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Organic matter and dissolved salts removal in a microbial desalination cell with different orientation of ion exchange membranes

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ABSTRACT

Two microbial desalination cells (MDCs), MDC-1 having anion exchange membrane (AEM) near anode and cation exchange membrane (CEM) near cathode and MDC-2 having CEM near anode and AEM near cathode were used for removal of total dissolved solids (TDS) and chemical oxygen demand (COD) from wastewater. Performance of MDCs was evaluated at different catholyte pH (8.0 and 3.0) using 0.2 g/L of $KMnO_4$ as catholyte under dissolved salt concentrations of 8, 20, and 30 g/L in the middle chamber. Maximum Coulombic efficiency in MDC-1 (15%) and MDC-2 (11%) was observed at acidic catholyte pH. Highest TDS removal was obtained in MDC-1 (50%) having AEM near anode at higher salt concentration in middle chamber and acidic catholyte pH. Slightly lesser TDS removal was observed with alkaline catholyte pH. However, COD removal was more in MDC-2 (91%), where CEM was placed near anode.

Keywords: Anion exchange membrane; Cation exchange membrane; Microbial desalination cell; Total dissolved solids removal; Wastewater treatment

1. Introduction

Wastewater emanating from domestic and different industrial activities when discharged, untreated, or partially treated contaminate the water source, creating bad impacts on environment. The effluent from these activities contains high level of organic and inorganic matter which are pollutants, if not treated effectively before discharging into the water body. The industrial effluents from food processing industry, tannery or leather industry, petroleum refineries, paper mill industry, distillery industry, textile industry, chlor-alkali industry, pickling industry, etc. not only contain high organic matter but also contain higher dissolved solids. The salt concentration varies widely from industry to industry. For certain industries like pickling industry and industries generating phenolic waste, the salt concentration can even be as high as 70 g/L, with very high organic matter concentration in the wastewaters [1].

High dissolved salts concentration in wastewater affects the longevity of biological unit processes. Treatment of these types of saline wastewaters usually involves two separate processes, primarily biological process are employed for removal of organic contaminants, and then salts are removed in physical and chemical separation processes such as distillation, ion-exchange, electrodialysis (ED), or reverse osmosis. The technologies currently used for desalination are

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energy and capital intensive [2], and thus are not sustainable. Availability of energy source is now reducing to drive this desalination processes. So people are inclined to renewable energy sources like solar energy, wind energy, tidal energy, hydropower, etc. [3], but all these renewable energy technologies need high capital cost to install this desalination process.

In recent years, bioelectrochemcial systems (BES) such as microbial fuel cells (MFCs) are gaining much attention as it utilizes bacteria to convert chemical energy stored in the organic matter into renewable energy [4]. MFCs are capable of treating both domestic and industrial wastewaters by removing organic matter from the wastewater and producing electrical energy [5,6]. This electrical energy generated by bacterial action on anode can be utilized for removal of inorganic ions, through ion exchange membranes (IEMs)-based reactor. BES has been used recently by researchers for desalination of saline water or wastewater using ion-selective membranes [7–14]. Microbial desalination cell (MDC) is a modified form of MFC made by inserting pair of IEMs viz. anion exchange membrane (AEM) and cation exchange membrane (CEM) between anode and cathode. MDC is capable of partly or fully desalinating sea water and saline wastewater [15].

The principle of MDC is based on desalination that occurs to the liquid that is present in the middle chamber, i.e. between anodic and cathodic chamber; while bacteria oxidizes organic matter from wastewater in the anodic chamber and releases electrons to the anode and protons to the anolyte. Protons and other cations are prevented from leaving the anodic chamber by placing AEM near the anode. The balancing of ions takes place by movement of anions from middle desalination chamber to the anodic chamber. In cathodic chamber, limited protons from the catholyte combine with electrons and oxygen to form water; while the charge is balanced by movement of cations from middle desalination chamber through CEM into cathodic chamber. This concept of desalination in the middle chamber is clearly demonstrated by Cao et al. using ferricyanide as catholyte [7]. The principle of MDC for removal of dissolved salts from the liquid present in the middle desalination chamber is very similar to ED [16,17], except that the ED process needs an external applied voltage while MDCs use the produced voltage by segregating bacterial oxidation and oxygen reduction.

In most of the MDC studies, the performance of the system was improved by use of chemical catholyte to support the cathodic reduction reaction. Bioelectrochemical systems using AEM between anodic and cathodic chambers show better performance than those with CEM [18–20]. Simultaneous removal of organic matter and salt ions from the saline wastewater in the anodic chamber was also observed by placing CEM near anode and AEM near cathode [21]. This study reported the simultaneous organic matter and salt removal in the anodic chamber; while liquid present in the middle chamber gets concentrated. Voltage was applied externally to this system to reduce the dilution effect in the middle chamber because of osmosis and to produce hydrogen gas in cathodic chamber. Wastewater treatment and water desalination improves significantly by hydraulically coupling an osmotic MFC with MDC [22]. The coupled system improves the desalination efficiency through both dilution and salt removal. Recently, multiple MDCs were used in continuous flow condition to enhance the extent of desalination as compared to a single reactor [23]. The desalination performance of the MDCs can be influenced by the inter-membrane distance, as smaller inter-membrane distance results lower internal resistance of the system [24].

In the present study, the performance of two MDCs was evaluated with different orientations of membranes, MDC-1 having AEM near anode and CEM near cathode and MDC-2 having CEM near anode and AEM near cathode. Performance of these MDCs was evaluated for total dissolved solids (TDS) removal and chemical oxygen demand (COD) removal. The desalination efficiency of these MDCs was investigated with different salt concentration in the middle chamber and with different catholyte pH using potassium permanganate as catholyte.

2. Materials and methods

2.1. Reactor configuration

Two MDCs, MDC-1 and MDC-2 were fabricated with polyacrylic sheets having three chambers; anodic, middle desalination, and cathodic chamber with volume of 52 ml and width of 3 cm. The working volumes of anodic and cathodic chambers were decreased to 43 ml after inserting electrode material. The desalination chamber was separated from anodic chamber and cathodic chamber by IEMs with an intermembrane distance of 3.5 cm. In MDC-1, the desalination chamber was separated from anodic chamber by an AEM (RALEX AMH) and from the cathodic chamber by a CEM (RALEX CMH); whereas in MDC-2, CEM was placed in between anodic chamber and middle chamber and AEM separated cathodic chamber and middle chamber (Fig. 1). All three chambers along with IEMs were clamped together by rubber gaskets, providing watertight seal between these chambers. Carbon cloth (Zoltek Panex 35®, Inc., USA) was used for both anode and cathode material without any pretreatment. The two electrodes were connected by concealed copper wires with an external load resistance of 100 Ω.

The heterogeneous IEMs were preconditioned by immersing it into deionized water for 48 h before application. The thickness of each membrane was < 0.45 mm in dry condition. The ion exchange capacities of the membranes were 2.2 meq/g for CEM and 1.8 meq/g for AEM, and the resistivities of the membranes were < 160 Ω cm for CEM and < 120 Ω cm for AEM.

2.2. MDC operating conditions

Mixed anaerobic sludge collected from a septic tank bottom was used as inoculum in anodic chamber. The sludge inoculum was preconditioned and added in required volume to the anodic chamber [25]. The anodic chamber of the MDC was fed with synthetically prepared wastewater containing 3.8 g/L of sodium acetate as a carbon source in a nutrient buffer solution containing, per liter of tap water, $NaHCO_3$, 4.5 g; NH_4Cl, 0.954 g; $CaCl_2 \cdot 2H_2O$, 0.75 g; $MgSO_4.7H_2O$, 0.192 g; K_2HPO_4, 0.081 g; KH_2PO_4, 0.027 g and 3 ml of trace metals solution [25]. The middle desalination chamber of the two reactors were fed with 4, 16, and 26 g/L of NaCl along with mixed ion solution containing K^+, Ca^{2+}, Mg^{2+}, Cl^-, SO_4^{2-}, PO_4^{3-}, etc. to make the TDS concentrations of 8, 20, and 30 g/L, respectively. The cathodic chambers were fed with 0.2 g/L $KMnO_4$ prepared using tap water and pH of 8.0 and 3.0. The two MDCs were operated for more than 90 d at an ambient temperature of 25 ± 3°C under each experimental condition with a feeding frequency of 4 d.

2.3. Analyses and calculations

The samples were collected from anodic chamber using pipette from the two reactors for analysis of initial and final COD and it was determined by closed reflux colorimetric method as described in Standard Methods [26]. The TDS, conductivity, and pH inside these three chambers were measured at an interval of 24 h in each experimental run. Liquid pH in all the chambers was measured by inserting the pH electrode (Cyber Scan, pH 620, Eutech Instruments, Singapore) through the openings provided at top of each chamber. Similarly, the TDS and conductivity were measured by inserting the conductivity probe (Cyber Scan, CD 650, Eutech Instruments, Singapore) through the respective openings at the top of each chamber.

A digital multimeter with data acquisition unit (Agilent Technologies, Malaysia) was used to measure potential and current across the external resistor of 100 Ω. The anode and cathode potential was measured using Ag/AgCl reference electrode (Bioanalytical Systems Inc., USA). The Coulombic efficiency (CE, %) is the fraction of electrons transferred to the anode to the total electron released by substrate consumption. CE of the MDCs operated under feed batch mode over a time period of t was calculated [27] as per Eq. (1).

$$\mathrm{CE} = \frac{8\int_0^t I dt}{F V_{\mathrm{an}} \Delta\mathrm{COD}} \tag{1}$$

where I = electric current; 8 is a constant used for COD, based on molecular weight of O_2; F, Faraday's constant = 96,485 C/mol; V_{an} = the volume of anolyte; ΔCOD is the difference in the influent, and effluent

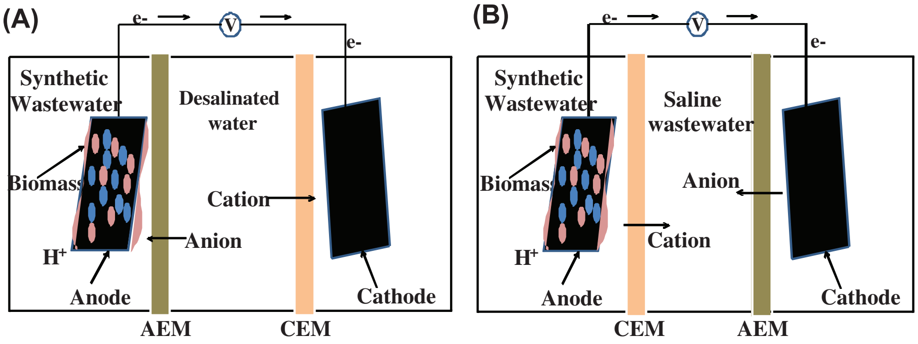

Fig. 1. Schematic diagram of two MDCs: (A) MDC-1 having AEM near anode and CEM near cathode; (B) MDC-2 having CEM near anode and AEM near cathode.

COD. Polarization study was performed by varying the external resistance from 40,000 Ω to 1 Ω. The slope of the line of voltage vs. current obtained from the polarization was treated as the internal resistance.

The desalination efficiency was determined as the percentage of dissolved salt concentration decreased over time of 96 h of batch cycle. Faradaic efficiency or current efficiency (η_F) of the system was calculated as the ratio of the theoretically necessary amount of coulombs (Q_t) required in order to remove the NaCl to the coulombs harvested through the electrical circuit ($Q = \int I\,dt$, assuming that removal of one mole of NaCl will require one mole of electrons [11,12,14,24]. The faradaic efficiency of the system was calculated as per Eq. (2) [28].

$$\eta_F = \frac{Q_t}{Q} \tag{2}$$

3. Results and discussion

3.1. Desalination performance

The removal of TDS under different salt concentrations in the middle chamber was observed in both of these MDCs for 96 h of batch cycle. Highest TDS removal was observed with 30 g/L of dissolved salts concentration in the middle chamber of both MDC-1 and MDC-2 as compared to 8 and 20 g/L of dissolved salts concentrations. TDS removal of 50 ± 3.5% and 46 ± 4.2% was observed in MDC-1 and MDC-2 at acidic catholyte pH and 30 g/L of TDS concentration in the middle chamber (Fig. 2). The TDS removal was higher in MDC-1 than MDC-2 under all conditions of catholyte and different concentrations of salt solution in middle chamber. The maximum output voltage (OV) of 180 mV and 110 mV was observed across a 100 Ω external resistor as in MDC-1 and MDC-2, respectively. Gradual decline of the OV was observed in each of the batch cycle with time of operation. It may be due to the increase in internal resistance during desalination process in the middle chamber and consumption of permanganate catholyte as electron acceptor. Such decline in voltage produced could be reduced by decreasing the size of desalination chamber and using air cathode [29]. A maximum TDS removal of 74 and 71% was observed for an extended time of batch cycle (200 h) in MDC-1 and MDC-2, respectively.

3.1.1. Effect of different orientation of IEMs

In MDC-1, the TDS removal was more under all the conditions of catholyte and different salt concentrations in saline water admitted to middle chamber as compared to MDC-2. Previous studies reported high TDS removal performance in middle desalination chamber when AEM was placed near anode [8–10,12,30]. In MDC-1, the AEM placed near anode balances the cationic charge by transportation of negatively charged ions from middle chamber to anodic chamber and avoid H^+ accumulation in anodic chamber. The phosphate ions can carry the protons and buffered the pH drop in anodic chamber and hence increases the performance of the system [31]. In MDC-2, the CEM was placed near anode which restricted the movement of anions from middle desalination chamber to anodic chamber. Since anode is positively charged, it resisted movement of cations from middle desalination chamber to anodic chamber and allowed the transfer of cations from anodic chamber to middle chamber. Similarly, AEM placed near cathode restricted cations movement from middle chamber to cathodic chamber. Instead of desalination, the liquid in middle chamber should get more concentrated in MDC-2. However, due to concentration gradient between middle and anodic/cathodic chambers, the dissolved salt concentration declines due to dilution caused by osmosis effect in MDC-2 and extent of dilution was more with high dissolved salt concentration of 30 g/L in middle chamber.

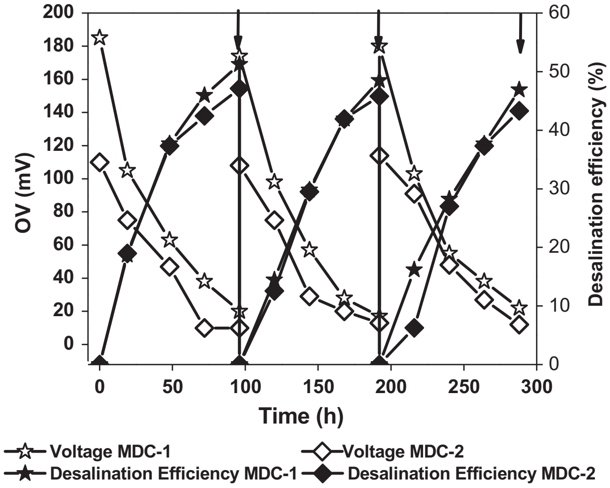

Fig. 2. Desalination performance of MDC-1 and MDC-2 with voltage production over the batch cycles. Arrow shows change of electrolyte in MDCs.

3.1.2. Effect of different TDS concentration

In MDC-1, the TDS removal was higher as compared to MDC-2 for all the TDS concentrations tested, and the extent of TDS removal was higher with high dissolved salt concentration (30 g/L) as compared to 8 and 20 g/L. The variation in TDS concentration in all three chambers of MDC-1 with 8, 20, and 30 g/L of initial TDS concentration in the middle desalination chamber is shown in Fig. 3. Similar increment in TDS concentration of anolyte was observed even in MDC-2. Resistance forces such as: membrane resistance, electrical double layer resistance, and diffusion boundary layer resistance restricts the transport of ions from middle chamber to anodic and cathodic chambers [32,33]. Therefore desalination process was not achieved fully. The overall internal resistance of the system strongly increases with decreasing salt concentration. During polarization of MDC-1 and MDC-2, the internal resistances of 1,362 and 1,500 Ω, respectively, were observed with 30 g/L of initial TDS concentration. Higher internal resistances of 3,500 and 3,650 Ω, respectively, were observed while using initial TDS concentration of 8 g/L. At low salt concentrate solution, the diffusion boundary layer resistance (caused by concentration polarization) is the dominant resistance that determines the overall resistance of the system. However, at high salt concentrate solution, the resistance force decreased due to compression of interfacial electrical double layer, thickness of which is in nanometer scale [34]. At initial TDS concentration of 30 g/L, a higher salinity gradient between the middle desalination chamber and the adjacent anode/cathode chamber intensified the dilution effect as compared to 20 and 8 g/L of TDS concentration [24]. Hence, due to lower internal resistance and higher dilution effect, the TDS removal was more under high salt concentration in the middle chamber.

3.1.3. Effect of different catholyte conditions

TDS removal in the middle chamber of both of the MDCs was highest under acidic $KMnO_4$ as catholyte compared to alkaline $KMnO_4$. The standard oxidation potential of the permanganate is higher in acidic condition than in alkaline conditions, as shown in Eq. (3)–(5) [35]. Under acidic conditions the half-reactions are:

$$MnO_4^- + 4H^+ + 3e^- \rightarrow MnO_2 + 2H_2O \quad (E_0 = 1.68V) \tag{3}$$

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O \quad (E_0 = 1.51V) \tag{4}$$

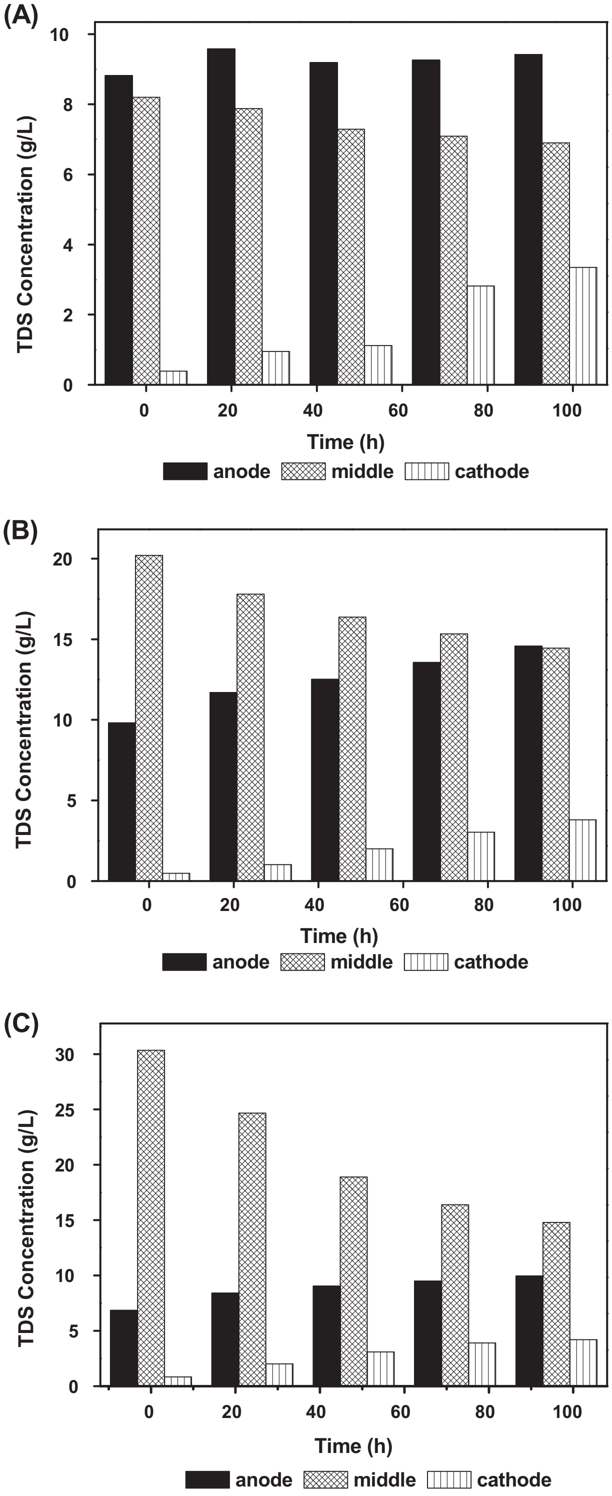

Fig. 3. TDS concentration in anode, middle and cathode chamber with (A) 8 g/L, (B) 20 g/L and (C) 30 g/L of total dissolved salts concentration in middle desalination chamber in MDC-1.

Under alkaline conditions the half-reaction is:

$$MnO_4^- + 2H_2O \ + 3e^- \rightarrow MnO_2 + 4OH^- \quad (E_0 = 0.60V) \tag{5}$$

Under both acidic and alkaline conditions, permanganate (Mn^{7+}) accepts three electrons and thus reduced to manganese dioxide (MnO_2), which precipitates out of solution. When permanganate was added as cathodic electron acceptor, the pH of the catholyte was 7.5. The maximum open circuit voltage (OCV) of 1.02 V was obtained with maximum cathode potential of 0.514 V. The cathode potential gradually reduced to 0.10 and 0.08 V after 4 d of operation in MDC-1 and MDC-2, respectively. Similarly under acidic catholyte condition (pH of 3.0), the maximum OCV and cathode potential were 1.204 and 0.564 V, respectively. The cathode potential then gradually reduced to 0.094 and 0.109 V after 4 d with increase of pH (8.5 and 8.27) due to cathodic reaction in MDC-1 and MDC-2, respectively. This decrease in voltage may be due to the permanganate consumption in the catholyte and also due to increment in pH of the catholyte with migration of ions. It was reported earlier that with increase of pH, the voltage gets dropped [36]. The desalination efficiency reduced with decrease in cell potential.

3.2. COD removal and CE

The COD removal in anodic chamber was observed for 96 h of each batch cycle with different catholytes and 8, 20, and 30 g/L of dissolved salt concentration in the middle chamber. The performance of COD removal was not affected by different dissolved salt concentration in middle desalination chamber due to migration of ions depending on IEM placed; however, it was slightly affected by the catholyte pH (8.0 and 3.0). Under acidic catholyte pH, COD removal was slightly higher than the alkaline catholyte in both the MDCs (Table 1). The COD removal in MDC-2 was more than MDC-1 under all the operating conditions. In MDC-2, the protons from anode chamber were allowed to pass to middle chamber through CEM, that helped in maintaining pH of anolyte and favored the bacterial activity. However, higher CE was observed in MDC-1 as compared to MDC-2 under all dissolved salt concentrations and catholyte conditions. Maximum CE of MDC-1 with AEM near anode was 15%, which was higher than MDC-2 (11%) in acidic condition of catholyte (Table 1). Kim et al. [31] also found higher power generation and CE using AEM than with Nafion and CEM in the MFC. Improved CE was reported with AEM near anode, due to better proton charge transfer facilitated by phosphate anion, as compared to system using CEM as a separator.

3.3. Faradaic efficiency with different TDS concentrations

Faradaic efficiencies of the two MDCs increased with increasing TDS concentration of the liquid present in the middle chamber. Faradaic efficiencies were 46, 280, and 355% with 8, 20, and 30 g/L of TDS concentration in the middle chamber in MDC-1; while in MDC-2 faradaic efficiencies were 48, 370, and 570% with 8, 20, and 30 g/L, respectively. It was assumed that 1 mol of NaCl removal would require 1 mol of electron. In MDC-1, with AEM near anode, lower faradaic efficiencies were observed under all TDS concentrations in the middle chamber compared with MDC-2 (CEM near anode). The theoretical coulombs required in the MDC-1 to facilitate the observed salt removal were 2.8 and 3.5 times higher than the actual coulombs observed in the system with 20 and 30 g/L of TDS concentration, respectively; while in MDC-2 for respective TDS concentration, the theoretical values were 3.7 and 5.7 times higher than the actual coulombs harvested. Under low TDS concentration (8 g/L), the faradaic efficiency was significantly less as compared to operation under higher TDS concentrations in both of the MDCs due to higher resistance offered by the systems for charge transfer. The faradaic efficiencies of more than 100% in both these MDCs at high TDS concentrations (20 and 30 g/L) indicate that the TDS removal in middle chamber may be caused by diffusion of ions from desalination chamber to anodic and cathodic chamber due to higher salt concentration gradient. Previous studies also proved contribution of this mechanism in

Table 1
COD removal and CE obtained in MDC-1 and MDC-2

	COD removal (%)		Coulombic efficiency (%)	
Catholyte condition	MDC-1	MDC-2	MDC-1	MDC-2
0.2 g/L of $KMnO_4$ with pH of 8.0	82 ± 2.8	89 ± 3.2	13.2	9.2
0.2 g/L of $KMnO_4$ with pH of 3.0	88 ± 1.7	92 ± 2.6	15.0	11.4

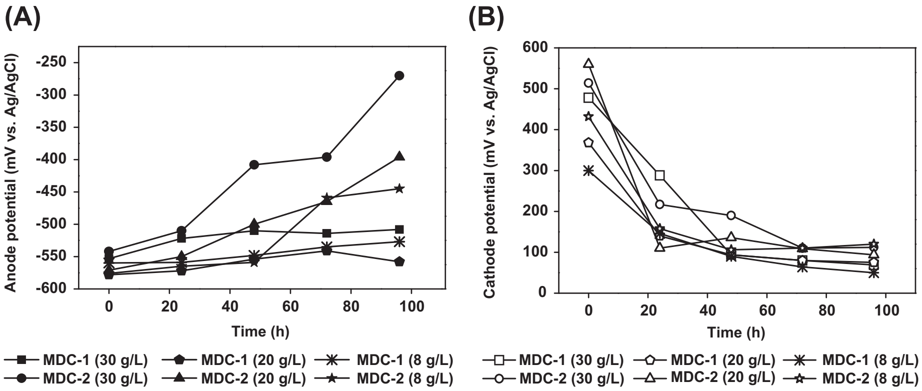

Fig. 4. (A) Anode and (B) Cathode potential of MDC-1 and MDC-2.

desalination process other than electric current produced by the system itself [9,11]. Other processes such as water osmosis, dialysis, or ion exchange can also cause TDS reduction in the middle desalination chamber. It was reported that with higher initial salt concentration in the desalination chamber, the dilution effect contribute significantly over the electric current produced by the system due to higher salinity gradient between the middle chamber and adjacent anode/cathode chamber [24].

3.4. Electrode potentials

The anode potential of MDC-1 and MDC-2 were initially similar under all salt concentrations in the middle chamber (Fig. 4(A)). The anode potential gradually increased in MDC-2 where CEM was placed near anode; however in MDC-1, there was no significant change in anode potential throughout the batch cycle. Increase in anode potential was more with higher salt concentrations (20 and 30 g/L) than low (8 g/L) salt concentration. This increase in anode potential in MDC-2 indicates that the entry of cation from middle desalination chamber to the anodic chamber was detrimental for the anodic reaction. Increment in TDS concentration of anolyte was observed even in MDC-2. It is suspected that this change in cations in the anolyte might have adversely affected the metabolism of the electrogenic bacteria on the anode biofilm and hence increasing its potential. The cathode potential of both MDCs also decreased gradually due to the decrease in concentration of permanganate used as a catholyte and also due to increase in pH of catholyte with time of operation (Fig. 4(B)). Upon replacing the catholyte, the cathode potential used to be restored.

3.5. Effect of catholyte pH

The pH of anolyte, catholyte, and liquid from middle desalination chamber for both MDCs were observed in each of the batch cycle. It was observed

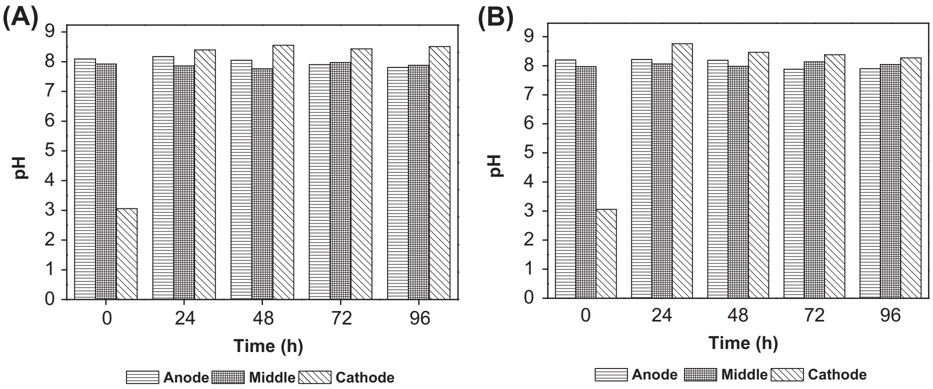

Fig. 5. pH of anolyte, middle chamber and catholyte in (A) MDC-1 and (B) MDC-2.

that the pH of the liquid in all three chambers was more than 7.0 at the end of each run. However; when the pH of catholyte was acidic (pH 3.0) initially in both of the reactors, even in this case within 24 h of experimental run, the pH of catholyte increased (Fig. 5). Slight increase in TDS removal was observed from the liquid present in the middle chamber when pH of catholyte was acidic as compared to alkaline catholyte pH. Under acidic condition, $KMnO_4$ has more oxidizing effects (capable of taking more electrons) than in alkaline condition. The COD removal performance was also higher under acidic catholyte than that observed with alkaline catholyte (Table 1). No drop of pH was observed in anodic chamber of MDC-1 due to proton accumulation. It might be due to the HCO_3^- ion transported from middle chamber to anode chamber through AEM which maintained the anolyte pH in MDC-1. Also, the phosphate ions can carry the protons and buffered the pH drop in anodic chamber.

4. Conclusions

MDC having three chambers with AEM near anode exhibits better desalination performance as compared to CEM near anode. MDC with AEM near anode also shows higher CE than CEM near anode; however the COD removal is more when CEM is placed near anode. Higher TDS in the middle chamber reduces the internal resistance of the system, hence demonstrating better desalination performance. Permanganate in acidic condition as catholyte shows better desalination performance as compared to alkaline condition. It is essential to improve performance of the cathode to improve desalination efficiency. Irrespective of type of ion-selective membrane used between anodic chamber and middle chamber, organic matter removal and TDS removal can occur. High inter-membrane distance prevented the system to achieve highest desalination performance. Further studies are necessary to improve MDC design and understand the effect of operating parameters for optimizing power generation and TDS removal.

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