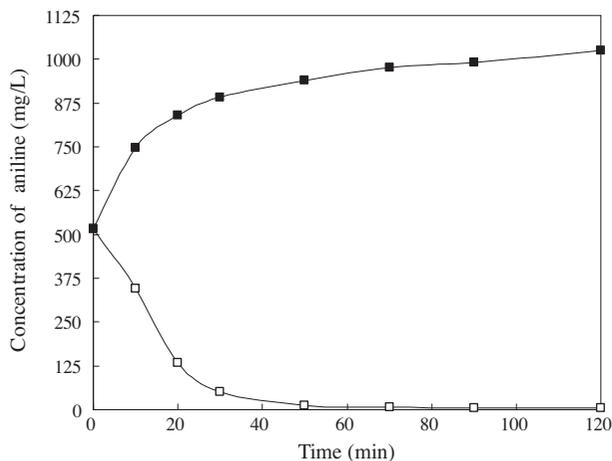


Fig. 2. Variation of aniline concentration during ED: (■) aniline concentration of concentrate chamber; (□) aniline concentration of dilute chamber. Reaction conditions: flow rate, 60 L/h; voltage, 32 V; aniline and HCl at molar ratio of 1:5.

switched off. In this work, two parameters of removal ratio (η ,%) and concentration ratio (ξ ,%) were defined to evaluate the performance of the process. Removal ratio (η ,%) from the dilution was calculated using Eq. (3). Concentration ratio (ξ ,%) from the concentrate was calculated using Eq. (4). The concentration of aniline in the concentrate compartment and dilute compartment were measured at regular intervals, and the results are shown in Fig. 2.

$$\text{Removal ratio } \eta(\%) = \frac{c_0 - c_e}{c_0} \times 100\% \quad (3)$$

$$\text{Concentration ratio } \xi = \frac{c'_e}{c'_0} \quad (4)$$

where c_0 is the initial aniline concentration and c_e is the final aniline concentration in the dilute chamber. c'_0 is the initial aniline concentration, and c'_e is the final aniline concentration in the concentrate chamber.

3. Results and discussion

3.1. The feasibility of aniline wastewater treatment by ED

As shown in Fig. 2, the aniline concentration of the dilute compartment decreased from 500 to 4 mg/L, which met the city standards of pollution discharge (namely 5 mg/L) and achieved 99% aniline removal efficiency after 120 min operation. Meanwhile, the aniline concentration from concentrate compartment increased gradually from 500 to 1,025 mg/L. The

shape of these curves can be explained that the $\text{C}_6\text{H}_5\text{-NH}_3^+$ and Cl^- ions from dilute compartment migrated through the CEMs and AEMs entered into concentrate compartment as a result of an electrical driving force, respectively. In this manner, it is possible to determine the efficiency of the process for the removal of aniline from wastewater.

3.2. Effect of hydrochloric acid amount

Experiments were conducted under the limiting current density at different molar ratio of aniline and hydrochloric acid (HCl) (1:0, 1:1, 1:3, 1:5, 1:6). Aniline concentration in the concentrate compartment and dilute compartment were measured, and the results are shown in Fig. 3.

With the absence of HCl, the aniline solution could only be concentrated and desalinated from 1,000 to 1,279 mg/L and 697 mg/L, respectively, and only 30% removal ratio could be achieved after 120 min operation. The reason is that in neutral aqueous environments aniline is mainly present as $\text{C}_6\text{H}_5\text{-NH}_2$. Therefore, with the absence of HCl, ED processes are not effective for aniline wastewater treatment.

As shown in Fig. 3, the aniline concentration and removal rate could be much more rapid with the increase in the amount of HCl; meanwhile the aniline concentration and removal amount could be more increased with the increase in the amount of HCl.

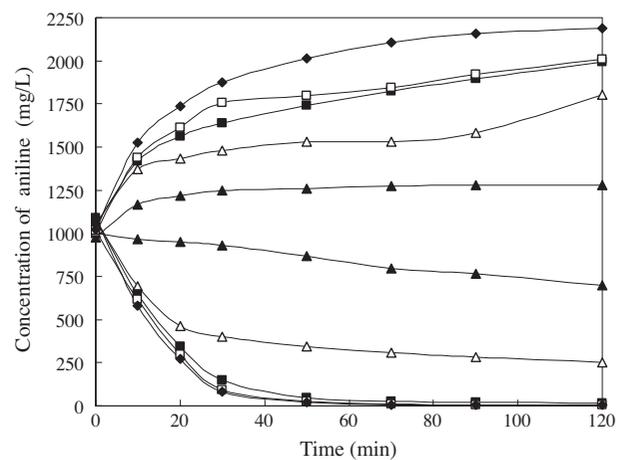


Fig. 3. Effect of additional HCl amount on the concentration of aniline in the concentrate compartment and dilute compartment during ED: dotted line, aniline concentration of concentrate chamber; solid line, aniline concentration of dilute chamber; symbols for initial molar ratio of aniline and HCl (▲) 1:0; (△) 1:1; (■) 1:3; (□) 1:5; (◆) 1:6. Reaction conditions: flow rate, 60 L/h; voltage, 32 V.

These results could be explained that the $C_6H_5-NH_3^+$ and Cl^- ions were increased with the increased concentration of HCl. Increasing current density which caused by the increase in ions contributes to $C_6H_5-NH_3^+$ ion migration. The amount of concentrated aniline in the concentrate chamber was 746, 948, 1,001, 1,167 mg/L and in the case of the mixture solution of aniline and HCl at molar ratio of 1:1, 1:3, 1:5, 1:6, respectively. With an initial concentration of 1,000 mg/L, the removal efficiencies of aniline could be up to 77, 98, 99, and 99%, and the final aniline concentrations after treatment were 249, 18, 7, and 6 mg/L when the molar ratio of aniline and HCl were 1:1, 1:3, 1:5, and 1:6, respectively.

It was observed that the aniline removal rate was rapid at the early stage of the experiment and gradually slowed down. As the concentration of Cl^- ions was maintained high in the earlier experiments, aniline mainly formed $C_6H_5-NH_3^+$ ions, which can be removed by electromigration. In the later experiments, where the Cl^- ion concentration was reduced, a very low concentration of natural form of aniline, that is, $C_6H_5-NH_3^+$ ion was formed with a very slow rate of removal. Consequently, aniline and HCl at molar ratio of 1:6 results in high concentration and removal efficiency. Concerning the energy consumption and the observations mentioned previously, aniline and HCl at molar ratio of 1:5 was chosen for the subsequent experiments.

3.3. Effects of initial aniline concentration

The effects of initial aniline concentration were investigated by changing the concentration from 100 to 1,000 mg/L (aniline and HCl at molar ratio of 1:5). The relationships between removal and concentration ratio and initial aniline concentration are shown in Fig. 4.

Fig. 4 shows that the initial concentration of aniline has an important influence on ions migration rate in the compartment. The number of ion migration from the dilute compartment to the concentrate one can be increased by raising the initial concentration of aniline. As the initial concentration of aniline increased, the ions concentration increased, and the membrane stack resistance decreased. Similarly, as the electric current increased, the ion migration rate was accelerated. These phenomena would consequentially favor to aniline wastewater treatment. The concentrations of aniline in the concentrate compartment ranged from 100, 300, 500, and 1,000 mg/L was concentrated to 206, 605, 1,025, and 2,008 mg/L, respectively. While different initial concentrations of aniline essentially

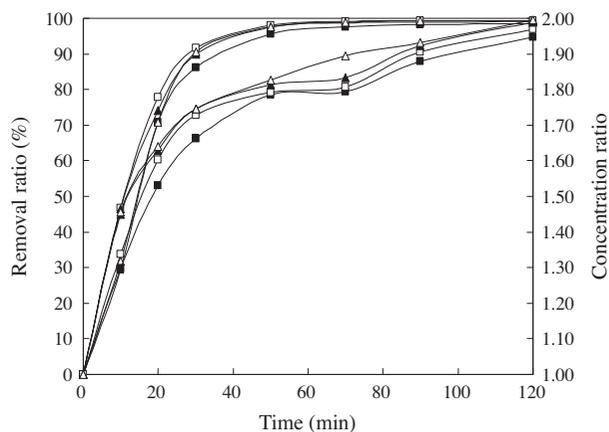


Fig. 4. Effect of initial aniline concentration on the concentration of aniline in the concentrate compartment and dilute compartment during ED: dotted line, aniline concentration ratio; solid line, aniline removal ratio; symbols for different initial aniline concentration (■) 100 mg/L; (□) 300 mg/L; (▲) 500 mg/L; (△) 1,000 mg/L. Reaction conditions: flow rate, 60 L/h; voltage, 32 V; aniline and HCl at molar ratio of 1:5.

have almost the same removal efficiency, which maintained at 99%, and the final concentration of aniline is 2, 2, 4, and 7 mg/L, respectively. Although the initial concentration of aniline has some influence on the treatment efficiency, concentrating capacity and desalinating capacity are always roughly the same.

It could also be proved that ED has a good performance in high-concentration aniline solution. The aniline concentration in dilute compartment could be diluted from 2,000, 5,000, 6,500, and 8,000 mg/L to 11, 19, 25, and 46 mg/L, respectively, and the removal of aniline is more than 99% after 120 min operation. This showed that ED has an ideal effect for the treatment of high- and low-concentration aniline solutions. For the high-concentration aniline solutions, the concentration of aniline in dilute compartment reduced significantly and the result could achieve the discharge standard of industrial wastewater after subsequent treatment. This process could reduce the cost of wastewater treatment.

3.4. Effect of applied voltage

Since the voltage applied has great influence on the ED efficiency, the experiments were conducted at five voltages (10, 20, 26, 32, and 38 V). Fig. 5 shows the profiles of the aniline concentration in the concentrate and dilute compartments at the different voltages.

As shown in Fig. 5, aniline could be concentrated and desalinated much more rapidly with the increase

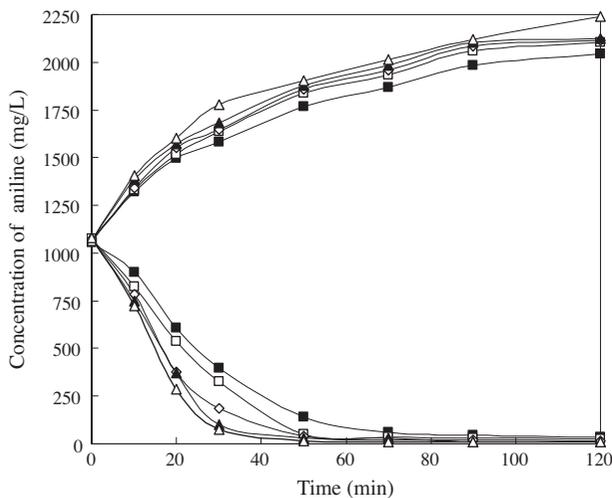


Fig. 5. Effect of applied voltage on the concentration of aniline in the concentrate compartment and dilute compartment during ED: dotted line, aniline concentration of concentrate chamber; solid line, aniline concentration of dilute chamber; symbols for different applied voltage (■) 10 V; (◇) 20 V; (△) 26 V; (▲) 32 V; (▽) 38 V. Reaction conditions: flow rate, 60 L/h; aniline and HCl at molar ratio of 1:5.

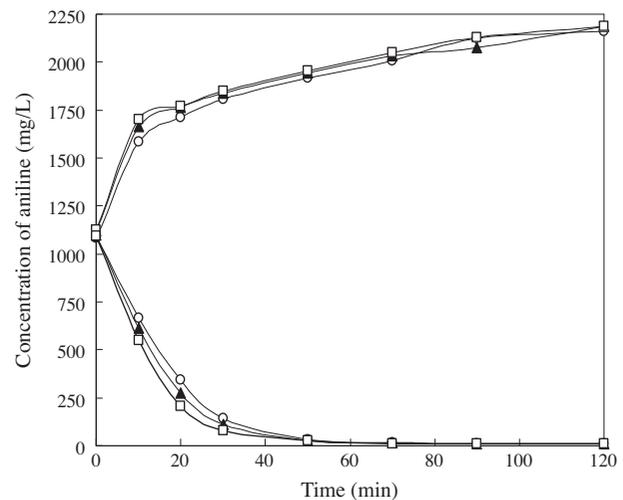


Fig. 6. Effect of flow rate on the concentration of aniline in the concentrate compartment and dilute compartment during ED: dotted line, aniline concentration of concentrate chamber; solid line, aniline concentration of dilute chamber; symbols represent different flow rate (○) 40 L/h; (▲) 60 L/h; (□) 75 L/h. Reaction conditions: voltage, 32 V; aniline and HCl at molar ratio of 1:5.

in the voltages applied. Under the voltage of 38 V, the aniline concentration in the concentrate compartment could be enriched from 1,000 to 2,000 mg/L, and dilute compartment could be desalinated from 1,000 to 10 mg/L after 70 min. In contrast, the aniline concentration would be concentrated from 1,000 to 2,000 mg/L, and dilute compartment could be desalinated from 1,000 to 35 mg/L after 120 min operation in the case of 10 V voltage. It was expected that the experimental process could be completed within a short time so as to reduce permeability of water caused by the osmotic pressure of the concentrate solution. Therefore, a relatively high applied voltage was required. However, too high applied voltage is associated with high-energy consumption. Take all of these into consideration, 32 V was chosen for the subsequent experiments.

3.5. Effect of flow rate

Fig. 6 depicts the variation of aniline concentration in the concentrate chamber and dilute chamber with the flow rate. It was noted from the figure that the aniline concentration in the concentrate and dilute chamber obtained at three different flow rate of 40, 60, and 75 L/h varied within a narrow band. Fig. 6 indicates that the concentrating and diluting rates in two compartments at the flow rate of 60 and 75 L/h are slightly faster than that at 40 L/h, while the aniline

removal ratio is almost same at 120 min. Although increasing flow rate decreases concentration polarization due to residence time reduction, the high flow rate usually requires high energy. Therefore, a medium flow rate of 60 L/h is recommended in this study.

3.6. The concentration capacity

In this study, we presented a cycling concentration process with relay style. Aniline solution was prepared in the dilute chamber with the same concentration of 500 mg/L (aniline and HCl at molar ratio of 1:5) in the concentrate chamber in the beginning of the experiment. Then, the second process was furthered, maintaining the solution in the concentrate chamber unchanged, while aniline solution in the dilute chamber was also prepared with the same concentration of 500 mg/L (aniline and HCl at molar ratio of 1:5) again. The process would not terminate until the concentration of the solution varied insignificantly in the concentrate chamber. Fig. 7 shows the profiles of aniline concentration and mass in the concentrate compartment with cumulative experiment.

Fig. 7 indicates that the aniline concentration in concentrate chamber is from 500 up to 6,390 mg/L through a cycling concentrating process, and the concentrated ratio is limited by the working system and the operation conditions. In general, the aniline concentration in concentrated chamber was increased

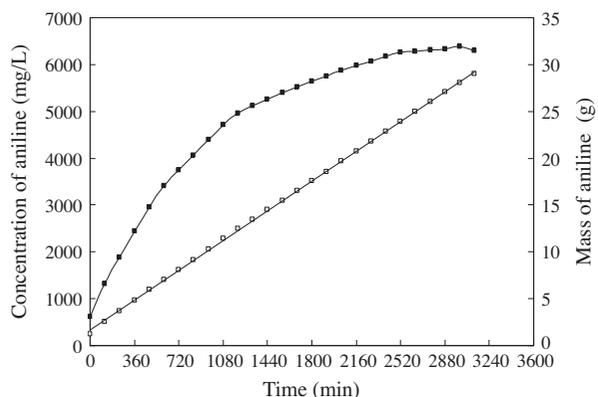


Fig. 7. Variations of aniline concentration in the concentrate chamber with cumulative experiment: (■) concentration of aniline in concentrate chamber; (□) mass of aniline in concentrate chamber. Reaction conditions: flow rate, 60 L/h; voltage, 32 V; aniline and HCl at molar ratio of 1:5; initial aniline concentration of 500 mg/L in dilute chamber.

with an increase in the current density. After a maximum value, the concentration of aniline in concentrate chamber began to drop from 6,390 to 6,304 mg/L. The

increase in the initial concentration would favor the increase in the overall current efficiency, but it led to a decrease in the concentrated ratio. The volume change of the concentrated solution is seriously affected by the current density and the ratio of the initial concentration in the concentrated compartment to that in the dilute compartment. With the increase in the current density and the ratio of the initial concentration in the concentrated compartment to that in the dilute compartment, the volume of the concentrated solution would increase greatly. In our experiments, we also found that the electroosmosis and back diffusion can great affect the ED efficiency [13].

Although the concentration diffusion has great influence on concentration efficiency, concentrating mass and desalinating mass are almost the same due to the rising liquid level of concentrated chamber. The removals of aniline decreased slightly with the increase in the number of the tests. In the last experiment, the removal of aniline still keeps 97% and final concentration of aniline is 15 mg/L. Therefore, removal and concentration of aniline from wastewater by ED has great potentialities.

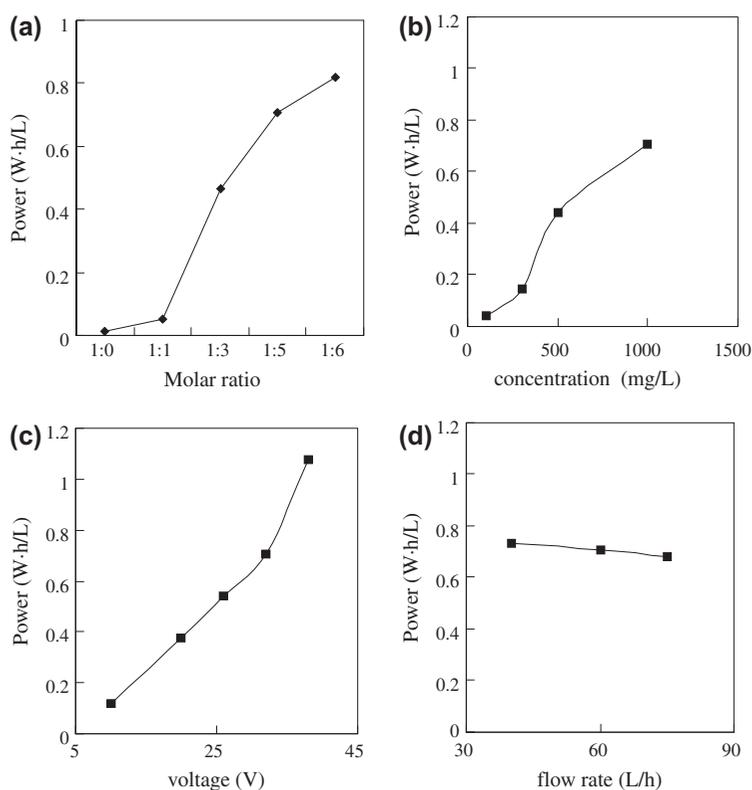


Fig. 8. Variations of energy consumption with additional HCl amount, initial aniline concentration, applied voltage and flow rate: (a) variations of energy consumption with the molar ratio of aniline and HCl; (b) variations of energy consumption with initial aniline concentration; (c) variations of energy consumption with applied voltage and (d) variations of energy consumption with flow rate.

3.7. Analyses of energy consumption

The energy consumption required for treating 1 L of aniline is defined in the following Eq. (5) [14]:

$$P = \frac{\int_0^t EIdt}{V_0} \quad (5)$$

where E is the applied voltage, I is the electric current, t is the operation time, and V_0 is the initial volume of feed solution. Variations of the energy consumption with additional HCl amount, initial aniline concentration, applied voltage and flow rate are shown in Fig. 8.

It can be seen from Fig. 8, (1) more total energy was consumed with the addition of HCl, owing to the fact that more HCl resulted in a higher current in the system under a consistent voltage. (2) The total energy consumption was also increased with the initial concentration of aniline, since the ED operation current is proportionally increased with the initial concentration. (3) The total energy consumption increases with the operating voltage due to the resulting higher operation current. (4) Total energy consumption decreased slightly with the increase in the flow rate. Flow rate has little influence on the current value. Thus, under different flow conditions, the power consumption of the ED will be basically the same.

It is apparent from above results that the operating conditions of the ED processes, such as the operating voltage of 32 V, the flow rate of 60 L/h, and the aniline and HCl at molar ratio of 1:5, bring the maximum value of treating aniline wastewater. For an initial aniline concentration of 1,000 mg/L under selected conditions, the energy consumption was only about 0.7 W·h/L.

3.8. Applicability of the present approach

The amount of aniline wastewater generated from chemical industry is generally not much, but in high concentrations. For this kind of aniline solutions, the concentration of aniline reduced significantly by the ED and achieved the discharge standard of industrial wastewater after subsequent treatment. On contrast, the amount of aniline wastewater from fine chemical industry, such as pharmaceutical and pesticide manufacturing plants, is often quite high, although the concentration is relatively low. The low concentration of aniline can be removed effectively by the ED process, and the discharged water can meet the standards of city pollution discharge (namely, 5 mg/L). Meanwhile, the overall treatment cost of the present process is quite low for both high- and low-concentration of aniline wastewaters. This process might also be applica-

ble for the treatment of wastewater containing weak bases such as other amines.

4. Conclusions

This study presents a novel approach to remove aniline from wastewater using ED apparatus in the presence of HCl. Considering the treatment efficiency and energy consumption, the operating condition of ED processes was chosen as follows: the operating voltage of 32 V, the flow rate of 60 L/h and the aniline and HCl at molar ratio of 1:5. Under this condition, the aniline solution can be desalinated from 500 to 4 mg/L. About 99% of aniline could be successfully removed and the energy consumption was 0.44 W·h/L. With a cycling concentration process, $C_6H_5-NH_3Cl$ solution can be concentrated from 500 to 6,390 mg/L by the ED apparatus.

Acknowledgments

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