

Efficient Desalination of Brackish Ground Water via a Novel Capacitive Deionization Cell Using Nanoporous Activated Carbon Cloth Electrodes

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Received 24 September 2014; Accepted 18 March 2015

Abstract: Sea water intrusion in ground water sources has made brackish water desalination a necessity in Oman. The application of capacitive deionization (CDI) for the deionization of ground water samples from wells in Al-Musanaah Wilayat is proposed and demonstrated. A CDI cell is fabricated using nanoporous activated carbon cloth (ACC) as the electrodes and is shown to be power efficient for desalting ground water samples with total dissolved solids (TDS) of up to 4,000 mg/l. The CDI cell was able to remove up to 73% of the ionic scaling and fouling contaminants from brackish water samples. The power consumption for deionization of brackish water was estimated to be 1 kWh/m³ of desalinated water, which is much lower than the power required to process water with equivalent TDS by the reverse osmosis processes. The CDI process is elaborated, and observations and analysis of the ion adsorption characteristics and electrical properties of the capacitive cell are elucidated.

Keywords: Capacitive deionization, Activated carbon cloth, Brackish water desalination.

التحلية الفعالة للمياه قليلة الملوحة بواسطة مكثف مستحدث لإزالة الأيونات بالسعة باستخدام أقطاب منسوجة من ألياف الكربون المحفزة

كارثك لكسمان، ميوتي زار مينت، محمد العبري، ليلي الغربية، بثينة النعمانية، حاج بوردوسن، وجويدب دوته

المستخلص: إن تداخل مياه البحر مع مصادر المياه الجوفية قد جعلت تحلية المياه قليلة الملوحة ضرورة ملحة في عمان. نقترح ونظهر تطبيق مكثف لإزالة الأيونات بالسعة وذلك لإزالة الأيونات من عينات المياه الجوفية المستخرجة من الآبار في ولاية المصنعة. إن مكثف إزالة الأيونات بالسعة يصنع من أقطاب منسوجة من ألياف الكربون المحفزة مما يجعلها فعالة في تحلية عينات المياه الجوفية وذلك عن طريق إزالة المواد الصلبة حتى تركيز 4000 ملي جم/لتر. لقد تمكن مكثف إزالة الأيونات بالسعة من إزالة ما يصل إلى 73% من الترسب الأيوني للملوثات من عينات المياه قليلة الملوحة. ويقدر استهلاك الطاقة لإزالة الأيونات من المياه قليلة الملوحة إلى 1 كيلو واط ساعة / متر مكعب من المياه المحلاة، وهي أقل بكثير من الطاقة المطلوبة لمعالجة نفس الشوائب بواسطة عمليات التناضح العكسي. نحن ندرس العملية في مكثف إزالة الأيونات بمزيد من التفاصيل، ونراقب ونحلل خصائص أيون الامتزاز والخواص الكهربائية للخلية بالسعة.

الكلمات المفتاحية: مكثف إزالة الأيونات بالسعة، ألياف الكربون المحفزة، تحلية المياه قليلة الملوحة.

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1. Introduction

Seawater intrusion into ground water sources is a major problem in coastal regions worldwide. Groundwater is the primary source of fresh water for more than two-thirds of the world's population living in these regions. Particularly in the Sultanate of Oman, which has more than 2,000 kilometers of coastline, rapid population growth and increased agricultural activities have severely stressed groundwater sources, which constitutes about 65% of available fresh water in the Sultanate. Increased demand for groundwater (1,650 Mm³/annum) compounded by falling water tables has led to rapid sea water intrusion, costing the Sultanate an estimated US\$19–36 million annually (2012; 2006). To supplement the groundwater supply, the government has prioritized water desalination, enabling it to grow from 3% of the total water supply in 1999 to 13% in 2008 (Gastli *et al.* 2010). Although reverse osmosis (RO), with its modular technology, is at the forefront of this industry, the membranes used in RO are generally prone to fouling by dissolved macromolecular organic substances, soluble inorganic compounds, and suspended particles and microorganisms, leading to reduction in water recovery and membrane life (Van Der Bruggen *et al.* 2002; Vrouwenvelder *et al.* 2003).

Generally, extensive pretreatment like coagulation, flocculation, pH adjustment, and the addition of antiscalants, disinfectants, and media filtration are required to increase water recovery, which further increases operational costs of RO plants (Ghabayen *et al.* 2004; Hassan *et al.* 1998). Typically, RO plants desalinating brackish water can remove 98% of the salt at total dissolved solid (TDS) levels of 2,500–3000 mg/L, using pressures of 13.6–17 atmospheric pressure (atm) and a flux of 24 L/m²/day. Recent technological advancements like high efficiency pumps and energy recovery systems have reduced the expected energy requirements of brackish water RO to ~2–3 kWh/m³ (Zhao *et al.* 2013). However, the RO process is restricted to certain operating conditions, including maximum temperatures of 45°C, pH values of 2–10, a silt density index (SDI)<3, and mandatory dechlorination, amongst others. This makes changes due to variations in water quality uneconomical in operational RO plants (Nicolaisen, 2002). Additionally, water recovery in RO is governed by the scale formation potential of brackish water, which is rich in scaling elements including calcium carbonate, calcium sulfate, magnesium sulfate,

strontium sulfate, silicates, and aluminosilicates (Amiri and Samiei 2007; Antony *et al.* 2011; Farhat *et al.* 2012; Ghafour 2003; Tzotzi *et al.* 2007). Scaling reduces productivity and the water recovery ratio, which are necessary to maximize water production due to restrictions on brine reinjection in Oman (Ahmed *et al.* 2001; Mohamed *et al.* 2005).

An alternative and emerging desalting technique which can circumvent these issues is called capacitive deionization (CDI), wherein emphasis is laid on removing salt from the water rather than separating the water from the salt as is done in RO (Porada *et al.* 2013; Zhao *et al.* 2010). Since the volume fraction of salt is considerably lower than the volume of water in ground or sea water, the process has shown promise as a good alternative to RO, especially for brackish water deionization (Lee *et al.* 2006; Mossad *et al.* 2013; Mossad and Zou 2013; Zhang *et al.* 2013). Salt removal by CDI involves the electrosorption (potential induced ion adsorption) of ions on electrodes and can be reasonably fitted with the Langmuir adsorption isotherm (Mossad *et al.* 2013; Ryoo *et al.* 2003; Welgemoed and Schutte, 2005) and modified Donnan model, where equilibrium achieved between the adsorption and desorption of ions defines the salt adsorption capacity of the electrodes. Since ion adsorption takes place on the surface of the electrodes, desalting efficiency is directly proportional to the surface area of the electrodes (Zhao *et al.* 2010). The magnitude of the applied potential for CDI is generally kept below 2.0 V DC to avoid hydrolysis of the aqueous media (Biesheuvel *et al.* 2009; Oren 2008).

In a CDI cell, one electrode is connected to the positive potential terminal and the other to the negative terminal of the DC power supply. When salt water is passed through the cell, anions (negative ions) will be electrosorbed to the positive terminal while cations (positive ions) will be electrosorbed to the negative terminal, thus depleting the water of ions and leading to desalted water at the output. Since the adsorption is a surface related phenomenon, continuous deionization leads to saturation of the electrodes and subsequent inability to adsorb any further. To circumvent this problem, every deionization cycle is followed by a regeneration cycle, during which the applied potential is removed, leading to a release of the electrosorbed ions from the activated carbon cloth (ACC) surface into the flowing solution, leading to a highly concentrated solution, known as brine, at the output. A key advantage of electrosorption is that the ions are

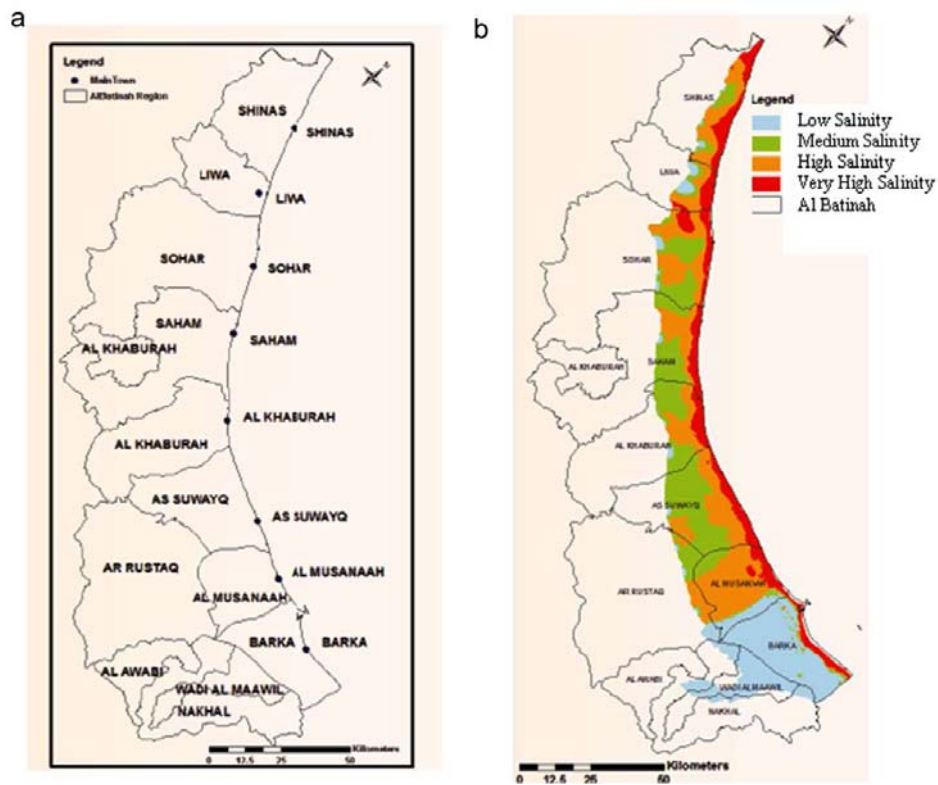


Figure 1. (a) Location of the Al Batinah Region, which accounts for more than 50% of Oman’s agriculture production and (b) Salinity increase due to sea water intrusion in the Al Batinah region over the years (Ministry of Agriculture and Fisheries, 2012).

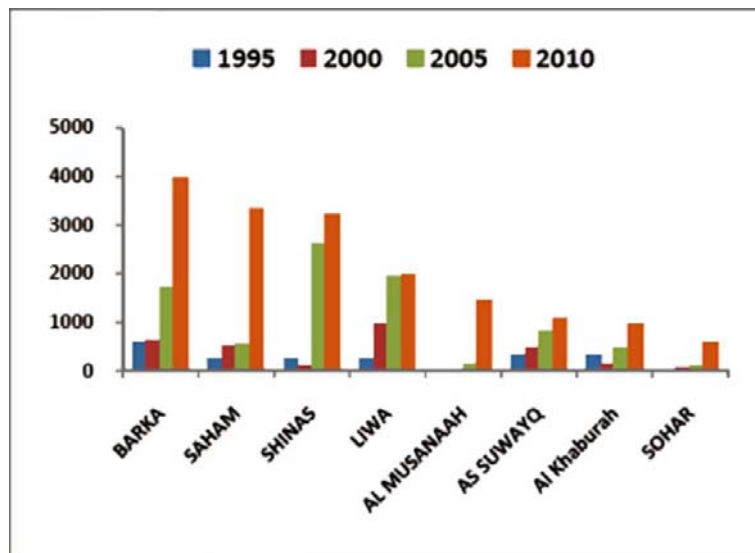


Figure 2. Representation of total land area affected by sea water intrusion showing that Al-Musanaah was heavily intruded (Ministry of Agriculture and Fisheries, 2012).

physisorbed onto the electrode surface, minimizing electrode fouling and leading to longer operational life of the electrodes. Further, the absence of ion exchange membranes and the chemically inert nature of ACC enables the facile removal of any organic or microbial contamination without adversely affecting the electrode integrity by washing in an acid bath (Laxman *et al.* 2015; Mossad *et al.* 2013).

This manuscript demonstrates the use of CDI incorporated with ACC electrodes for efficient removal of salt from brackish water collected from wells in Al-Musanaah Wilayat in Oman. Emphasis is laid on the effect of an ionic charge on desalting capacity along with power consumed for the deionization of a well water sample to drinking water standards.

2. Methods

Analytical grade hydrochloric acid 34% (HCl) (Merck KGaG, Darmstadt, Germany) was used without any further purification. FM-100 ACC Zorflex (Calgon Carbon Corporation, Pittsburgh, Pennsylvania, USA) with a mean pore diameter of 0.7 nm and a total pore volume of 1.417 cm³/g was used. Micropores accounted for 0.56 cm³/g and macropores for 0.85 cm³/g, while mesopores were very few (<0.001 cm³/g), making the material predominantly microporous. The ACC also possesses a ribbed surface with a hierarchical pore structure which reduced the pressure drop across the electrode and is preferable for flow systems like CDI. Nuclear magnetic resonance measurements estimated the surface area of pristine ACC to be ~1000 m²/g. In addition to the high surface area, previous reports have suggested that the sheet resistance of symmetrically cut ACC varies between 4–5 Ω/sq cm, all of which make it suitable for application in CDI (Subrenat *et al.* 2001). The electrode substrates (Brasquet and Le Cloirec 1999; Shim *et al.* 2001) were cleaned with highly concentrated 2 M HCl and heated to 115 °C, which is slightly above the boiling point of HCl solution, for 12 hours to remove mineral contaminants. Subsequently, samples were thoroughly rinsed with deionized water, dried in a vacuum oven at 150°C for 12 hours, and stored in a desiccator until needed for further use.

The CDI device (cell) used for the deionization consisted of two 10 x 10 cm ACC electrodes separated by a spacer having a thickness of 650 μm. It was comprised of a reservoir made up of

polymethyl methacrylate (PMMA) from the SYLGARD® 184 silicone elastomer kit (Dow Corning, Midland, Michigan, USA), a current collector (graphite electrode), and an acrylic plate to support the CDI cell [Figs. 3a–b].

For brackish ground water deionization, well water from two different wells located in Al Musanaah of the Al Batinah Region in Oman's northwest was chosen. The Al Batinah region as a whole accounts for 50% of the country's agricultural production, and 95% of the abstracted water in the governorate is used for agriculture and livestock. However, this area has experienced a steep increase in groundwater salinity due to a high abstraction rate and seawater intrusion [Figs. 1a and b; Fig. 2]. Of Al Musanaah's farms, 40% are irrigated with 2–5 mS/cm brackish water, while only 35% are irrigated with <2 mS/cm of ground water (2012).

The surface morphology of ACC electrodes was studied with a JSM-7200 field emission scanning electron microscope (FESEM) working at 20 kV (JEOL USA, Inc., Peabody, Massachusetts, USA). Water conductivity was determined using EUTECH CON 700 conductivity probe with a cell volume of 5 ml (Thermo Fisher Scientific, Inc., Waltham, Massachusetts, USA) which was modified for online real time recording. Active surface area measurements were conducted using a nuclear magnetic resonance (NMR) relaxation technique in XigoNanotools equipment (Bethlehem, Pennsylvania, USA) with water as the solvent. The changes in the T2 relaxation time after perturbation was measured and analyzed by Acorn Area software (XigoNanotools) to interpret the surface area of the electrode using the specific surface relaxivity of activated carbon in contact with the solvent. Cations were determined by inductively coupled plasma optical emission spectrometer (ICP-OES) 710 (Varian, Medical Systems, Palo Alto, California, USA). Power consumption was calculated by integrating the charging current decay curve in Matlab (Mathworks, Natick, Massachusetts, USA) to obtain the total charge deposited in one cycle. The charge deposited was multiplied by the applied voltage to obtain the work done in joules, which was subsequently converted to watts by dividing it by the duration of the deionization cycle. Rates of ion adsorption were obtained by fitting the ion adsorption rates (with respect to time and number of deionization cycles) as obtained by ICP with a cubic polynomial function with an R² value greater than 99%, followed by taking the first derivative of the cubic fitting function.

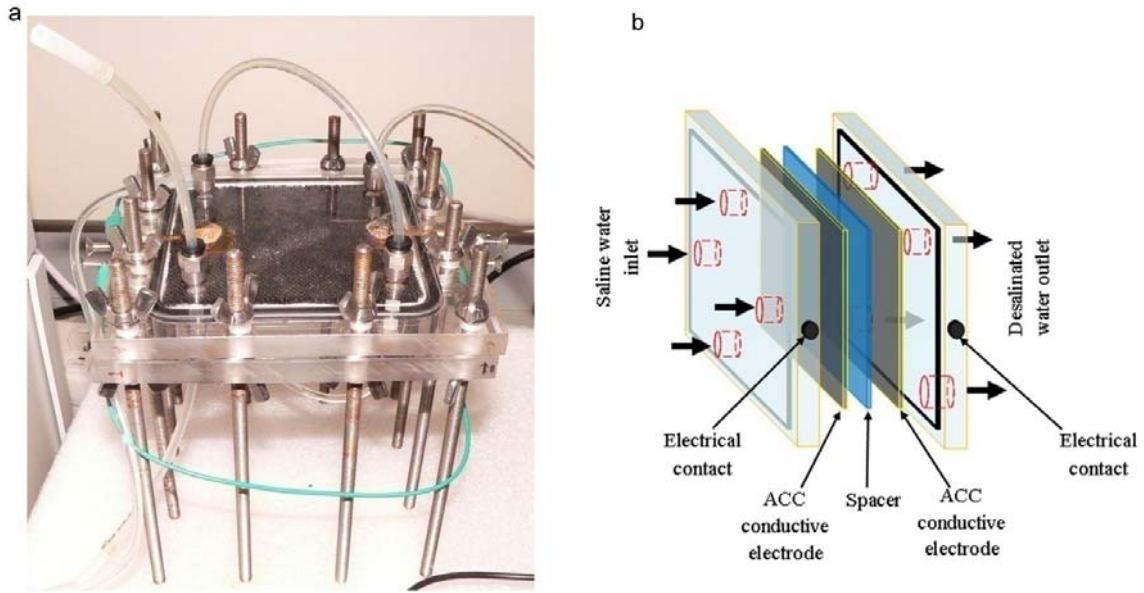


Figure 3(a). Prototype of the CDI cell comprising two 100 cm² ACC electrodes separated by a spacer and (b) building blocks of the CDI cell showing the placement of the electrodes, spacer and electrical contacts.

3. Capacitive Deionization Experiments

The deionization/regeneration experiments were conducted using flow through capacitive deionization cell. Capacitive deionization experiments were performed in a batch mode, where 50 ml of feed solution was passed through the cell at a flow rate of 5 ml/minute in a continuous loop for 15 minutes. A peristaltic pump drive 5201 (Heidolph Instruments, Schwabach, Germany) was used to maintain a constant flow rate of the feed water into the CDI cell. During this period, a DC potential of 1.6 V DC was applied across the CDI electrodes, leading to electrosorption of the ions on the ACC electrodes [Fig. 4a]. Water samples were collected every five minutes during the deionization period to measure the ion content using ICP-OES. The change in solution conductivity, current and voltage across the cell was monitored using GW Instek GDM-396 online multimeters (Good Will Instrument Co., Taipei, Taiwan) for power consumption analysis. After 15 minutes of deionization, regeneration was carried out by removing the applied potential across the electrodes and letting the previously desalinated water flow through the system at five ml/minute.

During this period, ions which were previously electrosorbed onto the electrode surface were released and removed from the cell [Fig 4b]. The

regeneration cycle was continued until original feed water feed water conductivity was recovered, confirming complete regeneration of the electrodes.

In a second experiment, the above deionization period of 15 minutes was followed by a regeneration period of 20 minutes to complete one deionization-regeneration cycle. A total of seven such cycles were conducted on water from well two to attain water fit for drinking. Water samples were collected after every cycle to measure the ion content and adsorption characteristics at different cycles.

4. Results and Discussion

A FESEM image shows the interwoven fiber structure of the conductivity of the ACC electrodes [Fig. 5] used for the deionization process. Each fiber is composed of macro, meso, and micropores, which lead to a highly active surface area available for ion adsorption (García-Quismondo *et al.* 2013; Huang *et al.* 2013). The deionization performance of the CDI cell was studied with ground water samples from two different wells in Al-Musanaah. The analyzed composition and properties of the as received well water samples are given in Table 1.

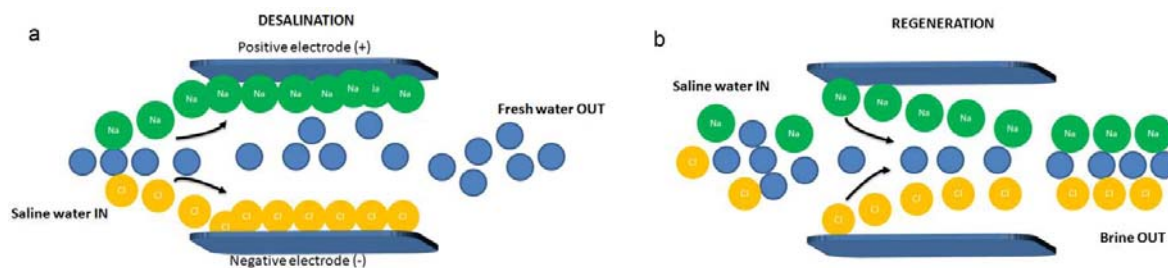


Figure 4. Schematic showing the functioning of a capacitive desalination cell where (a) the desalination step involves the application of a 1.6 V DC potential across the electrodes and the subsequent electro-sorption of ions from the inlet feed water on to the ACC electrode surface and (b) is followed by the regeneration step wherein the surface adsorbed ions are released to the inlet water feed by removing the applied potential to regenerate the electrode surface prior to the next desalination step.

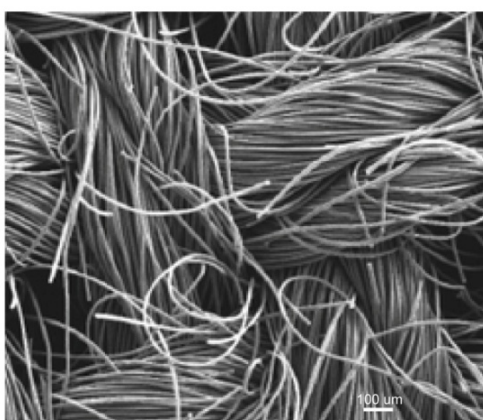


Figure 5. Field emission scanning electron microscopy image of activated carbon cloth (ACC) showing the inter-woven fibers.

Table 1. Measurements of properties of ground water samples from two wells in Al-Musanaah.

Sample	Conductivity (mS/cm)	pH	Sodium (Na) (ppm)	Calcium (Ca) (ppm)	Potassium (K) (ppm)	Chloride (Cl) (ppm)
Well 1	5.5	8.2	293	330	10	784
Well 2	3.1	8.0	252	244	8	507

Table 2. Cation removal efficiency in the CDI cell for water from well one after a 15-minute deionization cycle as determined from inductively coupled plasma measurements. The conductivity of the water reduced from 5.5 mS/cm to 3.6 mS/cm.

Ion type	Initial concentration (ppm)	Final concentration (ppm)	% Removal
Sodium (Na ⁺)	293	190	35
Calcium (Ca ²⁺)	330	184	44
Potassium (K ⁺)	10	7.7	23

Table 3. Cation removal efficiency in the CDI cell for water from well two after 7 deionization cycles as determined from inductively coupled plasma measurements. The conductivity of the water was reduced from 3.2 mS/cm to 0.84 mS/cm.

Ion type	Initial concentration (ppm)	Final concentration (ppm)	% Removal
Sodium (Na ⁺)	252	39	85
Calcium (Ca ²⁺)	242	35	86
Potassium (K ⁺)	8	3	63

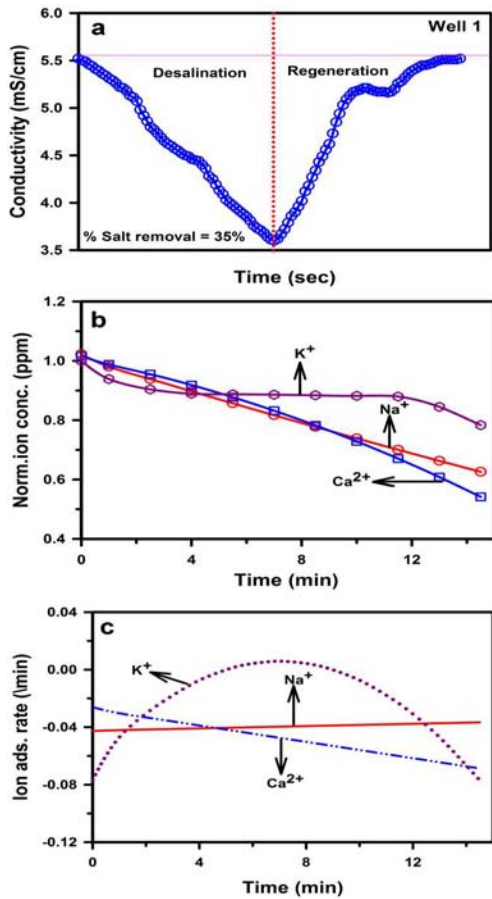


Figure 6. (a) Desalination and regeneration performance of a batch mode process for water from well 1, showing 35% overall salt removal efficiency. The desalination was carried at a flow rate of 5 ml/min for 15 minutes at an applied potential of 1.6 V DC; (b) normalized ion adsorption curve for sodium (Na⁺), potassium (K⁺) and calcium (Ca²⁺) with time, suggesting that there was no surface saturation of the electrode during the period of desalination and (c) ion adsorption process was relatively constant for high concentration ions like sodium and calcium.

Water from well one has a TDS of ~4 g/L with an initial conductivity of 5.5 mS/cm. From ICP measurements, the calcium content in well one (330 ppm) was found to be more than that of sodium (293 ppm), while the potassium content was low (10 ppm). On being passed through the CDI cell continuously for 15 minutes (without any regeneration), the solution conductivity was reduced to ~3.6 mS/cm, effectively a 35% reduction in TDS of the water with an electrode-specific salt adsorption capacity of ~8 mg/g [Fig. 6a]. ICP analysis of the desalinated water showed that Na⁺ was reduced by 35%, Ca²⁺ by 44%, and K⁺ by 23% following the well-established adsorption trend of higher selectivity for divalent and higher concentration ions over monovalent and low concentration ions (Hou and Huang 2013) [Fig. 6b and Table 2]. Na⁺ ions had preferential selectivity for adsorption over K⁺ ions although they had the same ionic charge. In cases where the magnitude of charge is the same, ions with a higher concentration, like Na⁺ in this case, will be selectively adsorbed due to a stronger driving force mediated by the concentration gradients, resulting in easier ion transport through the ACC pores (Hou and Huang 2013).

Figure 6b shows the comparison of normalized ion adsorption curves for the three ions, which confirms that Ca²⁺ has a higher adsorption capacity as compared to the other two ions, and that the electrode surface is not saturated after the 15-minute deionization period. This was further confirmed as the first order differential plots of the ion adsorption curves showed that the slope of the tangent lines for Na⁺ and Ca²⁺ were either constant (Na⁺) or still increasing (Ca²⁺), which would not be the case if the electrode was saturated [Fig. 6c]. The absence of electrode saturation is an important aspect of the CDI operation to maximize the charge efficiency, which is expressed as a ratio of electrical charge input to ionic charge adsorbed. The charge efficiency was found to be ~68%, expressing the

power efficiency of the deionization process (Huang *et al.* 2012; Zhao *et al.* 2010). Based on these results, the deionization period of further experiments was fixed at 15 minutes.

To estimate the total power and time required to bring a saline ground water sample to drinking water standards, water from well two was passed through the CDI cell in multiple deionization-regeneration cycles. The deionization was carried out for seven cycles in a repeated batch mode for 200 ml of water from well two, where each cycle comprises a deionization period of 15 minutes followed by a regeneration period of 20 minutes. The solution conductivity was reduced from 3.2 mS/cm to 0.84 mS/cm, effectively removing 73% of the ion content from the brackish water from well two, with an average electrode salt adsorption capacity of 6.2 mg/g [Fig. 7a]. The rate of ion adsorption was determined by taking the first order differential of the normalized ion adsorption curve, plotted with respect to deionization cycles. As the adsorption function continuously reduces with time, its derivative will have a perpetual negative slope, where a high negative value indicates a high adsorption rate. Similarly, a positive slope (rising) indicates a reducing adsorption rate and a negative slope (falling) an increasing rate. As observed, the adsorption rate curves suggest that for both singly and doubly charged ions, the adsorption rate initially decreases with time to reach a steady state where the rate shows a value close to zero and then increases again when the solution ion concentration reduces further [Fig. 7b].

This trend suggests that adsorption rates and solution ion concentrations are not constant and change with each cycle. In essence, the water was successively depleted of the salt ions in each cycle, with a total Na⁺ removal of 85%, Ca²⁺ removal of 86%, and K⁺ removal of 63% [Table 3]. As the conductivity of the water sample decreased with an increasing number of deionization cycles, the power consumption also decreased [Fig. 8], showing that the power was directly related to the ionic strength of the solution being desalinated.

Thus, water with a high ion concentration will require higher power, limiting the application of CDI for low to moderate saline ground water deionization (Cohen *et al.* 2013; Zhao *et al.* 2013). Extrapolating the power consumption data from the desalinated one m³ of well two water to drinking water quality (from 3.2 mS/cm to 0.84 mS/cm), 1 kWh of power would be required, excluding power consumed for pumping. This in effect would be

lower than the power required for RO of a similar water sample. The absence of a membrane in the design, small footprint with low manufacturing costs, and chemically robust nature of the ACC electrode will further reduce the recurring costs of the system. Additionally, the low voltage and current requirements of the CDI cell as compared to RO make it more effective as a portable deionization unit for use in remote locations (Zhang *et al.* 2013).

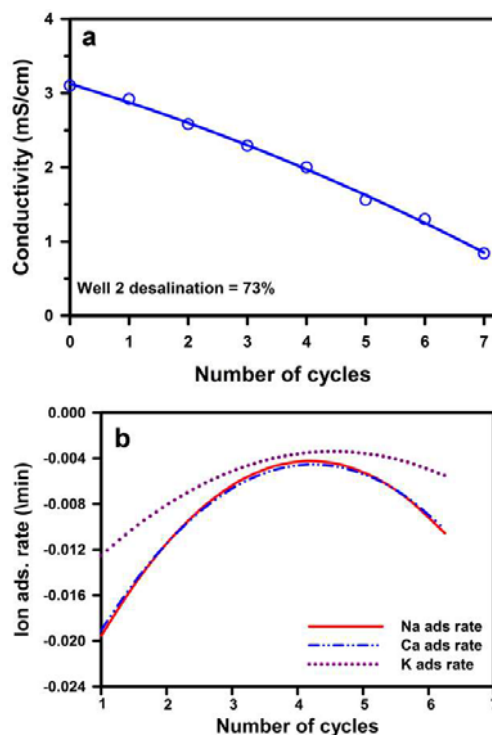


Figure 7. (a) Desalination performance of a multiple batch mode process for water from well 2, leading to 73% overall salt removal efficiency. The desalination was carried at a flow rate of 5 ml/min for 7 desalination (followed by regeneration) cycles of 15 minutes each at an applied potential of 1.6 V DC; (b) the ion adsorption rates for sodium, potassium and calcium increases and subsequently reduces with time, suggesting that ion removal rate varies linearly with the ion concentration in solution, with lower ion concentration leading to lower removal rates.

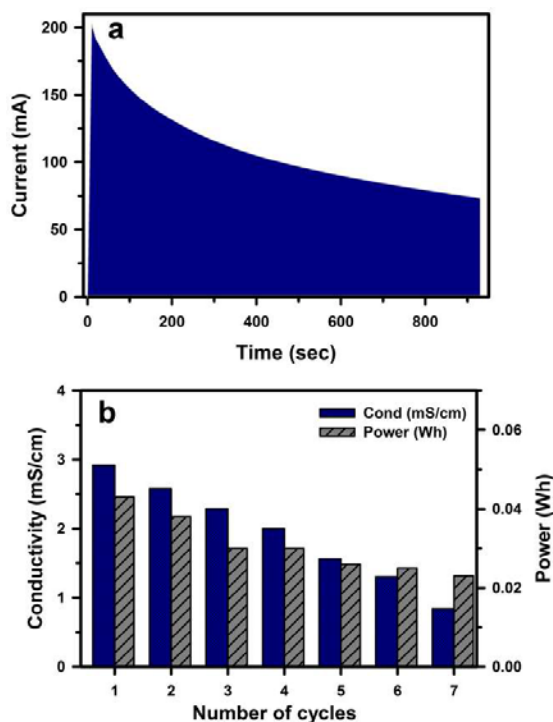


Figure 8. (a) Integration of the area under the desalination current curve for 1 cycle to calculate the total charge delivered to the CDI cell during the desalination process. This charge multiplied by the applied potential (1.6 V DC) and divided by the time duration of the desalination cycle (15 minutes) to give the power (watt) consumption which was converted to watt-hour format; (b) graph showing the change in conductivity per desalination cycle and its associated power consumption for the desalination cycle in watt-hours, which suggests that power consumption reduces with conductivity (ion concentration).

5. Conclusions

The viability of the CDI device in desalinating ground water samples from the Al-Musanaah Region of Oman has been shown. The ion content and conductivity of 200 ml of brackish water (well two) was reduced from its initial value of 3.2 mS/cm to 0.84 mS/cm, making it drinking water quality, with just 0.2×10^{-3} kWh of power consumption. It is estimated that one m^3 of water from well two could be desalinated to drinking water standards using one kWh of power, which is lower than that required by RO. Additionally, scaling and fouling ions were shown to be efficiently removed during the process, making the CDI a viable option for brackish water deionization.

Acknowledgments

The authors would like to thank the Chair in Nanotechnology, The Research Council of Oman (TRC), and Sultan Qaboos University for financial support.

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