



Desalination of concentrated wastewater from reverse osmosis by bipolar membrane electro dialysis

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ABSTRACT

The present study focused on the feasibility of bipolar membrane electro dialysis (BMED) for desalination of concentrated wastewater from reverse osmosis, including the reduction of total dissolved solids (TDS) and the generation of mixed acid/base. The effects of parameters such as current density, electrolyte concentration, and initial acid/base concentration were explored and discussed. The optimal current density was 10 mA/cm², the suitable electrolyte concentration was 0.25 mol/L, and the appropriate initial acid/base concentration was 0.05 mol/L. Experimental results indicated that 89.05% TDS could be removed, and the generated acid and base concentrations could reach to 0.12 mol/L and 0.18 mol/L respectively. The energy consumption for removing per kilogram TDS was 2.33 kWh. Besides, the energy consumption for acid generation was 2.55 kWh/kg, with a current efficiency of 67.7%; and for base production was 1.80 kWh/kg, with a current efficiency of 87.5%. All the results indicated that the BMED process was technical and environmental feasible for desalination of concentrated wastewater from RO. The generated mixed acid/base solution and fresh water (TDS less than 1.45 g/L) after treatment could be reused as resource.

Keywords: Bipolar membrane electro dialysis; Desalination; Concentrated wastewater; Acid; Base

1. Introduction

Reverse osmosis (RO) is a main technique in wastewater depth treatment, which applied widely in dyeing wastewater treatment [1–3]. After the high recovery rate of fresh water, the concentrated water from RO process, with upgraded of chromaticity, chemical oxygen demand (COD), salts, and other contaminants, is difficult for treatment [4]. The total dissolved solids (TDS) content in concentrated wastewater is usually over 10 g/L [5–9]. If the concentrated wastewater discharges into the environment without treatment may potentially cause environmental and human health problems such as cell dehydration and eco-toxicological risks [10].

Several methods have been proposed to treat the concentrated wastewater. Unfortunately, biochemical techniques are limited for the treatment due to the low survival of

organisms in concentrated wastewater [11,12]. The physico-chemical technique like membrane distillation, vaporization and coagulation-flocculation are utilized for treating the high salinity wastewater [13–15]. However, there are still several shortcomings such as membrane fouling, chemical consumption or produce waste salt which limit the applications of those technologies [16,17]. With higher removal rate of salt, fewer chemical reagents, and higher current efficiency as compared to several methods mentioned above, bipolar membrane electro dialysis (BMED) offers an environmentally friendly and sustainable platform in industrial application.

Bipolar membrane electro dialysis has been explored as a promising process to control water pollution and achieve sustainable development [18–20]. H⁺ and OH⁻ are split under a reverse bias of direct current field which combining anions and cations in aqueous into acid and base [21–23]. BMED can be used in several applications such as chemical processing (inorganic and organic acid/base production) [22,24], process integration (isolation of organic acids, food

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sector) [25,26] and pollution control (salt removal, acid/base regeneration) [16,27]. Guan et al. [28] found that BMED was suitable for producing of ammonium metatung state, and productivity of 99.50% was achieved. Lameloise et al. [29] used BMED to treat beverage industry wastewater, and the generated mixed acid was exceeded 50.30 g. Wei et al. [27] reported that 1 mol/L sodium hydroxide was regenerated from spent caustic stream of petrochemical industry at current density of 50 mA/cm². In terms of the produced acid and base from industrial wastewater, many experimental studies have shown promising results. However, the application of BMED for desalination of the concentrated wastewater from RO process in dyeing factory, has not received much attention yet.

In many literatures, it was evident that many researchers had worked in the field of wastewater treatment by BMED [30–32]. Simultaneously, various parameters such as current efficiency, feed concentration, energy consumption, conductivity, solution pH and fluxes were investigated [33,34]. Although the principle to produce acid and base by BMED process is always the same, the applications are different for every case since the feed streams are very complicated which lead to many challenges in industrial application [18].

In this work, the concentrated wastewater from RO was treated by BMED for desalination and acid/base generation. Main operation parameters such as current density, initial electrolyte concentration and initial acid/base concentration, were investigated. The TDS removal, current efficiency and energy consumption were calculated to evaluate the feasibility and capacity of BMED on wastewater

treatment. Besides, a tentative description of the kinetics of TDS removal was also presented.

2. Experimental

2.1. Apparatus and materials

Schematic diagram of the BMED experimental setup is illustrated in Fig. 1. Each membrane had an effective membrane area of 189 cm². Electrodes were made of titanium coated with ruthenium. Compartments were separated by spacers with a thickness of 0.70 mm, and each compartment was connected to a submersible pump, which was placed in a 1000 ml beaker. Acid/base compartment was installed with deionized water, or different concentrations of acid/base solutions varying from 0.05 to 0.20 mol/L. Feed compartment was composed of wastewater. Besides, a Na₂SO₄ (0.10–0.25 mol/L) solution was added to the electrode compartment. To balance initial osmotic pressure, the volume of adding solution in each compartment was 500 ml. Before an electric current was applied, the solution of each compartment was recirculated for half an hour to eliminate the potential bubble trapped inside BMED stack which will increase the resistance of the stack [35].

The main characteristics of the membranes used in this study are listed in Table 1. The data about the membrane information were collected from its producers. The current applied in BMED was provided by a constant voltage/current-controlled DC power source (MPS 1005, TRADEX, China). The chemicals used, including hydrochloric acid, sodium hydroxide and sodium sulfate, were of analytical grade. Deionized water was used throughout.

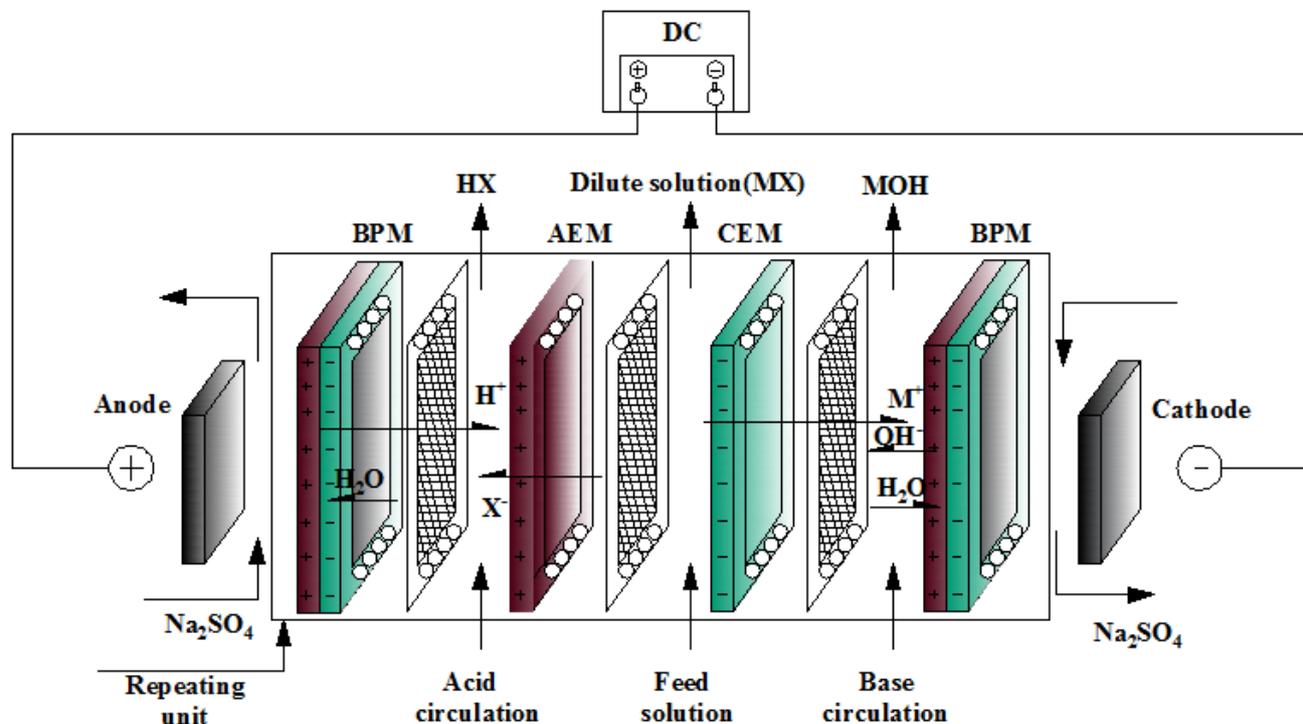


Fig. 1. Schematic of BMED setup operating principle. (BPM, bipolar membrane; AEM, anion exchange membrane; CEM, cation exchange membrane).

Table 1
Main characteristics of the membranes used in the BMED experiments

	Membrane	Thickness (mm)	IEC (meq/g)	Area resistance (Ω cm ²)	Transport number
1	BMP	0.16–0.23	–	–	–
2 ^a	FKB	0.11–0.13	1.20–1.30	<5	>0.98
3 ^b	FAB	0.11–0.13	>1.10	<3	0.95

^aCation exchange membranes (FKB from Germany).

^bAnion exchange membranes (FAB from Germany).

2.2. Pretreatment of the concentrated wastewater

The concentrated wastewater after RO process was collected from one dyeing factory located in the region of Shaoxing county, Zhejiang province, China. The information of the wastewater is listed in Table 2. The concentrated wastewater contains high concentrations of TDS and certain amount of organics. The concentration of TDS was 16.40 g/L and the COD value was 420 mg/L. Based on our previous report [36], the organic pollutants in concentrated wastewater would cause membrane fouling in the BMED system without a pretreatment process. Thus, electrochemical oxidation (EO) was used as pretreatment to eliminate the organic matters before BMED. In electrolysis system, a single-compartment electrochemical flow cell was used as reaction cell containing a modified Ti/PbO₂ anode and Ti cathode. The anode and cathode were both square (10 × 10 cm²) with mesh-like structures. The inter-electrode gap was 20 mm. Within an electrolysis time of 60 min and current density of 15 mA/cm², the COD concentration was effectively decreased from 420 mg/L to 40 mg/L.

2.3. Analysis and calculation methods

All the actual wastewater samples were preserved in sealed polyethylene containers and kept at room temperature before analysis. The concentrations of cations and anions were measured by inductively coupled plasma-mass spectrometry (ICP-MS) and ion chromatograph, respectively. The concentrations of generated acid and base from BMED were determined by titrating with a standard sodium hydroxide and hydrochloric acid solution. Phenolphthalein and methyl orange were used as the indicator of titration end-points, respectively. TDS and pH value of feed wastewater were monitored by using a conductivity meter and pH-meter, respectively.

TDS removal rate W (%) was calculated in Eq. (1) [37]

$$W = \frac{Y_0 - Y_t}{Y_0} \times 100\% \quad (1)$$

where Y_0 and Y_t were the concentrations of TDS (g/L) at time 0 and t , respectively.

The current efficiency CE (%) for acid/base generation was calculated in Eq. (2) [38].

$$CE = \frac{n(C_t - C_0)VF}{NI} \times 100\% \quad (2)$$

where n was the ion's absolute valence; C_0 and C_t were the acid/base concentrations (mol/L) at time 0 and t , respectively; V (L) was the recirculated volume of solution in the acid/base cycle; F was the Faraday constant (96485 C/mol); N was the number of repeating units ($N = 5$); I (A) was the

Table 2
The concentrations change of the main pollutant

Parameters	RO concentrate	EO pretreatment
pH	7.83	6.57
COD _{cr} , mg/L	420	≤40
TDS, g/L	16.40	13.24

current (assumed constant). Since the volume change of solution fed to each compartment was negligible during operation, V was equal to 500 ml throughout.

The energy consumption E (kWh/kg) for recycling per kilogram acid/base (100% concentration) was calculated in Eq. (3) [38].

$$E = \int \frac{UI dt}{(C_t - C_0)VM} \quad (3)$$

where U (V) was the voltage drop across the BMED stack; I (A) was the current; C_t and C_0 were the concentrations of acid/base (mol/L) at time t and 0 , respectively; V was the volume of the acid/base cycle (500 ml); and M was the molar mass of acid/base.

The electrical resistance R (Ω) was calculated in Eq. (4)

$$R = \frac{U}{I} \quad (4)$$

where U (V) was the voltage drop across the BMED stack; I (A) was the current.

The current efficiency CE' of desalination (TDS removal) was calculated in Eq. (5) [39].

$$CE' = \frac{mnF}{MQ} \quad (5)$$

where m was the weight of salt removed; n was the stoichiometric number ($n = 1$ in this case); F was the Faraday constant; M was the molecular weight of salt and Q was the electric quantity passed.

The energy consumption E' (kWh/kg) for desalination (TDS removal) was calculated in Eq. (6) [40].

$$E' = \frac{UI t / 1000}{m_{\text{final product}}} \quad (6)$$

where $m_{\text{final product}}$ (kg) was the mass obtained of the desired product (mixed acid and base produced by BMED).

All the experimental data were averaged from three independent experiments. The error was calculated to be approximately $\pm 5\%$.

3. Results and discussion

3.1. Effect of current density on desalination and acid/base generation

The current density refers to input power, is the main driving force in BMED. The electric force drove the anions (M^+) and cations (X^-) dissolved in wastewater to pass through the ion exchange membranes, then the TDS concentration in feed solution compartments decreased. As shown in Fig. 2, the removal efficiencies of TDS were 67.52%, 89.05%, 91.39% and 92.67% within the operation time of 25 min under the current densities of 5, 10, 15 and 20 mA/cm², respectively. The change curves of TDS concentration were linearly related to the electrolysis time [Eq. (7)] and the correlation coefficients were all above 0.99.

$$C_t = Kt + C_0 \quad (7)$$

where C_t was the TDS concentration at operation time t , mg/L; K was the line slope, t was the operation time, min; C_0 was the TDS initial concentrations.

From Eq. (7), the slopes K for five corresponding current densities were 0.37, 0.46, 0.49, and 0.50 (as listed in Table 3). The K increased about 1.35 times as current density from 5 to 20 mA/cm².

Fig. 3 shows the effect of current density on the acid/base yield and its energy consumption. The concentrations

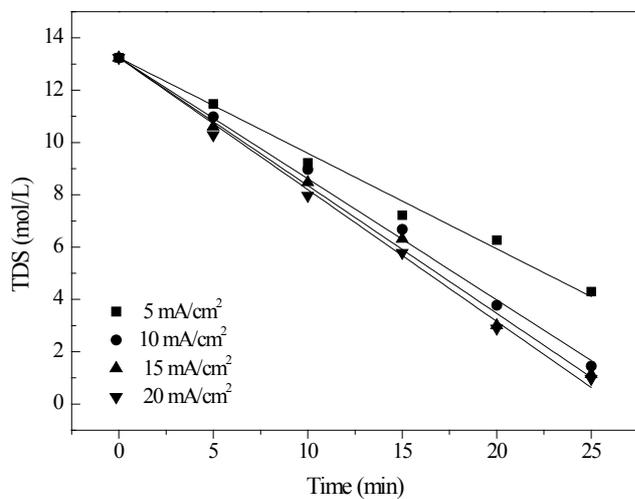


Fig. 2. Effect of current density on desalination. Other operation conditions: initial concentration of acid/base: 0.05 mol/L, concentration of electrolyte: 0.25 mol/L, liquid flow speed: 25 L/h.

Table 3
The apparent reaction rate constant of TDS removal under different current densities

Current density (mA/cm ²)	K	R ²
5	0.37	0.99
10	0.46	0.99
15	0.49	0.99
20	0.50	0.99

of the produced acid/base increased with the current density. However, the growth trend of the acid/base concentration was not so positive to current density (Fig. 3a). It could be explained that no sufficient salt ions were migrated into acid/base compartment, and combined with H^+ / OH^- generated by bipolar membrane to form acid/base. Simultaneously, with current density increased, the concentration of generated acid/base increased, which meant that the osmotic pressure in acid/base compartment also increased. The higher pressure accelerated the condition of ions leakage. That's the reason why acid/base concentration increased slightly with current density increased. Besides, the leakage of protons (H^+) was easier than OH^- according to Fick's first law and osmotic pressure [21], which caused the generated acid concentration was lower than the base concentration.

According to Eq. (2) and Eq. (3), the current efficiency CE (%) and the energy consumption under different current densities were calculated. As shown in Fig. 3b, the current

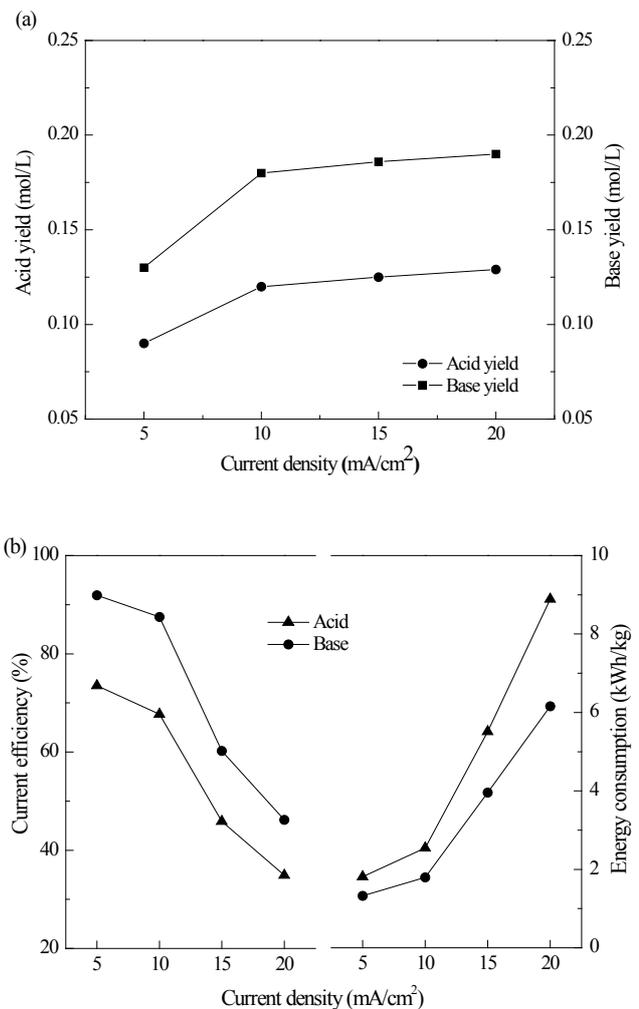


Fig. 3. Effect of current density. (a) Acid and base yield, (b) energy consumption and current efficiency. Other operation conditions: initial concentration of acid/base: 0.05 mol/L, concentration of electrolyte (Na_2SO_4): 0.25 mol/L, liquid flow speed: 25 L/h.

efficiency decreased and the energy consumption increased with the current density increased. A high current density made a high desalination rate, however, it also caused low current efficiency and high energy consumption, which was similar to others' studies [18,33]. Knee-points of CE and E curves were showed at current density of 10 mA/cm², which meant that 10 mA/cm² was the suitable current density in this system.

3.2. Effect of electrolyte concentration

In this work, Na₂SO₄ was served as supporting electrolyte. Fig. 4 shows the effect of electrolyte concentration on BMED. As shown in Fig. 4a, acid and base yield had been affected slightly by electrolyte concentration. The generated acid concentration increased from 0.09 mol/L to 0.12 mol/L, while the base concentration increased from 0.15 mol/L to 0.18 mol/L, with electrolyte concentration increased.

The total electrical resistance of BMED was mainly composed of electrodes, membranes, electrolyte, wastewa-

ter, acid and base compartments. In this section, electrolyte concentration was the crucial factor for the resistance. The curves of electrical resistance and energy consumption are shown in Fig. 4b. It could be observed that the electrical resistance decreased from 9.61 Ω to 7.91 Ω and the energy consumption also decreased with the electrolyte concentration increased. It meant that with the increasing of electrolyte concentration, lower electric energy was wasted in electrolyte compartment and more power was used for desalting and generating acid/base. According to the production of acid/base concentration and energy consumption, the optimal electrolyte concentration of 0.25 mol/L was selected in this system.

3.3. Effect of initial acid/base concentration on BMED

The electrical resistance of acid/base compartment is also one part of the total electrical resistance of BMED. Fig. 5a shows the effect of initial acid/base concentration on the acid/base yield. A knee-point of acid/base curve

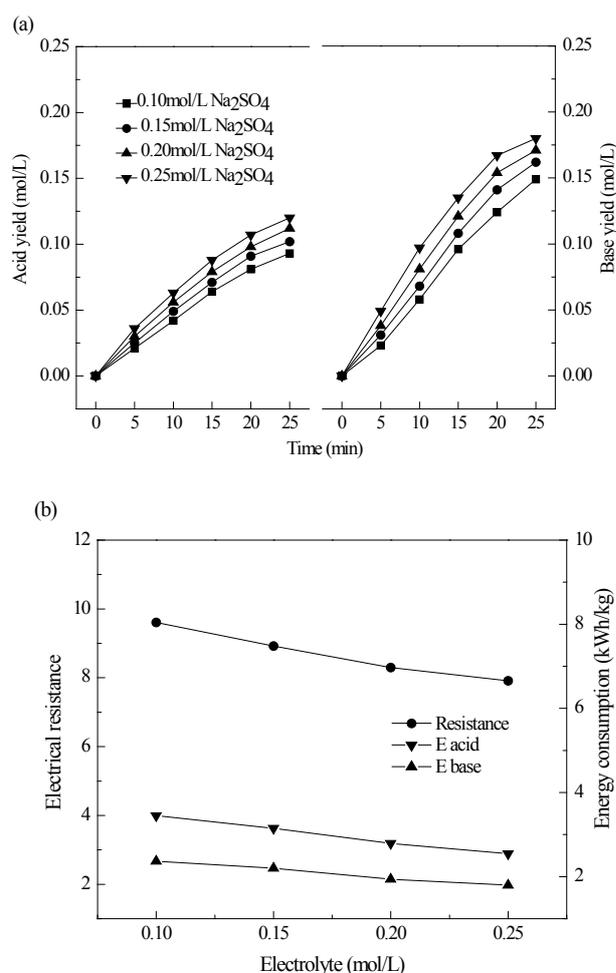


Fig. 4. Effect of electrolyte concentration. (a) Acid and base yield, (b) electrical resistance and current efficiency. Other operation conditions: initial concentration of acid/base: 0.05 mol/L, current density: 10 mA/cm², liquid flow speed: 25 L/h.

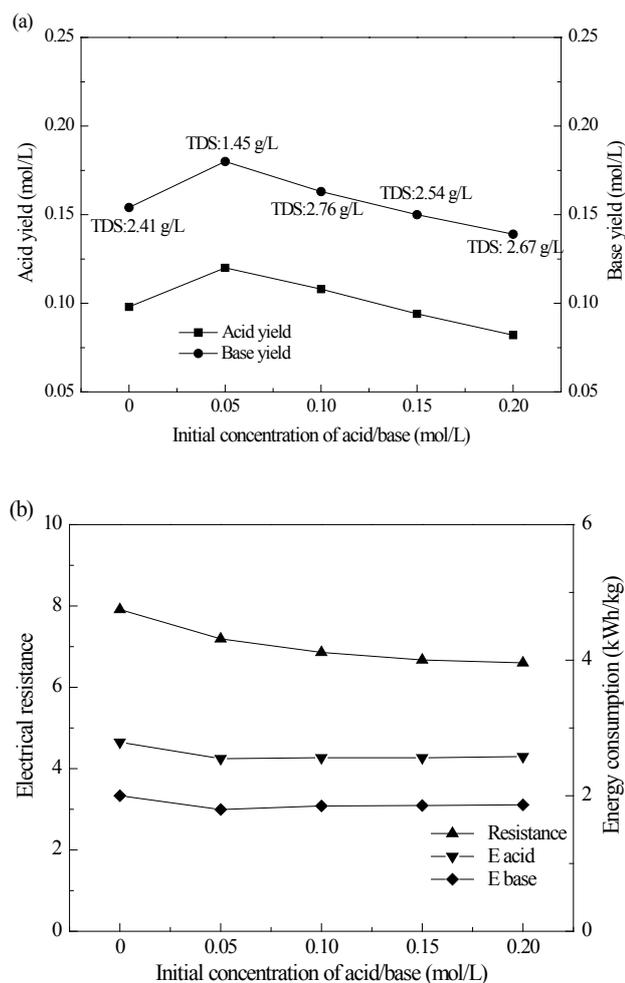


Fig. 5. Effects of initial acid/base concentration. (a) Acid and base yield, (b) electrical resistance and current efficiency. Other operation conditions: current density: 10 mA/cm², concentration of electrolyte (Na₂SO₄): 0.25 mol/L, liquid flow speed: 25 L/h.

was showed at initial acid/base concentration of 0.05 mol/L. In the beginning, the increase of initial acid/base concentration reduced the electrical resistance of BMED (Fig. 5b), which was similar with electrolyte concentration mentioned in Section 3.2. However, high initial acid/base concentration might result in high osmotic pressure which could accelerate the H^+/OH^- leakage, similar results were reported by Wang et al. [41] and Xu et al. [42].

As shown in Fig. 5b, the electrical resistance decreased as the increase of initial concentration of acid/base. But the curve of energy consumption showed a knee-point at initial acid/base concentration of 0.05 mol/L. The results indicated that the suitable initial acid/base concentration for this system was 0.05 mol/L.

3.4. Pilot test

In order to verify the stability of BMED in industrial application, a pilot test (a continuous experiment) was investigated. Fig. 6 shows the current efficiencies of acid/base and desalination, energy consumption of acid/base in the pilot test. The current efficiency of desalination increased due to the generated concentration of acid/base increased, similar to the phenomenon in Section 3.3. On the contrary, the current efficiencies for acid and base showed a slight decrease as the experiment increased from batch 1 to batch 4. This might be related to high pressure from acid/base compartment with operation time. After 4 batch operation, 0.43 mol/L mixed acid and 0.67 mol/L mixed base were produced. Besides, it could be seen that there was a slight decrease in energy consumption. The reason was that, with increase of the generated acid/base concentration, the electrical resistance of the stack decreased and more energy was used on wastewater treatment [43,44]. All the results indicated that it was feasible for efficient disposal of concentrated wastewater by BMED.

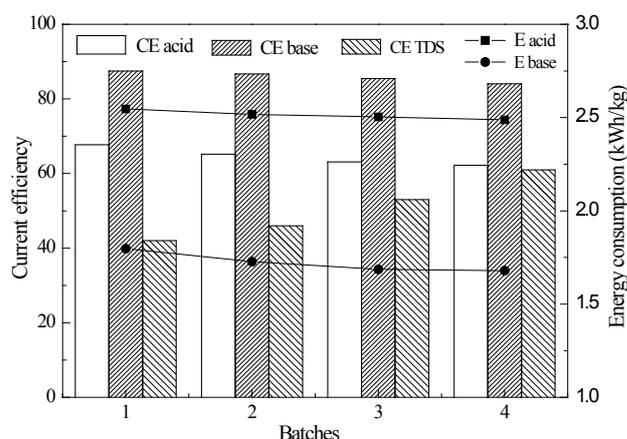


Fig. 6. The current efficiencies of acid/base and TDS removal, energy consumption of acid/base in pilot test. Other operation conditions: initial concentration of acid/base: 0.05 mol/L, concentration of electrolyte: 0.25 mol/L, liquid flow speed: 25 L/h, current density: 10 mA/cm².

4. Conclusion

The study shows that desalination of concentrated wastewater from reverse osmosis by bipolar membrane electro dialysis was not only technical but also environmental feasible. Experimental results indicated that, under the optimal conditions, 89.05% TDS could be removed, and the generated acid and base concentrations could reach to 0.12 mol/L and 0.18 mol/L respectively. The energy consumption for generating per kilogram acid was 2.55 kWh, with a current efficiency of 67.7%; and for per kilogram base was 1.80 kWh, with a current efficiency of 87.5%. Besides, the stability of BMED in industrial application had been verified by a pilot test. After treatment, the generated mixed acid/base solution and fresh water (TDS less than 1.45 g/L) could be reused as resource. It confirmed that BMED was an excellent process on desalination and mixed acid/base generation for concentrated wastewater treatment.

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