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Use of a Liter-Scale Microbial Desalination Cell As a Platform to Study Bioelectrochemical Desalination with Salt Solution or Artificial Seawater

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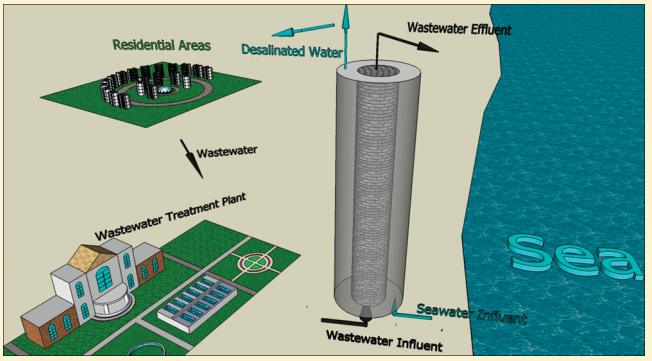
Supporting Information

ABSTRACT: Bioelectrochemical desalination is potentially advantageous because of bioenergy production and integrated wastewater treatment and desalination. In this work, the performance and energy benefits of a liter-scale upflow microbial desalination cell (UMDC) were evaluated. The UMDC desalinated both salt solution (NaCl) and artificial seawater, and the removal rate of total dissolved solid (TDS) increased with an increased hydraulic retention time, although TDS reduction in artificial seawater was lower than that in salt solution. Our analysis suggested that electricity generation was a predominant factor in removing TDS (more than 70%), and that other factors, like water osmosis and unknown processes, also contributed to TDS reduction. It was more favorable given the high energy efficiency, when treating salt solution, to operate the UMDC under the condition of high power output compared with that of high current generation because of the amount of energy production; while high current generation was more desired with seawater desalination because of lower salinity in the effluent. Under the condition of the high power output and the assumption of the UMDC as a predesalination in connection with a reversal osmosis (RO) system, the UMDC could produce electrical energy that might potentially account for 58.1% (salt solution) and 16.5% (artificial seawater) of the energy required by the downstream RO system. Our results demonstrated the great potential of bioelectrochemical desalination.



INTRODUCTION

The recent discovery of microbial desalination cells (MDC) has drawn great attention because it provides a promising approach for sustainable wastewater treatment and low-cost desalination.[1] The MDC process is accomplished in a bioelectrochemical reactor modified from microbial fuel cells (MFCs). Both MDCs and MFCs share the same principle of bioelectrochemical reactions: organic oxidation on the anode electrode supplies the electrons and protons that reduce the terminal electron acceptor (e.g., oxygen) on the cathode; thereby producing electricity when electrons flow between the two electrodes.[2] The unique feature of MDCs is an additional chamber containing saline water installed between the anode and the cathode, forming a bipolar process: when electricity is generated, cations in saline water move to the cathode while anions migrate into the anode, thereby reducing TDS (total dissolved solids) concentration.[3] Integrating wastewater treatment with desalination within MDCs allows bioelectricity produced from wastewater to be a driving force for desalination and incorporates salt removal as a part of the energy-producing process. MDCs can be either used as a predesalination process before conventional desalination to reduce salinity and the amount of energy required by downstream processes,[4] or used as a sole process for decentralized treatment.

Previous studies have focused on fundamental understanding of the MDC process. Cao et al. [3] proposed the first proof-of-concept that desalination could be achieved in an MDC. They experimentally found that more than 90% of NaCl was removed at an initial concentration of 35 g/L, and these results provided the basis for further development of MDCs. Mehanna et al. [4] replaced the ferricyanide cathode used by Cao et al. [3] with an air cathode. The air cathode is more sustainable than ferricyanide in practical application, but the study with the air cathode achieved lower desalination efficiency (43−67%). Our previous experiment developed a continuously operated upflow MDC (UMDC), which eliminated the cathode chamber by using a wet cathode directly exposed to the air.[5] The UMDC removed more than 99% of TDS at an initial NaCl concentration of 30 g/L. A recent study found that the total desalination rate can be significantly

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increased by adding more desalination cells in an MDC.[6] MDCs were also employed to produce hydrogen gas with a satisfactory energy recovery through a process similar to that in microbial electrolysis cells (MECs).[7−9] One of the key issues expected during MDC development is the reactor scale up, similar to that during MFC development. It has been found that scaled MFCs cannot maintain the same power density as that of the smaller-scale MFCs, suggesting that new problems appear at larger scales.[10,11] Therefore, while many efforts study fundamental issues with small-scale reactors, additional research with larger-scale MDCs (e.g., liter-scale) is critical for revealing and investigating new problems.

In this work, we developed the previously described UMDC to the liter-scale (total volume of 2.75 L) and attempted to use this L-scale UMDC to study desalination with either salt solution (NaCl) or artificial seawater. During a period of more than eight months, the long-term stability of this L-scale UMDC was examined through operation and various tests. The change in the conductivity of the salt solution and artificial seawater was monitored at different hydraulic retention times throughout the testing period. Power output was measured using a polarization technique. We investigated the effects of electricity generation and water osmosis on reducing TDS concentrations, and analyzed the potential energy benefits by using the UMDC.

MATERIALS AND METHODS

UMDC Setup. The UMDC was constructed as a tubular bioreactor with two compartments (the anode and salt chamber), which is an enlarged version of the UMDC reactor used in our previous study. The schematic of the UMDC is shown in Figure S1of the Supporting Information, SI, or in our previous publication.[5] Carbon brushes (Gordon Brush Mfg. Co., Inc., Commerce, CA) were used as the anode electrode materials in the present work instead of the graphite granules used in our previous study. The UMDC consisted of one anode tube made of anion exchange membrane (AEM, AMI-7001, Membrane International, Inc., Glen Rock, NJ) within a larger cation exchange membrane tube (CEM, CMI-7000, Membrane International, Inc.). The diameters of the AEM and CEM tubes were 6 and 7 cm, respectively, and the effective lengths of both tubes were ∼70 cm, resulting in an anode liquid volume of 1.9 L (excluding the anode electrode) and a saline water volume of 0.85 L.

A catalyst mixture (Pt/C power with Nafion solution) was applied to the outer surface of the CEM to obtain a Pt loading rate of ∼0.4 mg Pt/cm^2, then covered by two layers of carbon cloth (Zoltek Companies, Inc., St. Louis, MO) that acted as both the cathode electrode and current collector. A Pt wire connected the cathode electrode to the external electric circuit. The external resistance was set at 0.1 Ω, which was controlled by a high-accuracy decade box (HARS-X-3−0.001, IET Laboratories, Inc., Westbury, NY). During desalination, anions (e.g., Cl^-) moved into the anode compartment via the AEM and cations (e.g., Na^+) migrated to the cathode side through the CEM.

Operating Conditions. Synthetic wastewater containing acetate as a carbon source was fed into the anode compartment from the bottom of the UMDC and the treated water (effluent) was discharged from the top. The flow rate was 4 mL/min, resulting in a hydraulic retention time (HRT) of ∼8 h. The anode solution was recirculated at 200 mL/min. The synthetic wastewater contained (per L of tap water): sodium acetate, 3 g; NH_4Cl, 0.15 g; NaCl, 0.5 g; $MgSO_4$, 0.015 g; $CaCl_2$, 0.02 g; KH_2PO_4, 0.53 g; K_2HPO_4, 1.07 g; yeast extract, 0.1 g; and trace element, 1 mL.[12] The anode of the UMDC was inoculated with a mixture of aerobic and anaerobic sludge from local wastewater treatment plants (Jones Island Wastewater Treatment Plant and South Shore Wastewater Treatment Plant, Milwaukee, WI). Salt solution (35 g/L) was prepared by dissolving NaCl in tap water. Artificial seawater (35 g/L) was prepared by dissolving aquarium sea salts (Instant Ocean, Aquarium Systems, Inc., Mentor, OH) in tap water. The salt solution or artificial seawater was fed into the salt compartment of the UMDC in an upflow mode and the flow rate was adjusted to obtain the desired HRTs. Catholyte was the acidified water (adjusted with sulfuric acid) at a pH of 2.5 and was used to rinse the cathode electrode from the top to the bottom at a flow rate of 4 mL/min.

Measurement and Analysis. The cell voltage was recorded every 3 min by a digital multimeter (2700, Keithley Instruments, Inc., Cleveland, OH). The pH was measured using a Benchtop pH meter (Oakton Instruments, Vernon Hills, IL, USA). The conductivity was measured by a Benchtop conductivity meter (Mettler-Toledo, Columbus, OH). The concentration of chemical oxygen demand (COD) was measured using a colorimeter according to the manufacturer's procedure (Hach DR/890, Hach Company, Loveland, CO). The polarization curve was performed by a potentiostat (Reference 600, Gamry Instruments, Warminster, PA) at a scan rate of 0.1 mV/s. The maximum power density was calculated based on the anode liquid volume. To determine the theoretic NaCl removal as a result of current generation, we assumed that 1 mol of NaCl removal will require 1 mol of electrons. The TDS removal rate (g TDS L^{-1} d^{-1}) was calculated by the TDS removal per day (g d^{-1}) based on the volume (L) of either salt solution (salt compartment) or wastewater (anode compartment). The additional water flux in the salt compartment was determined by measuring the difference of the volume between the saline water influent and its effluent over the time.

It should be noted that it is not always accurate to use "conductivity" to represent salt concentration (TDS). Under certain conditions, for example, when ion exchange occurs and protons exchange sodium ions, TDS will decrease but conductivity will increase because of the stronger movement of protons than sodium ions. To simplify the scenario, conductivity was used here to reflect TDS.

Energy Estimation. The estimated energy requirement by reverse osmosis (RO) is based on a constant driving force and recovery rate of 50%. The energy to transport seawater from the sea to the pretreatment is assumed as 1.5 kWh/m^3. The energy requirement was calculated according to the following equations:[13]

$$E = 1.5 + \frac{(\mathrm{PI}/R + 20)x}{70} \quad (1)$$

$$\mathrm{PI} = \frac{25x}{3.5} \quad (2)$$

where E is the energy requirement (kWh/m^3), PI is osmotic pressure (bar), R is the recovery rate (50%) and x is salinity (e.g., 3.5 for 35 g/L).

RESULTS AND DISCUSSION

Desalination of Salt Solution or Artificial Seawater. During the operating period, the UMDC was capable of desalinating both salt solution (containing NaCl) and artificial seawater (containing

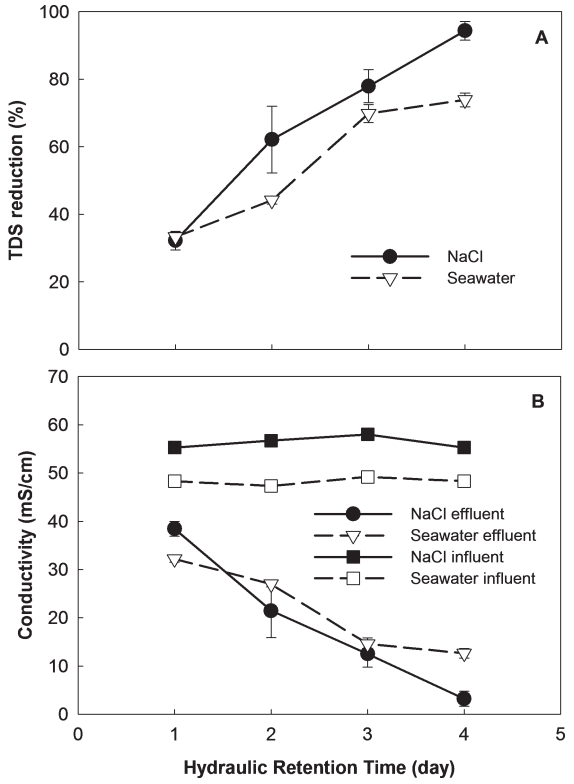

Figure 1. TDS reduction in salt solution and artificial seawater (A) and conductivity of the influents and effluents in the UMDC (B) at different HRTs of saline water.

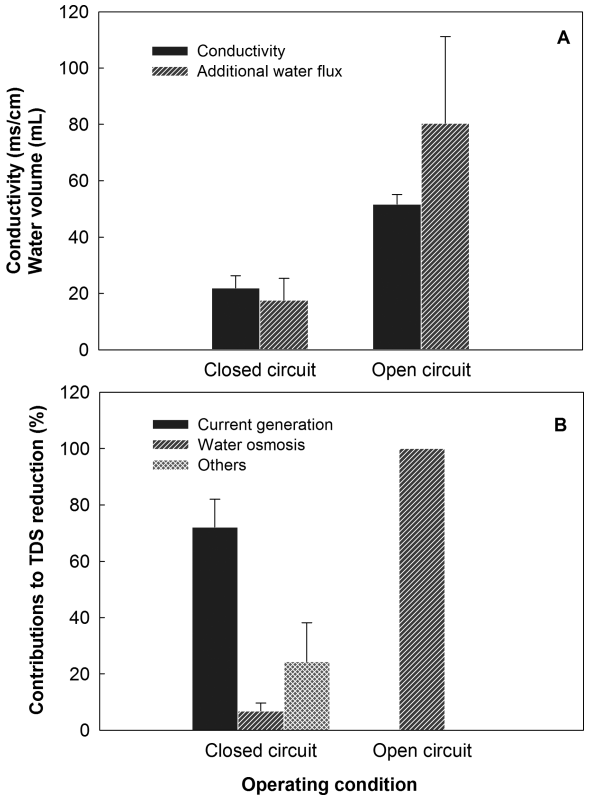

Figure 2. Comparison between the closed circuit (0.1 Ω) and open circuit: (A) conductivity of salt solution and additional water flux into the salt compartment; and (B) estimate of different contributions to reduce TDS. The HRT of salt solution was two days.

sea salts) with a notable difference in performance. The TDS reduction with both saline waters increased with an increasing (saline water) HRT (Figure 1A). At the HRT of 4 d, the UMDC removed 94.3 ± 2.7% and 73.8 ± 2.1% of the TDS contents in salt solution and artificial seawater, respectively. Accordingly, the conductivity of the effluents reached the lowest of 3.2 ± 1.5 mS/cm and 12.6 ± 1.0 mS/cm for two saline waters (Figure 1B). It should be noted that the influents of the saline waters contained different conductivities: 56.7 ± 1.4 mS/cm for salt solution and 48.3 ± 0.9 mS/cm for artificial seawater. The TDS removal rate for salt solution was 11.61 ± 1.69 g TDS L^{-1} d^{-1} (salt solution volume) or 5.20 ± 0.75 g TDS L^{-1} d^{-1} (wastewater volume). The removal rate for artificial seawater was 9.99 ± 2.61 g TDS L^{-1} d^{-1} (seawater volume) or 4.47 ± 1.17 g TDS L^{-1} d^{-1} (wastewater volume). Meanwhile, the UMDC removed 92.0 ± 0.4% of COD in its anode at the loading rate of 6.78 ± 0.36 g COD L^{-1} d^{-1}, irrespective of salt solution or artificial seawater.

Compared with our previous UMDC, the present UMDC maintained a similar TDS removal rate based on wastewater volume, or it improved the TDS removal based on salt solution volume, while the volume of the reactor was about three times larger. This is a positive indication that performance may be maintained at the similar level with MDC scaling up. The improved TDS removal rate based on salt solution volume was likely due to a larger ratio between the wastewater volume and salt solution volume (2.2:1) than that in our previous study (1.4:1).[5] As previously discussed, a larger ratio between the two volumes will benefit salt removal; the detailed reasons remained unclear, but we could attribute the better performance to less salt accumulation in the anode due to a larger flux of the anolyte, a sufficient organic supply for providing electrons, and a larger membrane surface for facilitating ion exchange. A detailed comparison between the UMDC and other smaller-scale MDCs can be found in our previous study;[5] however, it should be noted that the obvious difference in the reactor scale and configuration can greatly affect MDCs' performance.

Although MDCs are intended for desalinating seawater, previous studies did not investigate seawater. Our results demonstrated that seawater could be desalinated as expected, but at a lower efficiency than NaCl solution. The highest TDS removal with artificial seawater was about 20% less than NaCl solution in this study (Figure 1A). The lower efficiency was related to the complex composition of seawater. In addition to the predominant species such as Na^+ and Cl^-, seawater also contains Ca^{2+}, Mg^{2+}, SO_4^{2-}, K^+, and other various dissolved and suspended components. The lower conductivity of seawater compared with NaCl solution at the same concentration (35 g/L) resulted in a higher ohmic resistance of 6.69 Ω compared with 5.94 Ω with the NaCl solution. It also suggests the presence of nonconductive compounds in seawater such as silica and clay in a very fine or colloidal form.[14] Some of those compounds may form precipitation on the surface of the membrane and cause membrane fouling. Long-term operation may also introduce microbial growth and biofouling. Although we do not expect membrane fouling to be as serious as that in conventional desalination (e.g., RO) because of the different mechanisms of ion movement (ion exchange in MDCs vs filtration in RO), it is certainly of interest and requires further investigation.

Contributions to TDS Reduction. We have attempted to understand the factors that control TDS reduction in the UMDC. During the experiments, we observed additional water flux from the salt compartment; that is, more water flowed out of the salt compartment than what we fed in. The measurements at a HRT 2 d with NaCl solution showed that there was an additional water flux of 17.6 ± 7.7 mL and 80.4 ± 30.7 mL under the closed- and open-circuit conditions, respectively (Figure 2A). The added water was likely the result of water osmosis from both the anode and the cathode into the salt compartment due to the gradient of salt concentrations across the ion exchange membranes. The higher conductivity of 51.7 ± 3.5 mS/cm under the open-circuit condition compared with 21.9 ± 4.4 mS/cm under the closed-circuit condition, demonstrating that current generation could stimulate TDS removal in the UMDC. Consequently, the open-circuit condition had a higher gradient that tended to cause more water flux into the salt compartment to dilute salt solution, which might be why we still found about a 6% reduction in conductivity in the absence of current generation. Water osmosis will not remove TDS, but it will lower the TDS concentration through the dilution effect.

Factors included in our analysis were electric current, water osmosis, and others such as dialysis and ion exchange. The results suggested that under the open-circuit conditions, TDS reduction was primarily due to water osmosis. With the closed circuit, electric current accounted for 72.2 ± 9.9% of the reduction in TDS, water osmosis contributed 6.8 ± 2.8% in reducing TDS concentration, and the rest (24.4 ± 13.8%) was from others (Figure 2B). The data demonstrated that desalination was not the sole result of current generation, which confirmed the findings of Mehanna et al.;[4] however, others observed that MDCs generated more current than required for TDS reduction. In Cao et al.,[3] about 50% of the generated current was associated with salt removal. Our previous UMDC at a smaller scale [5] achieved 81−98% of charge transfer efficiency. Regardless of the difference in charge transfer efficiency, in both studies more than enough current was produced for desalination and salt reduction due to other factors was not observed. It is not clear at this time why current generation lags behind the desalination in some MDCs. The increased scale or inefficient configurations may create an additional niche for water osmosis and other unknown processes to contribute to desalination.

Water osmosis in the UMDC, although not significant under the closed-circuit condition, could be potentially beneficial because it can extract clean water, especially from the anode solution, and increase the water production of desalination. The existence of ion exchange membranes can preclude microorganisms and other contaminants from entering the salt compartment; therefore, the additional water will not affect the quality of the desalinated water. This is important to downstream the RO process, since biofouling has become a serious problem for RO operation.[15]

Factors other than electricity generation and water osmosis may include dialysis, ion exchange, and other unknowns. One study reported significant TDS reduction due to dialysis in MDCs.[4] In the present work, we did not obtain direct evidence or data to show the presence of dialysis, but it likely happened. Our analysis was very preliminary and the inaccuracies that might occur during measurement could exaggerate or understate the effects of some factors. Clearly, additional work is needed to better understand the contributions to TDS reduction in MDCs.

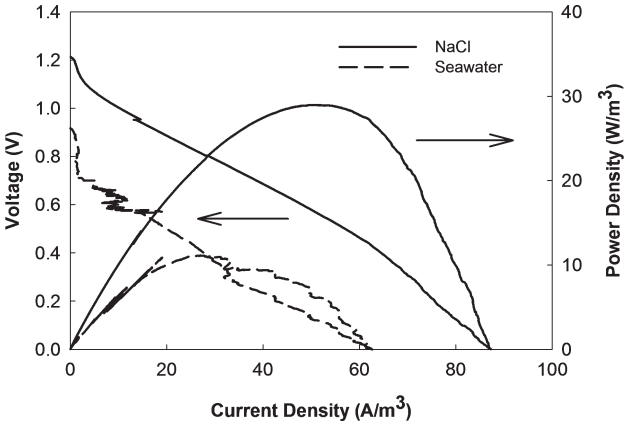

Figure 3. Polarization curves of the UMDC treating salt solution or artificial seawater. Scan rate was 0.1 mV/s. The HRT of salt solution or artificial seawater was two days.

High Power vs High Current. Our previous study raised the question of whether MDCs should be operated under high power output or high current generation.[5] MDCs will remove less TDS at high power output (near maximum power output) than at high current generation (near short circuit current), but the former condition can produce more electric power that will benefit downstream desalination when MDCs act as predesalination processes. In the present work, we quantitatively compared the energy production and desalination efficiency of the UMDC under those two conditions.

Polarization curves were used to determine the external resistance at which the maximum power output was achieved. The UMDC produced a maximum power density of 28.9 and 11.1 W/m^3 with salt solution and artificial seawater, respectively (Figure 3). The maximum power density with salt solution was close to that of our previous smaller-scale UMDC (30.8 W/m^3). According to the slope of the voltage drop, we estimated the internal resistance (ohmic resistance) of 6 Ω; therefore, the UMDC was operated at 6 Ω to reach a stable performance to collect data (Figure S2of the SI).

A significant discrepancy in power production was observed between potentiostat-measured polarization curves and actual operation. At 6 Ω, the UMDC produced a sustainable power that was 50−54% of the maximum power density obtained from the polarization curves (Table 1), although we employed a slow scan rate of 0.1 mV/s during the polarization test, which was expected to produce more accurate results. This difference required cautious reporting of the performance of bioelectrochemical systems using polarization curves to avoid false results (instant maximum power vs sustainable maximum power [16]). An interesting observation is that the open circuit potential (OCP) with salt solution reached 1.2 V, the highest OCP reported from the MFC-related studies. Nevertheless, we used the sustainable data obtained from the operation at 6 Ω to represent the condition of maximum power output, and the data obtained from the operation at 0.1 Ω to represent high current generation. The main results are summarized in Table 1 for both salt solution and artificial seawater.

It should be noted that analyzing systemic energy requirements and benefits is a complex effort and our estimate is preliminary. To facilitate the analysis, we made several assumptions. First, the UMDC acts as a predesalination system and its effluent

Table 1. Comparison of Performance of UMDC under Regular Operating Condition (0.1 Ω) and High Power Output (6 Ω) with Either Salt Solution or Artificial Seawater (HRT of 2 Days)

	external resistance of 0.1 Ω				external resistance of 6 Ω			
	k^a (mS/cm)	TDS^b (%)	I^c (mA)	P^d (W/m^3)	k^a (mS/cm)	TDS ^{b} (%)	I^c (mA)	P^d (W/m^3)
salt solution	21.9 ± 4.4	60.1 ± 6.5	143	1.1	31.7 ± 3.9	42.3 ± 7.0	70	15.6
artificial seawater	27.2 ± 0.6	42.5 ± 1.4	86	0.4	33.5 ± 0.5	29.1 ± 1.0	42	5.6

[a] Effluent conductivity. [b] TDS reduction. [c] Mean value of electric current. [d] Mean value of power density.

Table 2. Energy Estimate Based on One Day'S Operation (HRT of 2 days), Including Energy Production in the UMDC under Two Conditions and Energy Required by Downstream RO System

	water production (mL)	$E_{UMDC\text{-}0.1\Omega}$[a] (kWh)	$E_{UMDC\text{-}6\Omega}$[b] (kWh)	ΔE[c] (kWh)	$E_{RO\text{-}0.1\Omega}$[d] (kWh)	$E_{RO\text{-}6\Omega}$[e] (kWh)	ΔE_{RO}[f] (kWh)
salt solution	425	4.9×10^{-5}	7.1×10^{-4}	6.6×10^{-4}	9.8×10^{-4}	1.2×10^{-3}	2.5×10^{-4}
artificial seawater	425	1.8×10^{-5}	2.5×10^{-4}	2.4×10^{-4}	1.2×10^{-3}	1.5×10^{-3}	2.6×10^{-4}

[a] Energy production from the UMDC when desalinating 425 mL of saline waters at 0.1 Ω. [b] Energy production from the UMDC when desalinating 425 mL of saline waters at 6 Ω. [c] Difference in energy production between the UMDC at 0.1 and 6 Ω. [d] Energy required by ROs to treat 425 mL of saline effluent from the UMDC at 0.1 Ω. [e] Energy required by ROs to treat 425 mL of saline effluent from the UMDC at 6 Ω. [f] Difference in energy requirement by ROs when treating 425 mL of saline effluents from the UMDC between two conditions.

is further desalinated by an RO system. Second, the estimate is based on one day's operation and thus the water production of 425 mL (at saline water HRT of 2 d). Third, the specific energy of an RO system, when treating 3.5% saline water, is 3.7 kWh/m^3.[17] Last, to simplify the analysis, we disregarded the difference between salt solution and artificial seawater when estimating energy consumption by the RO system. In practice, energy consumption is affected by seawater quality.

The data indicated that, given high energy efficiency, it could be favorable if the UMDC operates under the condition of high power output when treating salt solution while high current generation would be desired with seawater desalination. With salt solution, the UMDC could bring the salinity down to ~22 mS/cm and ~32 mS/cm when operated at 0.1 Ω and 6 Ω, respectively (Table 1), resulting in an energy requirement of 9.8×10^{-4} kWh and 1.2×10^{-3} kWh by the downstream RO system for further desalination (Table 2); thus, 2.5×10^{-4} kWh is needed to reduce the gap of salinity between two operating conditions. Meanwhile, the UMDC produced 4.9×10^{-5} kWh and 7.1×10^{-4} kWh under two conditions. The difference of 6.6×10^{-4} kWh could be used to reduce the salinity gap and provide additional energy to the RO system; therefore, the high-power output condition was more favorable with salt solution, in terms of energy benefits. However, it should be noted that the analysis was based the assumption that 100% of the produced energy in the UMDC could be used by the downstream RO system; in reality, energy loss will occur during the transfer, storage, and use of electric energy. A similar analysis was applied to artificial seawater, but the results suggested no significant difference between two conditions. Considering the potential energy loss, high current generation is more advantageous because of lower salinity in the effluent. The total energy produced at the high-power condition, with 100% efficiency, could contribute 58.1% (salt solution) and 16.5% (artificial seawater) of the energy required by the downstream RO system, which is much higher than 5.0% and 1.4% when operated at the high-current condition. If the specific energy of the RO system can be further reduced, then the high-power operation of the UMDC will be more advantageous. The actual energy efficiency will greatly affect the results of the above analysis.

Perspectives of MDC Technology. The present work demonstrated that the UMDC could desalinate simple saline water, such as NaCl, and more complex water, such as artificial seawater, and provide energy benefits. The latter is especially interesting to the desalination industry, as high energy consumption is a key factor in the operating costs of desalination and high water prices. Using the UMDC developed in this work as an example, treating 1 m^3 of seawater and reducing more than 90% of TDS can produce an energy content of 1.8 kWh, while RO systems consume 2.2 kWh (energy for treatment). The net energy benefit by using the UMDC compared with RO systems is 4.0 kWh. In addition, the UMDC has more than 100% water recovery (due to water osmosis) while RO systems maintain about 50%, which means half of the water entering into ROs will be discharged as brine water. Higher water recovery will improve water use and minimize (waste) liquid discharge. In a scenario that employs the UMDC as predesalination before RO systems, if the UMDC reduces 30% of TDS in seawater, the energy required by RO systems can reduce from 3.7 to 3.5 kWh/m^3 due to lower salinity. The bioenergy produced in the UMDC can further reduce the energy requirement to 2.9 kWh/m^3, saving a total of nearly 22% in energy costs, compared with that without the UMDC. The detailed energy benefits require validation with pilot- or full-scale studies.

Despite the potential advantages, there is still a long way to go before MDCs can be applied in practice. Besides the high capital investment of bioelectrochemical systems, a major problem with MDCs is a low water production rate. To achieve similar water production, the UMDC requires a much longer retention time than RO systems, which may be compensated by a large reactor volume, but increases capital costs. Another issue is the discharge of the salt-enriched effluents: the common approach in the desalination industry is to return the brine water to the ocean, which could affect local aquatic ecology, but some studies found that the impact is not obvious because of the strong variability of the nature and rapid dilution.[18] When evaluating MDCs, one must also consider the benefits from wastewater treatment because MDCs are an integrated process of both wastewater treatment and saline water desalination. Such a system must be built in a location that can access both wastewater and saline

water. Because of the high possibility that the same water body will receive wastewater effluent and supply saline water for desalination, the effect of wastewater effluent discharge on saline water uptake should be minimized.

■ ASSOCIATED CONTENT

Supporting Information. The schematic of the UMDC is shown in Figure S1 and the current generation at 6 Ω is presented in Figure S2. This material is available free of charge via the Internet at http://pubs.acs.org.

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