Occurrence of re-adsorption in desorption cycles of capacitive deionization

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ABSTRACT

This research explored the occurrence of re-adsorption that may impose negative impact on electrode regeneration in capacitive deionization (CDI). Among the tested operational modes, polarity reversal was found to deliver faster salt desorption than a conventional short circuit and reduction of cell voltage to 0.3 V. Earlier occurrence of re-adsorption was observed when the CDI system was operated under a higher reversed discharge voltage. An optimized polarity reversal at 1.2 V followed by a short circuit is considered as a promising approach for efficient CDI operations for practical engineering use.

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Introduction

Inadequate access to purified water is one of the most critical problems worldwide. Among current water purification technologies, reverse osmosis and multistage flash distillation are widely used for separation of ions from electrolytes. While these conventional processes focus on the treatment of the majority water molecules and thus lead to high energy consumption, capacitive deionization (CDI) [1] with its membrane [2] and microbial [3,4] derivatives have emerged as a promising technology working on the direct removal of the minority ions by capacitive adsorption and has demonstrated many advantageous features (i.e. low pressure, no heating and chemical reagents requirements) in water desalination.

CDI consists of repeated cycles of salt adsorption (charging) and desorption (discharging) processes in spite of a recently introduced continuous CDI system based on flow electrodes [5]. During typical adsorption, ions are stored as counter-ions within the structures of porous electrodes. Simultaneous with counter-ion adsorption, co-ions are expelled from the electrodes. Because the co-ion outflow is always less than the counter-ion adsorption under the influence of an applied electrical potential difference, there is a net removal of

ions, leading to water desalination [6]. The salt desorption (electrode regeneration) process can be operated in three modes: (1) reducing the cell voltage; (2) short-circuiting the electrodes; and (3) reversing the polarity. During desorption, the co-ion expulsion becomes greater than the counter-ion adsorption, resulting in a net release of ions to the stream. When the co-ion expulsion equals to the counter-ion adsorption, no ions will be further released and the electrode is regenerated.

Although the simple short-circuit desorption is easily used, further research is need to explore other desorption modes to enhance the operational efficiency of CDI. For example, the use of polarity reversal was suggested for rejuvenation of a CDI system since a voltage reversal might momentarily expel the adsorbed ions from the electrodes [7], which could potentially reduce the electrode regeneration time and brine water production. In addition, complete electrode recovery could also be achieved [8]. Kim et al. [9], on the other hand, explored a discharge voltage of 0.3 V reduced from an adsorption cell voltage of 1.2 V and reported enhanced charge efficiency [10]. However, the salt adsorption capacity was impaired due to incomplete regeneration. The classical response of CDI was also tested under step voltages, i.e. inverted CDI, and the inversion was discussed by a recent theoretical study [11]. Despite the aforementioned advantageous aspects of polarity reversal, potential drawbacks include energy consumption and the occurrence of re-adsorption, that is, the reversal of cell voltage may drive the desorbed ions adsorbed again by the counter electrodes, which potentially limits the desorption

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efficiency. Although the introduction of ion-exchange membranes to the CDI (a processed named MCDI) may solve the re-adsorption problem and Liang et al. [12] reported an increased desorption rate using a reversed polarity, negative impacts such as the increasing of overall reactor volume, resistance for solution flow and ion transport, and cost of membranes cannot be neglected. Therefore, there is a need to further investigate the non-membrane CDI systems. Although optimization of a CDI system involves evaluation on the choice of practices resulting in sufficient time for ion desorption and diffusion from the electrodes and sufficient flushing of the cell for proper electrode regeneration, the regeneration cannot be shortened indefinitely [13]. An assessment on the re-adsorption during polarity reversal is crucial for understanding the theoretical foundations and suggesting engineering practices for enhanced CDI desorption performances.

In this work, a bench-scale CDI system was built with activated carbon cloth as electrode. The CDI system was operated at different desorption modes (6 cell voltages and 4 flow rates) and the patterns of conductivity as a function of time in a closed-loop operation were plotted. The objective of this work was to explore the occurrence of re-adsorption by studying the different desorption patterns. Information on the occurrence of re-adsorption and the associated salt desorption rates will initiate a better insight into the re-adsorption phenomenon and optimized desorption settings for efficient CDI operation. Because low desorption efficiency (e.g. long regeneration time, high flush water usage, high brine production, etc.) imposes potential barriers for practical applications, the research findings will facilitate the beneficial use of CDI for water desalination as the technology moves into more commercial applications.

Materials and methods

CDI configuration

Fig. 1a gives a schematic overview of the experimental setup. The CDI cell contained one pair of "flow-by" activated carbon electrodes that was attached to titanium wires and sandwiched between two polyethylene end plates. Rubber gasket was used to seal the system and the channel between the electrodes was 8 mm wide. The channel width was chosen according to reliable engineering practice of reducing the risk of short circuit, because there could be overlapping of protruding fibers from the electrodes at a narrow channel under prolonged operations. A DC potentiostat (RXN-305D, Shenzhen Zhaoxin Power Co. Ltd., China) was used to provide a constant voltage. The cell measured 28.2 cm long,

12.4 cm wide and 1.6 cm tall, and had an effective volume of 450 mL. The electrode material was commercially available activated carbon cloth (ACC, FM10) obtained from Chemviron, UK. It had a specific surface area of 1000 m^2/g and a thickness of 0.5 mm. The average pore size of the material was 3.6 nm and the pore size distribution and surface chemistry were described elsewhere [14]. Prior to the cell assembly, raw ACC was cut into two pieces with a dimension of 25.6 cm × 11.8 cm^2 each, weighing 18.4 g total, and was pre-rinsed with DI water to remove residual ions.

CDI operation

The CDI cell was operated in a closed-loop mode in both adsorption and desorption cycles to illustrate the occurrence of re-adsorption. The saline water (1613 μS/cm, 793 mg/L NaCl) was made by dissolving analytical grade NaCl into DI water. It resembled high salinity fresh water that contains an amount of total dissolved solids up to 1000 mg/L [15]. The conductivity of the saline water was continuously monitored by a conductivity meter (DDS-307, Shanghai Yueping Scientific Instruments Co. Ltd., China) with a sensor placed into the beaker, which contained 50 mL of the recycled water from the CDI cell, on top of a magnetic stirring plate. Prior to the electrosorption experiment, the CDI cell had been equilibrated with the original saline water to eliminate the influence of physical adsorption. The amounts of adsorption presented in this research were all electro-adsorption data.

Fig. 1b illustrates the six operational modes used in this study. The charging cell voltages during the adsorption cycles were all maintained at 1.2 V. The difference was the discharging cell voltages during the desorption cycles, which were maintained at −1.2, −0.9, −0.6, −0.3, 0.0 and 0.3 V. The negative sign in discharging cell voltage values implied a reversed polarity, while the 0.0 V and 0.3 V denoted a short circuit and a reduced cell voltage, respectively. It is noted that these cell voltages were constantly applied during the entire desorption processes to explore the occurrence of any potential re-adsorption.

Calculation and data analysis

The conductivity of the saline water in the closed-loop operation was plotted as function of time. A calibration curve was obtained prior to the experiments to convert the conductivity values into the corresponding mass concentrations, and the salt adsorption (q_a) and desorption amounts (q_d) were presented in a unit of milligram salt per gram ACC (mg/g) as follows:

$$q_a = \frac{\Delta C_a V}{m_{ACC}} \tag{1}$$

$$q_d = \frac{\Delta C_d V}{m_{ACC}} \tag{2}$$

where ΔC_a and ΔC_d were the changes of mass concentration of salt (mg/L) as measured in the beaker during the investigated adsorption and desorption periods, respectively; V was the volume of the saline water (0.5 L) recirculating in the system; m_{ACC} was the mass of ACC (18.4 g) in the cell.

The salt adsorption rate (R_a) and desorption rate (R_d) in units of mg/g/h were calculated as follows:

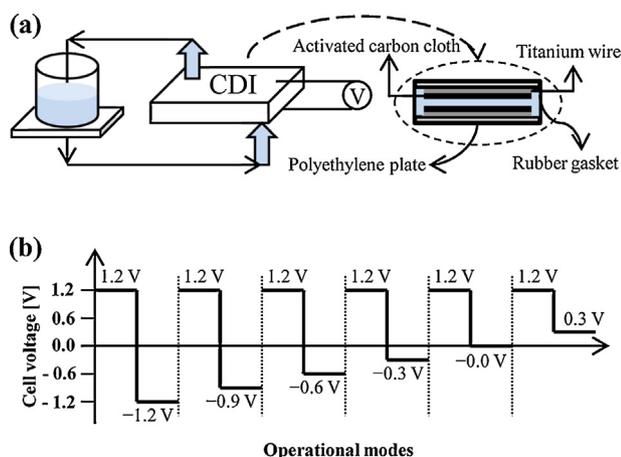$$R_a = \frac{q_a}{t_a} \tag{3}$$

Fig. 1. (a) Schematic diagram of the closed-loop CDI operation. (b) Charging and discharging cell voltages in adsorption and desorption cycles of the CDI system.

$$R_d = \frac{q_d}{t_d} \tag{4}$$

where t_a was the charging time (h) in the adsorption phase; t_d was the discharging time (h) prior to the occurrence of re-adsorption.

Results

Adsorption performance of CDI prior to desorption

Fig. 2 presents the decreased conductivity as a function of time in a typical adsorption cycle of the closed-loop CDI system. A cell voltage of 1.2 V was applied to the cell and the flow rate through the cell was maintained at 30 mL/min, which was equivalent to a hydraulic retention time of 15 min. In order to have the subsequent desorption cycles start with a same q_a for the sake of experimental control, the adsorption periods of all tests ended when the conductivity value decreased from 1613 to 1475 ± 3 μS/cm. The adsorption processes took 60 ± 4 min and the resultant adsorption rate was 1.84 ± 0.11 mg/g/h. The system started with pristine ACC operated less than 100 cycles under DC voltages of less than 1.23 V. The adsorption and desorption were highly reversible with good reproducibility among these tested cycles, and the degradation of carbon materials due to aging [16–19] was not observed throughout the experiments. It is also noted that the system had no pre-charging time, and the conductivity values were recorded immediately after a DC voltage was applied or adjusted. Despite that the level of desalination in the experiments was not high due to a relatively low ratio of ACC and electrolyte volume [20] using one pair of electrodes, the goal of this work was to explore a phenomenon on research basis rather than practically exhibit a full-scale CDI system using more pairs of electrodes in series to achieve a decent desalination performance.

Effect of discharge voltage on occurrence of re-adsorption

Fig. 3 shows the patterns of conductivity as a function of time with the 6 tested desorption modes. The peaks indicating the occurrence of re-adsorption was found in the polarity reversal based desorption modes (-1.2 V, -0.9 V, -0.6 V, -0.3 V) at 22 min, 44 min, 62 min, and 76 min, respectively. The desorption by reducing the cell voltage to zero (short circuit) or 0.3 V was much slower compared to that of polarity reversal, and no re-adsorption peaks were identified in their conductivity-time series patterns. Because the polarity reversal based desorption modes could potentially deliver faster salt desorption, they are considered as candidate desorption modes for further research and development.

The key obstacle for the use of polarity reversal based desorption modes is the identification of the re-adsorption peak time. By applying a reversed discharge voltage longer than the
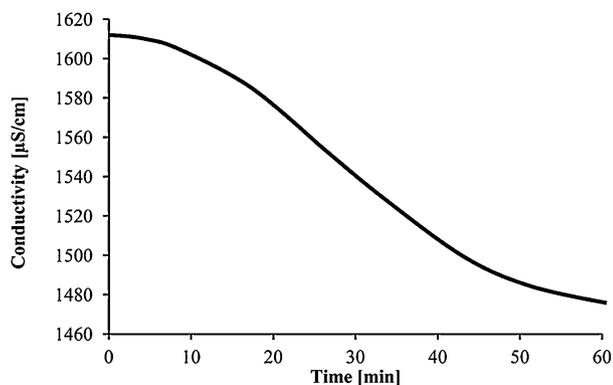

Fig. 3. Conductivity as a function of time in desorption cycles of the closed-loop CDI system operated at discharge voltages from -1.2 to 0.3 V while flow rates were maintained at 30 mL/min.

peak time, the conductivity decreased again and if the reversal continued, the electrode could never be regenerated. An ideal desorption mode for practical engineering use would be a combination of polarity reversal followed by a short circuit, and the duration of polarity reversal should be optimized based on the peak time obtained from bench-scale and pilot-scale experiments.

Among the tested desorption modes, a discharge voltage of -1.2 V was preferred due to the shortest time for the occurrence of re-adsorption. This could lead to the shortest electrode regeneration time after introducing a short circuit at the peak time. Noticeably, the heights of the re-adsorption peaks were also different, and the distance between the peak and the horizontal conductivity line of the original saline water suggested the unrecovered adsorption capacity at the peak for the polarity reversal based modes. Detailed discussion can be found in Section "Explanations to the impact of discharge voltage on desorption performance".

Effect of flow rate on occurrence of re-adsorption

Fig. 4 presents the conductivity–time patterns at 4 tested flow rates while the discharge voltages were maintained at -1.2 V throughout the desorption cycles. The results showed that the occurrence of re-adsorption appeared to be affected by flow rates. The longest peak time was obtained at 54 min since the start of desorption at a flow rate of 22.5 mL/min. Increasing the flow rate to 30 mL/min and 45 mL/min tended to result in faster salt desorption and decreased the peak time to 22 min and 20 min, respectively. A further increase of flow rate from 45 mL/min,

Fig. 2. Conductivity as a function of time in adsorption cycles of the CDI system operated at a cell voltage of 1.2 V and a flow rate of 30 mL/min.
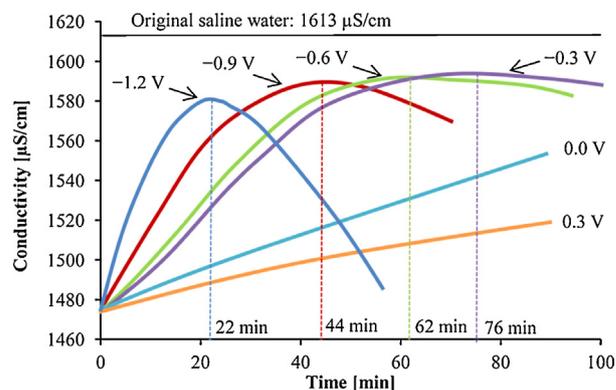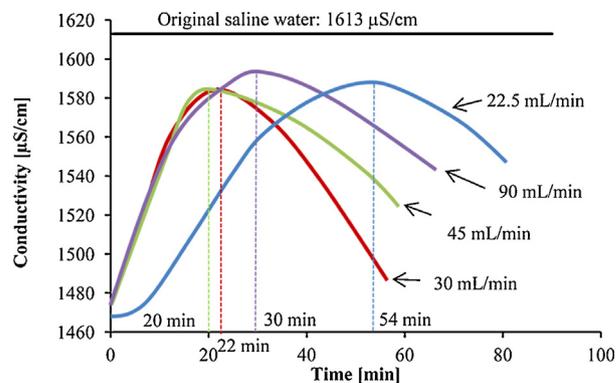

Fig. 4. Conductivity as a function of time in desorption cycles of the closed-loop CDI system operated at flow rates from 22.5 to 90 mL/min while discharge voltages were maintained at -1.2 V.

however, did not guarantee the same trends. At a flow rate of 90 mL/min, the peak time was identified at 30 min while the salt desorption rate did not appear to change much. The late occurrence of re-adsorption and the fast salt desorption contributed to a short distance between the peak and the horizontal conductivity line of the original saline water, implying a high recovery of adsorption capacity at 90 mL/min. Because flow rate is a parameter describing the treatment capacity of the CDI cell, a significantly low flow rate (22.5 mL/min) indicated that only a small volume of water was treated, which delayed the occurrence of re-adsorption in the closed-loop CDI operation. The similar salt desorption rates prior to the peak at flow rates of 30, 45 and 90 mL/min could be attributed to the same discharge voltage at −1.2 V, because the reversed potential was a main driving force for salt desorption and the role of flow rate could be minimum. Detailed discussion can be found in Section "Role of flow rate between closed-loop and single-pass CDI operation".

Discussion

Explanations to the impact of discharge voltage on desorption performance

The essence of CDI is the formation of the electrical double layer (EDL) [21] for ion storage in the micro- (<2 nm), meso- (2–50 nm) and macro-porous (>50 nm) structures of electrode materials under an applied cell voltage [1]. A higher cell voltage corresponds to a thicker EDL and stronger Coulombic interactions between the electrodes and charged ions [22]. When the EDL thickness is of similar magnitude as the pore size, the EDL inside the pores may overlap, and the overlapping effect exists in microporous and part of mesoporous regions of the electrode [23] but turns out not to negatively influence salt adsorption. Instead, it is shown by Porada et al. [24] that the smallest micropores (<1.5 nm) have the highest salt adsorption capacity per volume of electrodes and the sub-nanometer pore sizes are considered beneficial for water desalination [25]. The mechanism on the complex process of charge storage and the ion arrangement in the nanopores via EDL formation is an active research area. In addition to counter-ion adsorption and co-ion expulsion, Prehal et al. [26] also discussed "ion swapping" as the combination of both mechanisms. The dominance of each mechanism may depend on a variety of parameters, such as ion concentration, the cation–anion size ratio, or hydration energies [27].

In this research, reducing the discharge voltage from 1.2 V to 0.3 V decreased the EDL thickness which initiated the release of ions from the macropores back to the electrolyte. The salt concentration in the macropores may drop immediately [28,29], because the ion movement out of the macropores occurs with negligible resistance. Nevertheless, the problematic step in this desorption mode is the ion transport from the micropores to macropores. The EDL overlapping effect in the micropores may still exist at 0.3 V which may add resistance for the escape of the stored ions from the micropores into the macropores. In addition, the charge on the electrode at 0.3 V may affect the strength of the chemical bonds between the charged ions and functional groups of the electrode [30], which may further play a negative role on desorption. These possible causes could explain the slow electrode recovery observed during the desorption at 0.3 V.

Although the desorption mode at 0.0 V demonstrated a better performance compared with the one at 0.3 V benefiting from complete removals of potential and EDL by a short circuit, the rate was still not satisfying compare to those of polarity reversal. This could be attributed to the presence of affinity between the ions and adsorbing surface as a result of the chemical bonds [31]. In addition, it has been reported that as the electrodes are polarized,

even after the potential is removed, there are remaining counter-ions trapped in pores, which charge can hold some co-ions [32,33] and thus impairs the desorption efficiency.
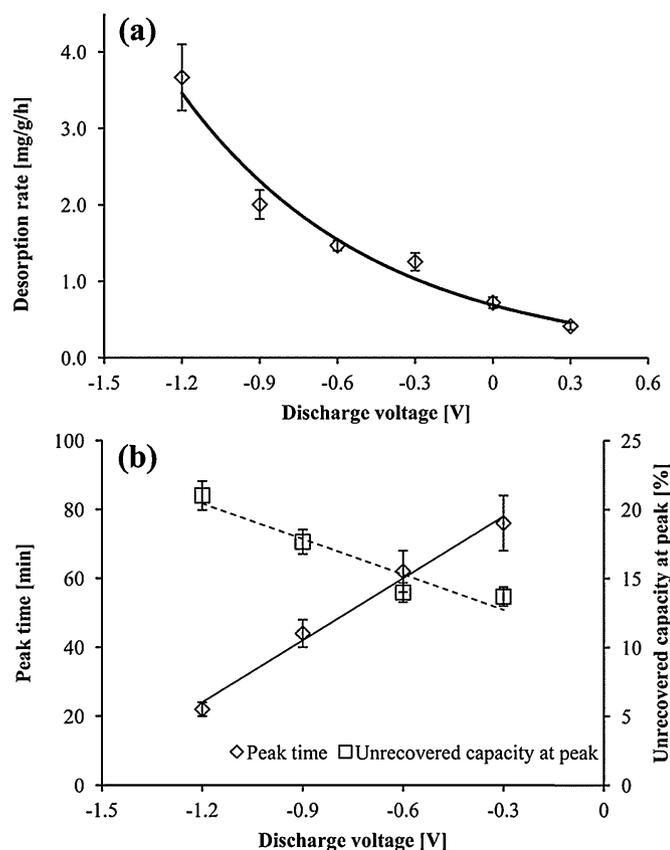
Polarity reversal is a promising way to overcome the affinity of the ions held on the electrodes and expedite desorption. It firstly discharges the counter-ions to the point that the electrodes become charge neutral. As the reverse charging continues and expels the co-ions from the EDL in the micropores, the co-ions are attracted to the counter electrode. Some researchers [28] suspected that the occurrence of re-adsorption is instantaneous and the polarity reversal is impossible to operate. This study implies that successful operation of polarity reversal is possible because the expelled ions were found to have sufficient chance to leave the cell via a suitable flush flow rate before being adsorbed again in the oppositely charged electrode. The observation of faster desorption at higher cell voltages during polarity reversal were attributed to a larger expelling driving force. The larger difference between the conductivity at the peak and that of the original saline water was also observed at a higher reversed discharge voltage, indicating greater portion of unrecovered adsorption capacity. This could be explained as follows: There is additional ion adsorption by the counter-electrode at high voltages, leading to a larger transport of desorbed ions into the counter electrode on a macroscopic scale during desorption [6].

Relation between discharge voltage and occurrence of re-adsorption

The effect of cell voltage on the adsorption process of CDI has been successfully modeled for predictions in practical applications [34,35]. Nevertheless, there is not much information on the effect of discharge voltage in the subsequent cycles of salt release, especially on the occurrence of re-adsorption when the polarity reversal based desorption modes are used. In this research, desorption rates were plotted against discharge voltages and an exponential trend line appeared to be able to give a good fit to the experimental data (Fig. 5a). Similar to the adsorption rates that are influenced by many factors, such as cell architecture, charging time, electrode material, electrode thickness, and all relevant experimental conditions [1], it must be noted that desorption rates may also be influenced by these factors and comparing values between different systems can be problematic [36,37]. The exponential response of CDI desorption to a high discharge voltage observed in this research resembles the adsorption response of an electrolyte reported by Porada et al. [24]. In addition to the "ion swapping" phenomenon that could explain a linear response of an electrolyte to a low voltage [38], it is deduced that the strongly nonlinear correlation was shown when co-ion expulsion started to exceed counter-ion adsorption into the EDL as the voltage increased further, leading on the macroscopic scale to a much larger co-ion transport outward into the bulk solution [6,39]. With the obtained trend, it is possible to estimate the desorption rates for the system operated at other discharge voltages. Besides, it is also important to describe how the re-adsorption peak time and peak heights respond to other reversed discharge voltages in the conductivity–time patterns. Fig. 5b shows that the re-adsorption peak time and unrecovered adsorption capacity at the peak may respond linearly to reversed discharge voltages. Although an explanation to the phenomena is not available so far, the trends of observations could be used to estimate the occurrence of re-adsorption for engineering use at other operating conditions.
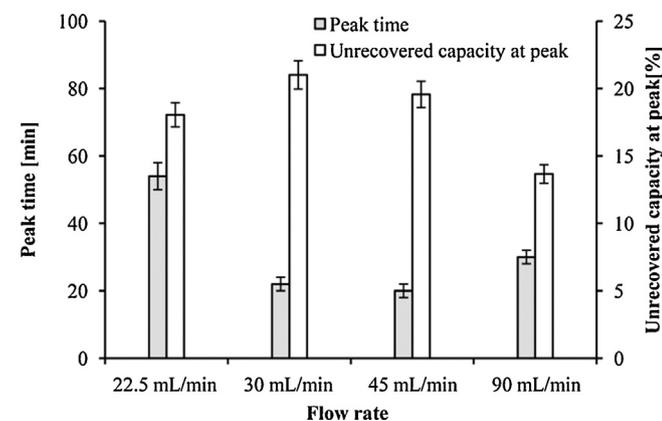
Role of flow rate between closed-loop and single-pass CDI operation

Jande and Kim [40] reported that the closed-loop (i.e. batch mode) CDI operation and single-pass CDI operation respond differently to flow rate. This research agrees with their findings

Fig. 5. (a) Desorption rate prior to the re-adsorption peak and (b) peak time and unrecovered percentage of adsorption capacity at the peak as a function of discharge voltage in desorption cycles of the closed-loop CDI system operated at flow rates of 30 mL/min.

concentration gradient and a decreased EDL thickness [42], which drove the release of more ions. Because a closed-loop system may not be used in engineering practice, the impact of flow rate is largely associated with the water residence time in a single-pass flow scheme. Since the desorption rates prior to the re-adsorption peaks were similar at flow rates of ≥30 mL/min under the same discharge voltage, it is possible to use an optimized flush flow rate in a single-pass flow scheme to (1) produce less brine water and (2) push the expelled ions out of the system without giving them sufficient chance to be adsorbed again by the counter electrode. Further research is needed to incorporate more operational variables (such as flow rate) into the modeling process [13] for development of a better tool for prediction of occurrence of re-adsorption for engineering use.

Conclusions

The research explored the impact of different desorption modes on the occurrence of re-adsorption in a closed-loop CDI system. The conductivity–time patterns revealed the time and height of the re-adsorption peak for each tested desorption mode. A higher discharge voltage during polarity reversal contributed to a shorter time for the occurrence of the re-adsorption and higher desorption rates prior to the re-adsorption peak. The impact of flow rate on the occurrence of re-adsorption in a closed-loop system is more obscure compared with that of discharge voltage. Optimization of flow rate is needed for reduction of brine water and successful operation of polarity reversal to carry the expelled ions to leave the cell without giving them sufficient chance being adsorbed again in the oppositely charged electrode. This research recommends the use of polarity reversal until the occurrence of re-adsorption followed by a short circuit to enhance desorption performance for practical applications. Further research is needed to fully predict the occurrence of re-adsorption by incorporating more operational variables into the modeling process.

Acknowledgements

that a reasonably high flow rate is preferred for closed-loop CDI operation. As revealed in Fig. 6, the highest tested flow rate at 90 mL/min resulted in the lowest unrecovered adsorption capacity at the re-adsorption peak despite that the peak time may not be the shortest. This is because increasing the flow rate in the closed-loop operation could drive the well-mixed water with a lower conductivity pass through the CDI channel. In addition, the EDL capacity decreases as the electrolyte solution concentration drops [41]. The enhanced recovery of adsorption capacity at the peak could be explained by the beneficial aspects of high flow rates on well-mixing of the closed-loop system and producing a favorable

Fig. 6. Re-adsorption peak time and unrecovered percentage of adsorption capacity at the peak in response to flow rate change from 22.5 to 90 mL/min in desorption cycles of the closed-loop CDI system operated at a discharge voltage of −1.2 V.

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