



Capacitive deionization of ground water using carbon aerogel based electrodes

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ABSTRACT

Energy-efficient desalination technologies are needed to address the growing requirements of potable water owing to population increase, industrial development, and increasing energy cost. Capacitive deionization (CDI) technique is receiving attention because it offers an energy-efficient method of removing dissolved salts by electro-adsorption. The technique has been explored for ground water with total dissolved solids (TDS) of 500–1,500 mg/l, typical of rural areas in India. We have therefore undertaken development of a test cell and report its use for electro-sorption of dissolved ions in groundwater. The active material used is mesoporous carbon aerogel having surface area $\sim 2,000 \text{ m}^2/\text{g}$, developed in house. The carbon aerogel-based electrodes have shown high salt adsorption capacity of 8.4 mg/g with NaCl electrolyte. CDI cell modules of 500 ml capacity with eight-electrode stack have been used in single and multiple stages, depending on the ground water TDS. Water having salt concentration of 1,500 mg/l was treated to achieve output of $\sim 238 \text{ mg/l}$ with power consumption $\sim 0.5 \text{ kWh/m}^3$. The low power requirement, chemical free regeneration, and applicability for the ground water make this system of potential interest for addressing the issue of potable water in rural and remote areas.

Keywords: Carbon aerogel; Desalination; Capacitive deionization; Specific capacitance; Adsorption capacity

1. Introduction

Scarcity of potable water is a serious global problem and is getting aggravated due to depletion and contamination of surface water sources. The situation is getting worse in remote and rural areas where people are dependent mainly on ground water sources. However, there is growing concern on the ground water quality due to the presence of dissolved

inorganic impurities, resulting from chemical interactions of geological substances with water. For potable applications, the salinity as well as concentration of contaminants such as chlorides, fluorides, and nitrates need to be within permissible limits. The salt concentration in water is represented by total dissolved solids (TDS) count and it is recommended that TDS level be less than 600 mg/l [1] because higher level of TDS may affect the palatability and also lead to

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excessive scaling in water pipes, heaters, boilers, and household appliances.

In recent decades, the most widely used processes in water desalination plants include reverse osmosis, electrodialysis, and thermal separations such as multi-stage flash distillation and multi-effect distillation. These technologies are energy intensive and therefore expensive option with typical energy requirement of $\sim 4 \text{ kW/m}^3$ [2]. This concern is important for the remote and rural areas particularly in developing countries where energy resources are limited. Considering large population associated, an energy-efficient technique is needed which can suffice the desalination requirements of these areas. These regions particularly require sturdy deionization technique which doesn't require frequent replacements of cartridges and membranes, and at the same time has low-energy expenditure [3]. In this regard, capacitive deionization (CDI) is one of the cost-effective and promising method with lots of potential [3]. CDI technique is based on electro-sorptive principle of adsorbing ions on high surface area electrodes in a capacitive action such that the outgoing stream becomes devoid of the ions present in the incoming stream. The ions electro statically separated from aqueous solutions form the electrical double-layer within the electrode/electrolyte interface. This being a electro-sorption-based technique, offers advantages of low-voltage requirement, ambient operational conditions and easy regeneration over the common desalination technologies [4,5]. In addition, CDI becomes an attractive option at moderate TDS levels, as it is more advantageous to remove relatively few salt molecules than to remove the water molecules [6].

For CDI, the key active component is the electrode material, which governs the salt adsorption and removal capacity based on surface area, pore structures, pore volume, and electrical conductivity. A variety of high surface area carbon nanomaterials have been reported in the literature for removal of ions or charged contaminants, as means of desalinating brackish water, softening hardness, or remediating ground water [7–12]. Carbon aerogels (CA) have been considered to be a good choice for CDI application as they provide inter connected carbon particles, good electrical conductivity and open pore structure that can be tailored by optimizing synthesis parameters [4,13]. Activated carbon (AC) is also a widely studied material. It offers the advantage of low cost, easy availability, and high surface area, but suffers from the drawback of having dominance of micropores [5,14]. Carbon nanotubes (CNT) and carbon nanofibers possess good stability, good electrical conductivity, but are expensive [15,16]. Mesoporous carbon material on the other hand offer suitable pore structure, but

complicated preparation methods [17,18]. Graphene, offers good surface area, electrical conductivity but its cost and pore structures are the main concerns [19]. Studies have also been carried out on composites of carbon materials of different characteristics as well as carbon and metal oxides or polymers to take advantage of the superior physical and chemical properties of constituent materials [6]. Some of the carbon composites studied include AC/CA [20,21], AC/graphene [22], CNT/nanofiber [23,24], and CNT/graphene [25]. Metal oxide-based AC carbon composites like TiO_2/AC , ZnO/AC have been reported to have enhanced performance due to their high charge storage densities [5,26,27]. Studies on optimization of the properties of a single component material by suitable chemical and physical treatments are also being actively pursued [28–30]. For example, CO_2 activation of CA can be used to enhance its surface area as well as mesoporous surface area [31]. In this paper, we report desalination studies performed using electrodes made of CO_2 -activated mesoporous carbon aerogel with high surface area ($\sim 2,000 \text{ m}^2/\text{g}$) developed in-house. The electrodes were characterized and their specific capacitance and salt adsorption capacity were evaluated. CDI cell modules consisting of hydraulically parallel electrode stacks have been used for the desalination experiments in single and multiple stages. The results of these experiments have been analyzed for evaluating effectiveness of this technique for ground water desalination.

2. Methodology

2.1. Synthesis of carbon aerogel

Carbon aerogel was synthesized by polycondensation of resorcinol (R) and formaldehyde (F) in an aqueous medium, similar to the method reported by Lee et al. [32]. RF solution was prepared in water with molar ratio of R/F fixed at 0.5 with a base catalyzing agent (C) maintaining R/C molar ratio as 450. The solution was gelled in convection oven at 80°C and exchanged with acetone after complete gelation. The dried gel was pyrolysed at 800°C , in inert atmosphere with argon gas flow of 2–3 lpm. This was followed by activation at 900°C for a period of 3–5 h under controlled atmosphere of CO_2 (flow rate ~ 4 lpm) to achieve high surface area carbon aerogel [33].

2.2. Electrode preparation and stack assembly for the modules

The working electrodes for the experimental cells were prepared using CO_2 -activated carbon aerogel

(a-CA) powder. Slurry was prepared using a-CA with 10-wt.% water solution of polyvinyl alcohol (PVA) and glutaric acid in 1:3 ratio [34]. The slurry was spread uniformly using a film casting knife on graphite sheet of the size 20×20 cm to achieve total active material loading of ~ 6 g for each electrode. Twenty-four such graphite sheets were coated and subsequently cured at $\sim 250^\circ\text{C}$ in inert atmosphere. Two-sided electrode panels were prepared by pasting two coated electrodes, back to back on a PVC sheet. Both the electrodes were electrically shorted to form a double-sided electrode. Four such double-sided plate electrodes were prepared and mounted with 2 mm separation between them to form eight-electrode stack Fig. 1(a). Alternate plate electrodes of the stack were used as anode/cathode. An acrylic water-tight cavity was used as housing with the stack mounted in a way to allow parallel flow of the effluent for increased throughput as shown in Fig. 1(b). This cell of 500 ml capacity, with eight-electrode stack forms a CDI cell module. Three such CDI cell modules have been prepared with an overall a-CA loading of $\sim 45 \pm 1$ g per module.

2.3. Characterization for surface area, specific capacitance and salt adsorption capacity

2.3.1. Surface area characterization

Surface area and pore size distribution of a-CA powder was characterized by analysis of nitrogen adsorption–desorption isotherms measured by ASAP-2020 analyzer (Micromeritics). BET method was used for total surface area measurements and t -plot was used for estimating mesopore surface area. Pore size distribution and average pore diameter were obtained by the BJH method from desorption branch of the

isotherms. Total pore volume was calculated from the adsorbed volume of nitrogen at $P/P_0 = 0.99$ (saturation pressure).

2.3.2. Specific capacitance

Cyclic voltammetry (CV) was performed on a sample electrode to preliminarily investigate the specific capacitance of the active electrode material. Measurements were performed with potentiostat for electrochemical analysis (Autolab, PGSTAT302N) in a conventional three electrode cell. Working electrode with loading of 15 mg active material was formed by coating a-CA on 1 cm diameter graphite sheet using similar procedure described in Section 2.2. Platinum electrode was used as counter electrode and Ag/AgCl electrode was used as a reference electrode. Measurements were conducted in the potential range of -0.5 to $+0.5$ V vs. reference electrode for 1–5 mV/s scan rate (S) in 0.5 M NaCl electrolyte [35]. The specific capacitance expressed in farads per gram, was evaluated using Eq. (1):

$$C = I / (S \cdot m) \quad (1)$$

where I (mA) is the current, S (mV/s) is applied scan rate, and m is the mass of active material in grams.

2.3.3. Adsorption capacity of electrode material

One of the important characteristics for evaluating CDI performance is the “salt adsorption capacity” of the electrode material defined as the amount of salt adsorbed per gram of the active electrode material. The salt adsorption capacity of the electrode material was evaluated using a batch mode experiment [36].

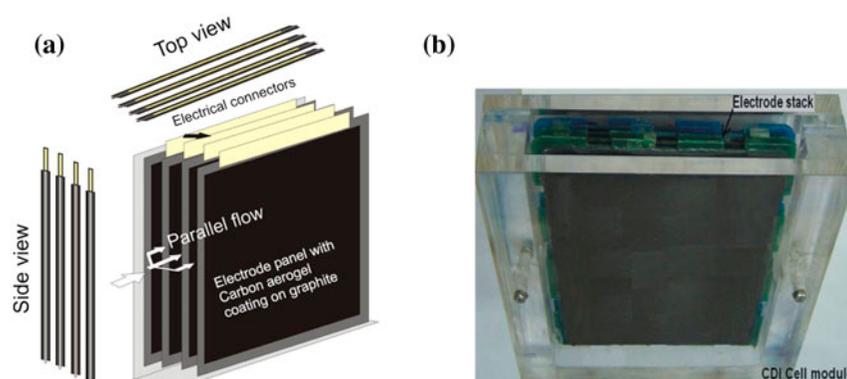


Fig. 1. (a) Schematic of the electrode stack mounted in parallel flow geometry and (b) assembled CDI cell module with multi-electrode stack.

The setup (shown in Fig. 2) consists of 500 ml capacity CDI cell module, a peristaltic pump and feed reservoir. The concentration of the feed reservoir was monitored by using pre-calibrated conductivity meter (WTW, Cond 330i) for obtaining corresponding TDS expressed in mg/l. As per the procedure, prior to the experiment, the electrodes of the stack were discharged for nearly one hour in low conductivity water. The experiment was performed with 2.0 l feed solution having initial TDS of 500 mg/l, circulated through the stack of module at a flow rate of 0.5 lpm. At applied potential of 1.2 V, the TDS of solution was monitored which nearly stabilized after ~20 min and final TDS was recorded. The saturation time of 20 min was hence taken as duration for the deionization and regeneration cycles during operation of modules in further experiments. During the desalination cycle, the electrodes of the cell were electrically shorted. The specific salt adsorption capacity (Q) was calculated according to Eq. (2):

$$Q = (C_i - C_f)V/m \quad (2)$$

C_i and C_f represent the initial and final TDS in mg/l, V is the volume of the feed solution in liters and m is the mass of active material used in the stack in grams [36].

2.4. Desalination experiments with CDI modules

Desalination experiments were conducted using electrolyte solutions with different TDS values and parameters such as salt removal, water recovery and energy/power usage were evaluated. Water recovery takes into account the quantity of output generated to the input feed. Schematic of the experimental setup with single CDI cell module, is shown in Fig. 3. The module has been mounted vertically, which allowed the effluent flow under gravity without requiring

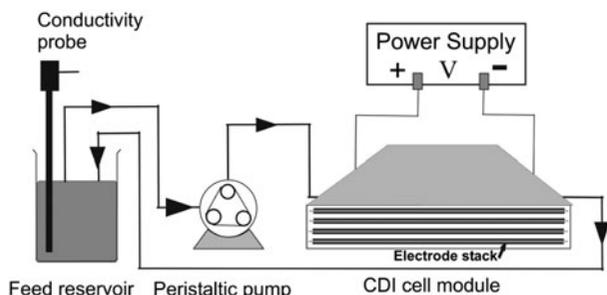


Fig. 2. Setup for evaluation of adsorption capacity of electrode material.

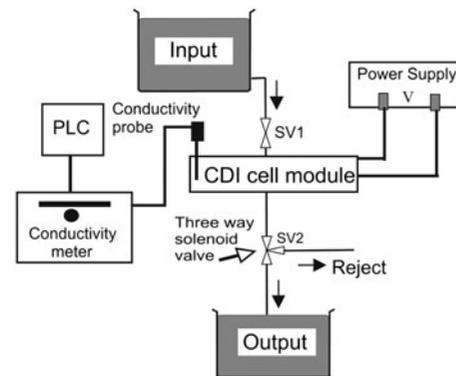


Fig. 3. Schematic of the setup with single CDI cell module.

pumping system. Suitable electromagnetically operated solenoid valves have been employed at the inlet and outlet of CDI cell module for filling and release of the effluent. Solenoid valve “SV1” is on/off type to enable filling of the module. The three way solenoid valve “SV2” at the outlet of the module has been provided with two way optional outlet, one way for the output and the other to the reject stream depending on the processing conditions. In the de-ionization cycle operation, SV2 is used to release the treated water to the output tank. During regeneration of the stack, valve SV2 is positioned to release the desorbed salts to the reject stream. A programmable logic control unit has been coupled to the setup for automated and timed valve operations during desalination and regeneration cycles.

The approach adopted is a batch operation in which the electrolyte was filled and held for treatment in the CDI module. For the desalination cycle, the electrolyte from the input tank was filled in the CDI module by opening of SV1 (SV2 being off) and treated by applying 1.2 V across the cells of the module for 20 min. At the end of the cycle, the logic control unit actuated the valve SV2 to release the treated deionized sample to the output tank. For the regeneration cycle, the electrolyte was filled in the module using SV1 and held for 20 min. In this cycle, the electrodes were electrically shorted to allow desorption of the ions to the effluent. SV2 was later actuated to release the effluent to the reject stream. The setup has been used for treating water samples with TDS up to 700 mg/l and salt removal results were analyzed.

For input samples with higher TDS, additional CDI cell modules can be connected in series, where the output of first module is fed to the next module to achieve desired deionization. The schematic of a three stage setup with three CDI cell modules is shown in Fig. 4. This setup has been used for treating samples up to TDS of 1,500 mg/l.

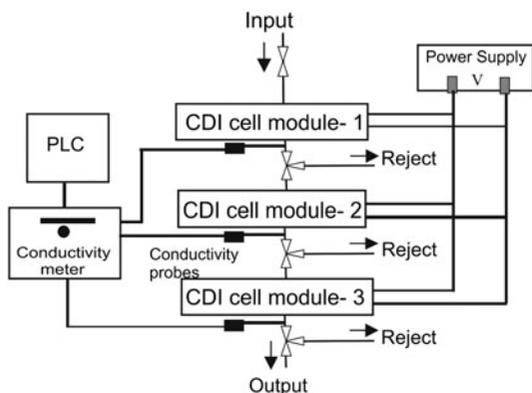


Fig. 4. Schematic of the three stage CDI setup.

3. Results and discussion

3.1. Surface area analysis

Surface area analysis results of the as prepared carbon aerogel sample (CA) and CO₂-activated (a-CA) sample obtained after activation have been compared. The N₂ adsorption–desorption isotherms at 77 K and the pore size distribution for CA and a-CA samples are shown in Fig. 5. The isotherm shows increase in volume adsorption of CA after CO₂ activation. The overlap of pore size distribution of CA and a-CA shows a marginal shift in the pore distribution but significant increase in pore volume after activation.

The surface area characteristics of these samples are summarized in Table 1. The results show significant improvement in specific surface area after activation of CA from 698 to 2,057 m²/g with mesopore contribution of 644 m²/g which compare favorably

with similar studies on CO₂ activation reported in the literature [31]. The a-CA powder obtained has been used for preparation of electrodes for CDI evaluation.

3.2. Specific capacitance measurements

The CV profiles of the sample electrode having 15 mg active material loading was obtained for scan rates of 1, 2, and 5 mV/s. The set of CV data generated for current versus voltage for each scan rate was computed using origin software to obtain the specific capacitance in accordance with Eq. (1). The graph displaying the specific capacitance versus voltage range of -0.5 to $+0.5$ V is shown in Fig. 6. The profiles have symmetrical and quasi-rectangular shape without any oxidation or reduction peaks, suggesting the ideal capacitive behavior. The average specific capacitance values evaluated by integration of the data were 108, 101, and 94 F/g for the scan rate of 1, 2, and 5 mV/s, respectively.

3.3. Adsorption capacity of electrode material

The adsorption capacity of electrode material was analyzed as described in section 2.3.3 and final TDS (C_i) of 311 mg/l was achieved. Taking into account the initial TDS (C_i) of 500 mg/l, a-CA loading (m) of 45 g, the calculated value of the specific salt adsorption capacity as per Eq. (2) is 8.4 mg/g. The value obtained compares favorably with the carbon aerogel variants as reported in the literature [6,7]. For repeatability testing of the module, repeated charge–discharge cycles were performed as shown in Fig. 7.

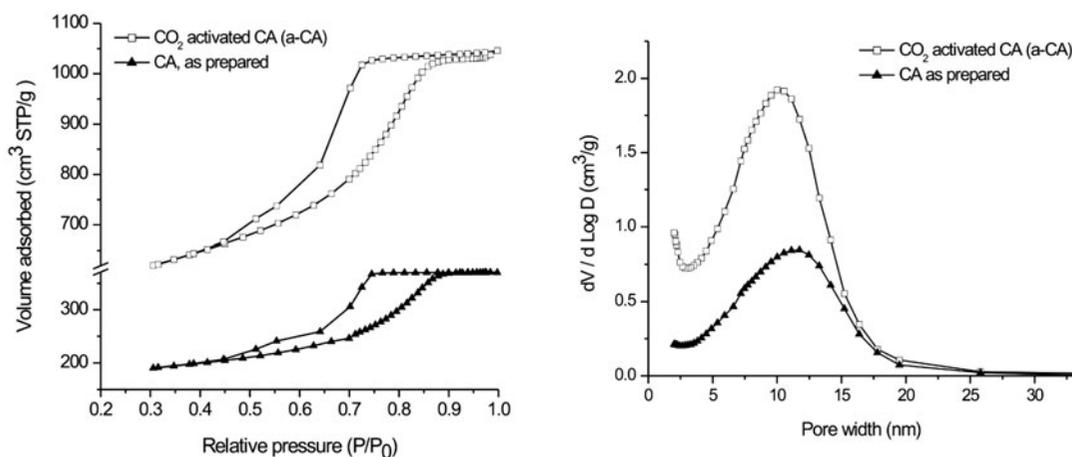


Fig. 5. N₂ adsorption–desorption isotherms and pore size distribution of CA and CO₂-activated CA.

Table 1
Surface area characteristics of CA samples

Surface area properties sample type	Total BET surface area (m ² /g)	Mesoporous surface area (m ² /g)	Total pore volume (cm ³ /g)	Average pore diameter (nm)
CA	698	274	0.6	7.2
a-CA	2,057	644	1.72	6.7

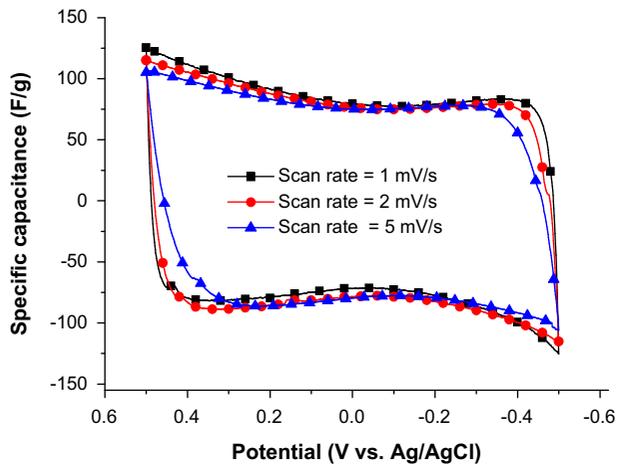


Fig. 6. CV-based specific capacitance plots at scan rates of 1, 2, and 5 mV/s.

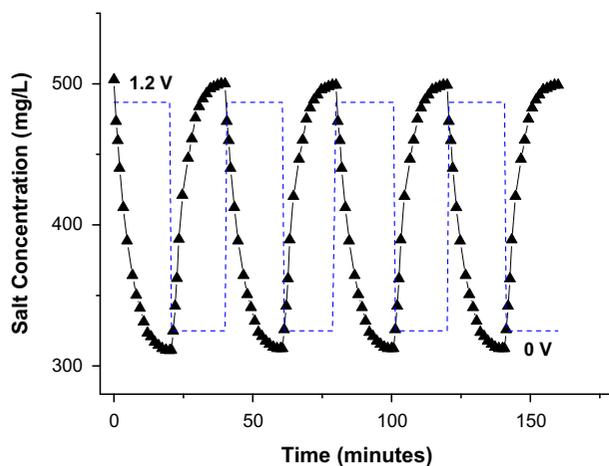


Fig. 7. Repeated charge–discharge cycles of the module.

3.4. Deionization experiments

Test samples with TDS of 400, 500, 600, and 700 mg/l were desalinated using single module-based CDI setup as described in Fig. 3 of methodology section. De-ionization followed by regeneration cycles were operated alternatively over repeated cycles and

the results of 1, 3, 5, and 10th cycles have been presented in Table 2. The results show that for the sample with input TDS of 400 mg/l, output TDS of 113 mg/l was achieved with salt removal of 72%. Further, the salt removal ratio decreased with increasing input TDS and for the input TDS of 702 mg/l, salt removal of ~60% was achieved with output TDS of 280 mg/l. The deionization and regeneration being conducted alternatively, water recovery for the setup was 50%. Concentration of the effluent released in the reject stream after regeneration cycle was also monitored for each cycle. The additional salt content released after regeneration matched with the salt adsorbed on electrodes during deionization.

A complete cycle of operation of 40 min produced 500-ml desalinated water and two set of complete cycles with total time duration of 80 min resulted in one-liter output. The voltages and current drawn were monitored and shown in Fig. 8. The average current computed for one liter output was 35 mA with a duration time of 80 min at 1.2 V. The power consumed was evaluated to be ~0.06 kWh/m³.

Samples with input TDS of 1,000, 1,200, and 1,500 mg/l were treated using two and three module setup and the outlet TDS of each stage were measured and the results have been presented in Table 3. While for the input TDS of 1,000 mg/l, two module setup was used, for higher TDS three stage setup was used. The results show that with above 80% salt removal, for input TDS levels of 1,000, 1,200, and 1,500 mg/l, output TDS of 197, 166, and 238 mg/l were obtained. The CDI cell modules showed almost no degradation with usage over six months of operation.

The salt removal values obtained for input TDS of 400 mg/l are ~72% with single cell module and ~86% for three cell module operated at 1.2 V. Our results are comparable with the studies reported in the literature. The CDI studies performed by Jung et al. used carbon aerogel with specific surface area of ~610 m²/g. They reported salt removal of ~64% for a single cell unit and ~97% for six cells unit operated at 1.7 V [37]. Nadakatti et al. utilized composite of mesoporous carbon black (mesopore surface area ~455 m²/g) with activated carbon (specific surface area ~855 m²/g) and reported

Table 2
Desalination results with single stage CDI setup

Sample	Input TDS (mg/l)	Cycle no.	Output TDS (mg/l)	Reject stream TDS (mg/l)	Av. output TDS (mg/l)	Salt removal (%)
1	400	1	110	689	113	72
		3	114	688		
		5	113	686		
		10	114	686		
2	501	1	145	855	150	70
		3	151	854		
		5	153	852		
		10	152	852		
3	598	1	204	993	205	66
		3	206	991		
		5	205	990		
		10	207	989		
4	702	1	277	1,126	280	60
		3	281	1,122		
		5	281	1,123		
		10	282	1,122		

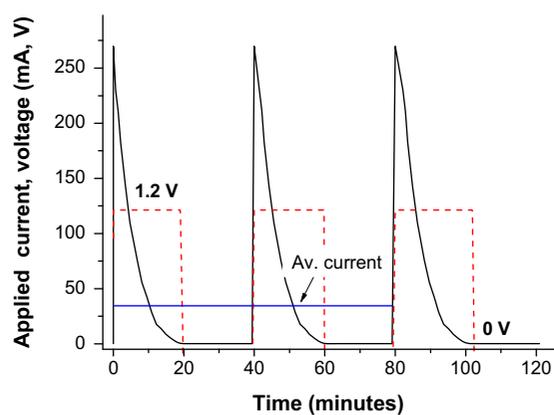


Fig. 8. Applied voltage and current for ~2 h operation of CDI cell.

salt removal ~70% for single cell operated at 1.2 V, which is comparable to the value reported in the present studies [20]. In our studies carried out at 1.2 V, the use of higher surface area CA allowed 72% salt removal with single cell module and ~86% for three cell module operation.

While for a single cell module the power consumption was ~0.06 kWh/m³, for the three cell module setup consisting of three modules in series that was used for desalination at higher TDS levels the power consumption increased to ~0.5 kWh/m³. This is because with a view to limit exposure to higher TDS level and possible contamination of subsequent stages, during regeneration cycle, the input electrolyte for the subsequent stages was drawn from the desalinated output of previous stage. For the three stage module

Table 3
Salt removal results with three stage CDI setup

Input TDS (mg/l)	Output TDS 1st stage (mg/l)	Output TDS 2nd stage (mg/l)	Output TDS 3rd stage (mg/l)	Total salt removal (%)
1,000	423	197	–	80 ^a
1,200	535	290	166	86
1,500	785	412	238	84

^aFor input TDS up to 1,000 mg/l, two stage modules were used.

this caused repeated operation of the desalination cycle for the first stage (four times) and second stage (three times) during regeneration of subsequent stages.

4. Conclusion

Mesoporous carbon aerogel having surface area of $\sim 2,000 \text{ m}^2/\text{g}$ has been used for developing electrodes for CDI application. The synergetic effect of high surface area along with mesoporous nature of the carbon aerogel resulted in achieving high salt adsorption capacity of 8.4 mg/g . CDI cell modules of 500 ml capacity with eight electrode stacks have been used to build single and multistage setups for treating water samples with moderate TDS ($500\text{--}1,500 \text{ mg/l}$). Single module-based CDI setup has been used for deionization of water samples up to TDS of 700 mg/l and output TDS of $\sim 250 \text{ mg/l}$ was obtained. For the input concentration of $1,500 \text{ mg/l}$, three module CDI setup has been demonstrated to achieve TDS of 238 mg/l , well within the desired range. Importantly, the power consumption was significantly low; $\sim 0.5 \text{ Wh/l}$ for three module desalination setup. In addition, non-dependence on chemicals and consumables and easy regeneration of electrodes make this technique a suitable alternate for rural and remote areas.

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