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Treatment and recovery of reverse osmosis reject by membrane capacitive deionization with integrated pretreatment

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ABSTRACT

Membrane capacitive deionization (MCDI) with integrated pretreatment was demonstrated to be a feasible solution to the reverse osmosis (RO) reject from the water reclamation process. Pretreatment comprised of biological activated carbon (BAC) and microfiltration (MF), which aimed to remove dissolved organic matter and suspended solids prior to the MCDI. Considering salt removal efficiency and process efficiency, the optimal applied voltage and flowrate of the MCDI unit were determined to be 1.3 V and 0.33 L/min. Except for iron and phosphate, the removal efficiency of calcium, magnesium, potassium, sodium, ammonium, chloride, nitrate, sulfate ranged between 68.8% and 90.3%. Removal efficiency for the TDS and TOC were in the range of 72.6%–85.8% and 21.6%–28.7%, respectively. Our study demonstrates that the MCDI desalting performance was stable during the continuous 35 d operation and the recovery efficiency and rejection efficiency of MCDI are 75% and 97%. Cleaning-in-place (CIP) was carried out every 10 d continuous operation to prevent cell fouling. CIP causes expansion of the fouling layer and ion exchange reaction with the polysaccharide-calcium complex in the fouling layer, thereby reducing membrane fouling. The increase in cell pressure was controlled within 50% as compared to the starting value, which suggests that the cell fouling is well controlled by cell cleaning strategy and the measures to treat MCDI feed. Calcium phosphate was found to be the major scalant accumulated in the cell, which can be effectively removed by the acid CIP.

Keywords: Membrane capacitive deionization (MCDI); Reverse osmosis reject; Pretreatment; Cell cleaning

1. Introduction

CDI is a technique in which an electric field is applied between two porous electrodes to remove salt from water. When there is a potential difference between the two electrodes, dissolved ions in the water are adsorbed on the porous electrodes to achieve desalination. CDI has played a role in brackish water desalination, seawater desalination, wastewater rehabilitation, and water softening [1]. The advent of membrane capacitive deionization (MCDI) is an important improvement over CDI. A cation exchange membrane (CEM) is placed at the cathode and an anion exchange membrane (AEM) is placed at the anode. Anions and cations are adsorbed on opposite charge electrodes through the ion exchange membrane, generating product streams with lower salt concentration [2,3]. During charging, co-ions are trapped in the macropores of the electrode because they cannot penetrate the ion exchange membrane. Therefore, the ion exchange membrane prevents the co-ions from entering the electrode, which makes counter-ions easy to be absorbed by electrodes, thus improving desalination efficiency [4]. MCDI combines the advantages of ion-exchange membranes and CDI, which shows great energy-saving potential compared with the traditional desalination process [3]. In addition, research in desalination technology contributes to fuel the world population for their improved living standard and to reduce specific energy consumption, and protecting the environment [5].

Reverse osmosis is the most reliable seawater desalination technology which separates dissolved solids from

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water by forcing water through a semi-permeable membrane [6]. The process produces two streams: the product water from the process and the RO reject, which is the waste stream. The RO reject treated in this study was the concentrate of secondary effluent from the biological treatment process. In addition to containing a variety of ions such as calcium, magnesium, potassium, sodium, ammonium, chloride, nitrate, sulfate, iron, and phosphate, there were also highly heterogeneous effluent organic matters (EfOMs) in the RO reject, such as polysaccharides, proteins, amino acids, nucleic acids, humic and fulvic acids, organic acids, and cellular components [7]. RO reject is usually discharged directly into the sea or into surface water, which can have serious environmental impacts and incur expensive costs [8]. These constraints are forcing industries to develop innovative approaches to RO reject management and reduce the volume of RO reject requiring disposal. The rejection produced by the dense membrane from water reclamation systems has been increasing worldwide. The reject generated from water reclamation contains a high concentration of ions and organic matter. The treatment and management of reverse osmosis rejection to become a critical water resource management problem [9]. CDI process with integrated pretreatment was demonstrated to be feasible to reduce the RO reject volume for disposal and recover water from RO reject [7,10]. Biological activated carbon (BAC) and ultrafiltration (MF) consist of integrated pretreatment to remove organic compounds and some particles from the solution to prevent CDI contamination [11]. It was found the organic compounds caused fouling on the ion-exchange membrane, which is possibly due to the movement of negatively charged compounds such as organic acids under the applied electrical field. Fouling usually causes an increase in the conductivity of the product stream and reduces the overall efficiency by increasing the volume of desorption concentrate [12]. Furthermore, organic fouling and scaling were reported to be the major cause for the pressure increase of CDI treating the RO reject with high TOC concentration, which resulted in the disruption of the CDI operation. Different chemical CIPs were carried out to restore CDI, which were found to be unsuccessful [10]. Till now, little data is available on the long-term (35 d) performance of CDI/MCDI for real water purification and the development of a cell cleaning strategy to remove foulants accumulated on the electrode and ion-exchange membrane.

In this study, the feasibility of treatment of RO reject by CDI process was verified by investigating the long-term desalting performance of MCDI. Moreover, in order to reduce the fouling of organic and inorganic compounds on the MCDI, a cell cleaning strategy was developed to restore its efficiency and extend the operation time.

2. Materials and methods

2.1. RO reject

This study used the RO reject from the second stage RO process of a water reclamation plant which recovers secondary effluent from a municipal wastewater treatment plant (WWTP). The WWTP adopted the conventional activated sludge treatment process, including the primary clarifier, aeration tank, and secondary clarifier. Ultrafiltration (UF) and reverse osmosis (RO) were used to recover the treated secondary effluent, and reverse osmosis reject accounted for 25% of the total treated water. Reverse osmosis membrane used was CPA5-LD, Hydranautics. Characteristics of the RO reject used in this study were stable as shown in Table 1.

2.2. Experiment set-up

As shown in Fig. 1, the experimental set-up used in this study for the reclamation of RO rejects consisted of BAC, microfiltration (MF), and MCDI. The BAC and MF were used as pretreatment of the MCDI process to remove organic matters and suspended solids. The packing material of the BAC column was granular activated carbon (Norit GAC 1240). The flowrate of BAC was maintained at 94.2 mL/min with an empty bed time (EBT) of 20 min. To prevent the particulate fouling of the MCDI cell, a pressurized MF unit (Memco XP-E4, Siemens Water Technologies, Singapore) was used before the MCDI. After BAC and MF, the RO reject was collected in an intermediate holding tank as an MCDI feed tank. Before feeding to MCDI, the pH of MCDI feed was adjusted to 4.7 ± 0.2 by 0.1 N hydrochloric acid (HCl), which can control inorganic scaling of MCDI unit, while sodium hypochlorite (NaClO) was dosed at 3 mg/L into MCDI feed tank, which can inhibit the breeding and reduce the biological pollution of MCDI cell. After 30 min mixing for disinfection, sodium bisulfite was added to the MCDI feed tank at the concentration of 38.5 mg/L which can prevent oxidation reaction on the MCDI electrode. The MCDI desalting performance is stable during the continuous 35 d operation with a treatment capacity of 100 L/d.

2.3. CDI process

The bench-scale MCDI (DesEL 400, ENPAR Technologies Inc., Canada) was used for pretreated RO reject desalination. The MCDI cell consisted of 10 pairs of electrodes and the

Table 1
Characteristics of the RO reject used in this study

Parameter	Concentrations
Conductivity, μS/cm	1,426–1,725
TDS, mg/L	1,025–1,287
pH	6.06–6.50
TOC, mg/L	17.1–23.7
Cations	
Sodium, mg/L	179–273
Potassium, mg/L	54.7–80.7
Ammonium, mg/L	0.15–0.54
Magnesium, mg/L	8.16–12.10
Calcium, mg/L	61.2–109.0
Iron, mg/L	0.10–0.19
Anions	
Chloride, mg/L	200–259
Nitrate, mg/L	59.8–132.1
Sulfate, mg/L	182–222
Phosphate, mg/L	34.0–46.8

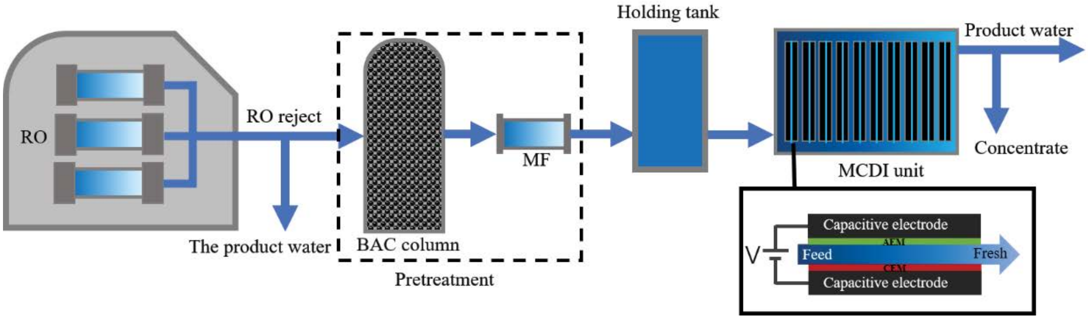

Fig. 1. Experiment set up.

total electrode area is 0.7 m^2. The desalination process of MCDI included three stages: purification, regeneration, and purge. During the purification phase, a product stream with low salt concentration was produced. By reversing the potential, the ions were released back into the aqueous solution and regeneration occurred. The aqueous solution with a higher salt concentration in the cell was purged out from the cell into a concentrated stream during the purge phase. Each cycle consisted of 1.6 min of purification (product generation), 1.7 min of regeneration, and 0.5 min of purge (concentration generation) [11]. Before the study, the applied voltage of the MCDI unit was adjusted from 1.1 to 1.7 V and the flowrate varied from 0.29 to 0.37 L/min to determine the optimum operating condition.

The MCDI was operated in batch mode in this study. For each batch, The MCDI was operated for 20 h and then flushed with 5 L of ultrapure water. Throughout this study, the water recovery of the MCDI was maintained at 75% by properly configuring its operating parameters. MCDI unit pressure was measured by the pressure gauge installed at the outlet of the MCDI and recorded daily.

2.4. MCDI cell cleaning

Chemical CIP was carried out regularly to prevent cell fouling. The details of the solution used for CIP are illustrated in Table 2. Acid CIP was to dissolve inorganic scalants at the low pH of 0.9 ± 0.1, while alkaline CIP was to remove organic by desorption of organic matters from the fouled ion-exchange membrane and electrode at the high pH of 10.25 ± 0.25. The composition of 5% NaCl in the alkaline CIP solution was to enhance the cleaning efficiency by loosening the fouling layer [13,14]. Acid CIP was carried out by circulation of 2 L acid solution two rounds with each round for 1 h, while 4 L CIP solution was circulated for 1 h by two times for the alkaline CIP.

2.5. Water quality analysis

The pH and conductivity measured with a portable meter (Model: 6Psi Ultrameter II, Myron L, USA). Install online conductivity meter (Model 8225 conductivity transmitter, Burkert fluid control systems, German) at the outlet of the MCDI cell to measure online conductivity. TDS was determined by standard methods (APHA-AWWA-WEF, 1998) [15]. Total organic carbon (TOC) was determined by a 1010 total organic carbon analyzer (O.I.Analytical, USA).

Table 2
Composition of CIP solution used in this study

Concentration	Acid CIP	Alkaline CIP
pH	0.9 ± 0.1	10.25 ± 0.25
NaCl	–	5%

The concentration of anions and cations were determined by ion chromatography (LC20 chromatography, Dionex, USA). The anions analyzed were chloride (Cl^-), nitrate (NO_3^-), sulfate (SO_4^{2-}), phosphate (PO_4^{3-}) and cations were sodium (Na^+), potassium (K^+), magnesium (Mg^{2+}), calcium (Ca^{2+}), and iron (Fe^{3+}).

3. Results and discussion

3.1. Pretreatment with BAC and MF

Characteristics of the RO reject used in this study were stable as shown in Table 1. Conductivity and TDS varied from 1,426 to 1,725 μS/cm and from 1,025 to 1,287 mg/L, respectively. TOC of the RO reject was 21.1 ± 2.4 mg/L within the range of 17.1–23.7 mg/L. The RO reject was the concentration of secondary effluent from the biological treatment process. Organic constituents in RO reject are highly heterogeneous, containing a variety of compounds as measured by dissolved organic carbon (DOC). Moreover, a high concentration of calcium cation (61.2–109.0 mg/L) was detected in the RO reject, which suggests that calcium-related fouling could take place in the MCDI process.

Fig. 2 shows the removal efficiency of TOC by BAC. The average TOC removal efficiency of BAC was 16% ± 4.1%, which was slightly lower than that of 17% ± 3.5%, which was reported for BAC treating secondary effluent [16,17]. Our BAC unit had been used to treat secondary effluent for more than 1 y without any chemical regeneration of activated carbon before this study. Therefore, biodegradation of organic matter by biofilm formed on the activated carbon should be the dominant removal mechanism [11], by which the removal rate is expected to be low as the RO reject is difficult to biodegrade.

As there were suspended solids such as slough-off biofilm and microorganisms in the BAC effluent, MF filtration was applied to minimize their adverse effect on the MCDI unit. TOC of BAC effluent was 18.9 ± 1.9 mg/L, while

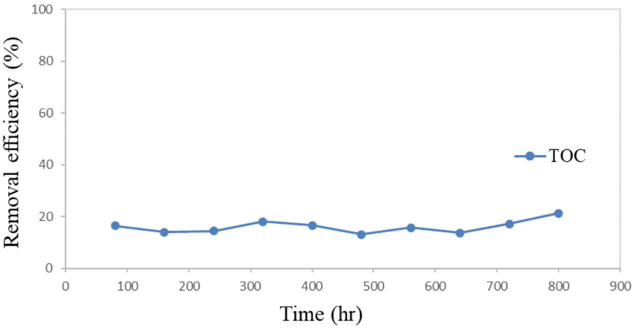

Fig. 2. Removal efficiency of TOC by BAC.

effluent of MF had a TOC concentration of 18.3 ± 1.6 mg/L. The TOC removal efficiency of MF was less than 5%, which suggests the MF unit cannot effectively remove dissolved organic matters. No chemical cleaning was conducted on the MF membrane during this study as the increase in the trans-membrane pressure was negligible. The low pH of the RO reject (6.06–6.50) could be the major reason for the low fouling rate of MF membrane as scaling potential was significantly decreased [11,18].

3.2. CDI operation condition

3.2.1 Optimum DC voltage and flowrate

Fig. 3 shows the effect of applied voltage on the MCDI. It can be seen that the optimal applied voltage was 1.3 V as the MCDI product had the best quality in terms of its conductivity. When the applied voltage increased from 1.3 to 1.5 V, the change of the conductivity of MCDI product was negligible. The theoretical voltage of water electrolysis in the standard state is 1.23 V, however, no electrolysis of water was found during the operation. The MCDI cell was composed of 10 pairs of electrodes. Between each pair of electrodes, there was a pair of current collectors, anionic-, and cationic-exchange membranes, and a spacer layer. When the voltage is applied to the MCDI cell, the potential applied to the carbon electrode significantly drops because of the sum of the resistance of each component and all the interfacial resistances [19]. Choi and Yoon [20] reported that for the fixed electrode CDI cell no electrode reactions occurred until the charge supplied to the CDI cell exceeded the maximum allowable charge of electrodes, irrespective of cell potential difference. In addition, TOC concentration of MCDI product was found to decrease by 7.2% when applied voltage increased from 1.3 V to 1.7 V, which may be the ability of the electrode to adsorb charged or polar substances in TOC increases with the increase of voltage, so the concentration of TOC decreases with the increase of voltage. Moreover, the main component of MCDI electrode material is carbon material, which has adsorption performance for organic matter.

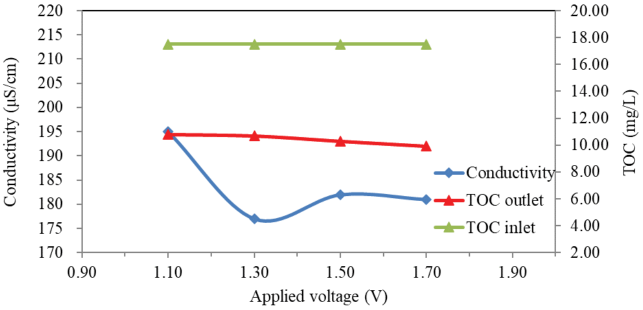

Fig. 3. Effect of applied voltage on the MCDI performance with RO reject at initial conductivity of 1,555 µS/cm.

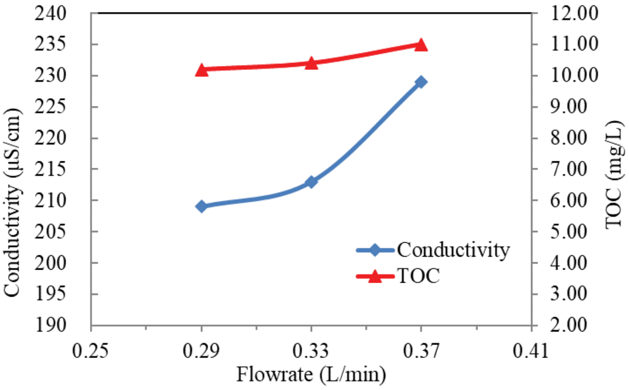

Fig. 4. Effect of flowrate on the MCDI performance with RO reject at initial conductivity of 1,555 µS/cm.

Effect of flowrate on the MCDI performance was investigated by varying flowrate from 0.29 to 0.37 L/min with fixed applied voltage at 1.3 V. Fig. 4 shows that conductivity and TOC concentration of the product increased with the increase of flowrate. Some studies have shown that it does not have enough retention time for mass transfer in the CDI cell under a high flowrate which does not synchronize with the adsorption rate of the active site [21]. Therefore, the conductivity and TOC concentration of the product increased with the increase of flowrate. The salt removal efficiency at the flowrate of 0.33 L/min was very close to that of 0.29 L/min in terms of product conductivity. Considering salt removal efficiency and MCDI process efficiency, the optimal flowrate was determined at 0.33 L/min. Based on the above experiments, the following study was conducted with an applied voltage of 1.3 V and flowrate of 0.33 L/min.

Fig. 5 shows the profile of MCDI product conductivity and stack current during its purifying phase. It can be seen that the product conductivity dropped sharply in the initial 10 s and then gradually reached a minimum value at 20 s. After that, product conductivity slowly increased with the increase of time until the end of purifying phase, indicating that ion concentration in the outflow would approach that of the inflow when the electrode reached the saturated adsorption capacity [22]. As shown in Table 3, the study indicates that when the purify duration was long enough, the MCDI electrode reached saturation with a stable electro-adsorption capacity. A different pattern was observed for the stack current, which decreased with the increase of time. It could be due to the continually increased salt concentration in the MCDI cell duriwng the purifying phase, which in turn lead to increased resistance for the ion transport and therefore a decreased stack current.

3.3. Long-term CDI desalination performance

The MCDI was operated for 850 h (~35 d) with the RO reject under the optimal operation parameters in this study. The optimal operation parameters of the MCDI unit are 1.3 V of applied voltage and 0.33 L/min of flowrate. The conductivity curves of feed, permeate, and concentrate is shown in Fig. 6. As shown in Fig. 6, the permeate conductivity was stable, and the conductivity removal efficiency was 83.1% ± 10%, which can indicate that the MCDI cell operates stably. The removal efficiency of anions and cations by the MCDI is shown in Figs. 7 and 8. At the initial 30 h, the MCDI had the highest ion removal efficiency, which could be attributed to the highest adsorption capacity of the virgin electrode. After that, the removal efficiencies for cations and anions were stable or slightly decreased throughout the study except for the ion of iron, which decreased significantly along the operation time. The removal efficiency for the ion of iron was 26.4% ± 10.5%, which were the lowest removal efficiency among all ions investigated. Ionic strength is the main factor affecting ion removal rate. The reason for the low removal rate of iron is that the concentration of iron ions is the lowest in RO reject. Phosphate had the lowest anion removal efficiency, which ranged from 45.5% to 68.7%. Slight higher removal efficiency for phosphate were reported when RO brine were treated by the CDI process [11]. Except for ions of iron and phosphate, the removal efficiency of others ranged from 68.8% to 90.3% with an average removal efficiency of 82.8% ± 6.8%. Even though CDI electrodes preferentially stored ions with higher valence and smaller hydration sizes [23,24], insignificant differences were observed in the removal of monovalent and divalent cations by the MCDI during the overall operation time. Since a few factors affect the ion removal by the

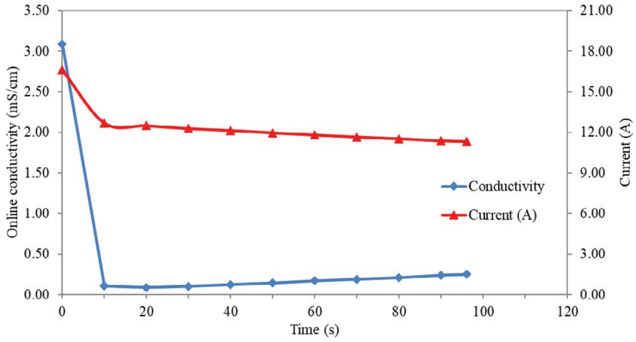

Fig. 5. Operation profiles of the MCDI during the purifying phase.

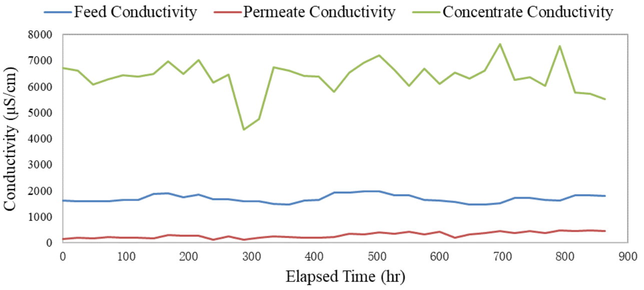

Fig. 6. MCDI feed, permeate, and concentrate conductivity vs. operation time.

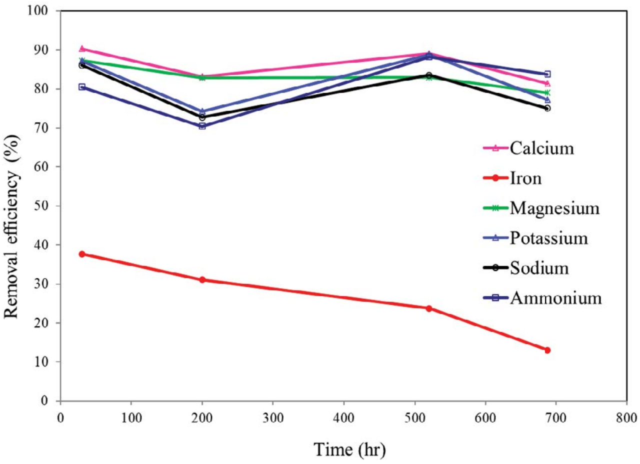

Fig. 7. Removal efficiency of cations by the MCDI along the operation time.

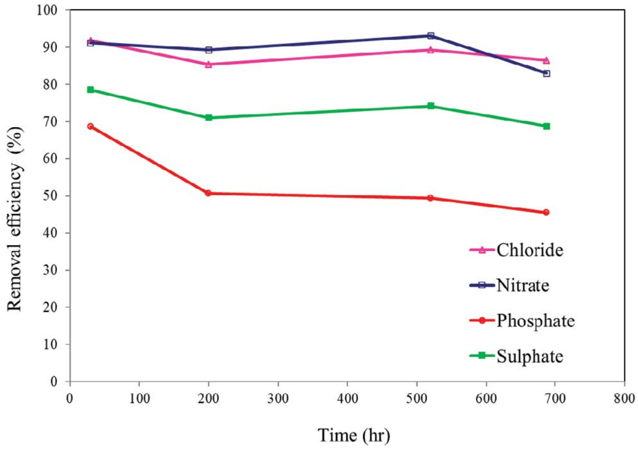

Fig. 8. Removal efficiency of anions by the MCDI along the operation time.

Table 3
Performance of MCDI cell under different purify duration

Purify duration (s)	MCDI feed conductivity (μS/cm)	MCDI permeate conductivity (μS/cm)
90	1,536	47
210	1,365	464
270	1,378	496

MCDI process, such as characteristics of ions, type of electrode, and chemistry of feed solution, further studies are necessary for the in-depth understanding of selectivity in the removal of monovalent and divalent ions.

Removal efficiency for the TDS, TOC, and silica were in the range of 72.6%–85.8%, 21.6%–28.7%, and 6.7%–16.5%, respectively (Fig. 9). With the increase of operation time, no significant change was found for the TDS, TOC, and silica removal, which agrees with the trend of ion removal efficiency. It suggests that the MCDI desalting performance is stable during the period investigated. Higher TDS and TOC removal efficiencies were reported using CDI to treat the RO reject-with more than two times lower TOC and TDS concentrations [11]. It could be attributed to the difference in the characteristics of feed stream and much longer purifying time (2.5 min) that was used for the purification by Lee's study.

3.4. MCDI cell fouling and cleaning

According to the previous study, cell fouling was the major issue for the sustainable MCDI/CDI operation. In this study, high TOC concentrations were detected in the MCDI feed steam, indicating strong organic fouling will occur and interrupt the desalting operation. The organic matter depends upon the chemical interactions to tightly bond with the electrodes. When the accumulation of organic matter onto the electrode surface lasts for a long run time, electrode pores may get blocked, thereby hindering the diffusion of ions into the interior of the electrode and release from the electrode. In addition, the natural organic matter (NOM) acids constituents are expected to be transported preferentially through an ion-exchange membrane during the CDI process because of their negative charge density. However, the molecular mass of the NOM acids was too high to allow them to pass through the ion-exchange membrane pores. It caused an accumulation or adsorption of the solutes on the membrane surface and led to membrane fouling [25]. Cleaning time selection is base on the cell pressure increase and operation time. According to Tao et al.'s [10] paper, it was found that CIP was not effective after a 290 h operation of the CDI unit. Therefore, CIP was carried out every 10 d continuous operation to prevent cell fouling even though no obvious fouling phenomena were observed.

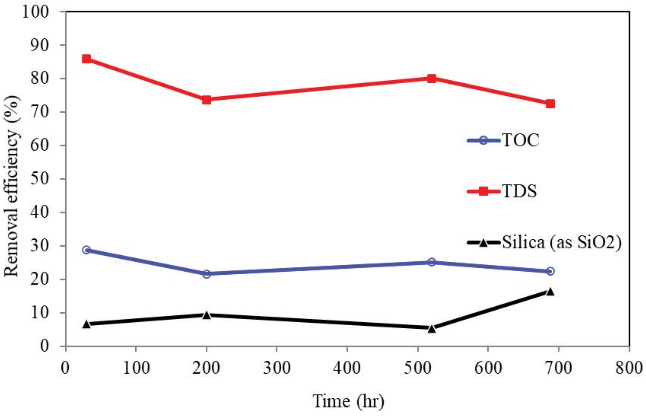

Fig. 9. Removal efficiency of TOC, TDS, and silica by the MCDI along the operation time.

Operating time and cell pressure were the two indicators to measure the cell fouling situation of the MCDI. Fig. 10 shows the cell pressure varies along the operation time. A CIP was conducted at the time of 240 h to prevent the cell from irreversible fouling after long-term operation. As can be seen, there was a sudden increase in the cell pressure from 32.4 to 51.7 kPa at 475 h, which was due to the acid and alkaline CIP conducted. As the alkaline CIP solution contained 5% NaCl, which would cause the expansion of the ion-exchange membrane, the increased headloss through the cell led to the increase in cell pressure [13]. The combined mixture of NaOH and NaCl was found to have better cleaning efficiency for membrane cleaning as compared to a single cleaning agent (NaOH or NaCl) [14]. The presence of NaCl in the cleaning solution could cause the swelling of the fouling layer and react with the polysaccharide-calcium complex in the fouling layer by ion-exchange reaction [26]. The cell pressure was observed to drop to slightly higher value in 24 h and gradually decreased

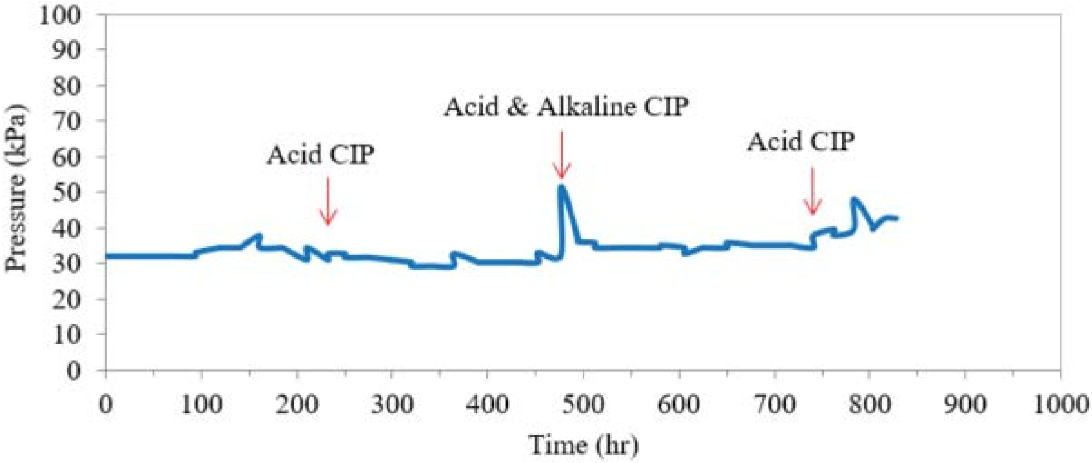

Fig. 10. Change in MCDI pressure along operation time during the purifying phase.

to the initial value, which confirmed that the expanded membrane returned to the previous condition. Except for the peak of pressure caused by the CIP, an increase in cell pressure was controlled within 50% as compared to the starting value, the minimal cell pressure. It suggests that the cell fouling is well-controlled by the measures to treat MCDI feed in our study and the MCDI process with efficient pretreatment can be an alternative solution for the RO disposal [7]. In contrast, cell pressure was reported to increase more than four times from the minimum of 35–160 kPa in 300 h of operation time using CDI to treat the RO reject [10]. As the nearly same MCDI device was used, the discrepancy in the cell pressure change could be attributed to the difference in the configuration of the CDI/MCDI device and the chemical dosing in the MCDI feed. As compared to Tao's study, a magnetic pump without any pump tubing was used to replace the peristaltic pump, which squeezed the silicon tubing equipped and might release SiO_2 colloids into the cell feed. As result, glue-like substance, which could be soluble microbial products mixed with SiO_2 colloids, was found to be the major foulants accumulated on the CDI cell [10]. In addition, pretreatment for the cell feed was enhanced by lower pH adjustment and more SBS dosing in comparison with Tao's study, which is expected to minimize the scaling and oxidation that was observed at the edge of the ion-exchange membrane in CDI cell [10].

In order to investigate the major foulants that fouled the MCDI, CIP waste was collected for analysis. Table 4 shows the composition of acid CIP waste, which was first CIP and conducted at the operation time of 232 h. As can be seen, the concentration of calcium and phosphate was 728 and 1,241.1 mg/L in the waste of 1st round circulation, respectively. After calculation, Q ($Ca_3(PO_4)_2$) = 2.64×10^{-14}, which is greater than Ksp ($Ca_3(PO_4)_2$). According to the solubility product rule, $Ca_3(PO_4)_2$ was precipitated, which indicates that calcium phosphate was the major scalant accumulated in the cell. A high concentration of TOC (20.9 mg/L) suggests the occurrence of organic fouling even though the MCDI only operated for about 10 d. It was found that inorganic scaling of calcium phosphate and organic fouling were the major cause of CDI pressure increase [10]. Furthermore, the concentration of calcium and phosphate in the 2nd round was only 11.7% and 7.8% of the 1st round, indicating inorganic scalants in the MCDI, such as calcium phosphate can be effectively removed by acid CIP.

Table 4
Composition of acid CIP waste from the MCDI cell

Parameter	Concentrations of acid CIP waste	
	1st round	2nd round
pH	1.4	1.3
TOC, mg/L	20.9	8.3
Sodium, mg/L	231.6	28.3
Potassium, mg/L	101.0	11.8
Ammonium, mg/L	0.32	0.13
Magnesium, mg/L	22.4	4.6
Calcium, mg/L	728	85
Iron, mg/L	2.3	2.2
Chloride, mg/L	3,170	2,398
Nitrate, mg/L	166.1	84.6
Sulfate, mg/L	223	327
Phosphate, mg/L	1,241.1	96.8

4. Conclusions

The 35 d continuous operation of the MCDI demonstrated that the MCDI process with integrated pretreatment can be used to recover water from RO reject and further reduce its volume. Even though RO reject contained a significant amount of inorganic and organic compounds, the desalting performance of the MCDI was stable, and the increase in cell pressure was well controlled within 50% in the period of 35 d continuous operation. CIP was carried out every 10 d to prevent cell fouling. Our results show that cell fouling was well controlled by the developed CIP strategy as an increase in cell pressure varied within 50% as compared to the starting value. Calcium phosphate was found to be the major scalant accumulated in the cell, which can be effectively removed by the acid CIP.

Acknowledgments

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