Capacitive mixing is a newly emerging technique for the production of renewable energy from the controlled mixing of river water and seawater. Energy extraction is provided by the potential rise in electrodes held in a fixed charge, as a response to the concentration change. Therefore, electrodes that exhibit high potential variation as a response to concentration change (negative rise for the cation-capturing electrode and vice versa) are highly desirable. In this work, electrodes that can accommodate mostly cations within their porous structure are discussed. In accordance to the modified Donnan (mD) model of the electrical double layer, it is expected that such electrodes will display the highest potential change as a response to concentration change (while being held with a fixed charge). Using appropriate selective activated carbon electrodes, high potential rise, around 50 mV, was observed as a response to concentration change, when the concentration of NaCl solutions was changed from 1 M to $10^{-1}$ M and from $10^{-1}$ M to $10^{-3}$ M. Such a capability of potential rise of selective electrodes can serve as a good basis for energy extraction by capacitive mixing.

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The energy that dissipates due to the mixing of two solutions with different salt concentrations, such as seawater and river water, can be, in fact, partially harvested by controlled mixing. The upper limit of energy that can be obtained from mixing two ideal solutions with different concentrations is given by the Gibbs free energy of mixing. For example, the maximum energy that can be obtained from mixing 1 Liter of seawater and 1 Liter of river water is about 2 kJ.

There are several methods for energy extraction by controlled mixing of seawater and river water, including pressure-retarded osmosis (PRO), reversed electro-dialysis (RED) and vapor-pressure difference utilization. Recently, energy extraction from salinity difference by swelling and shrinking of hydrogels has been suggested. However, to the best of our knowledge, these methods have to be proven as commercially viable.

Capacitive mixing (CAPMIX), first proposed by Brogioli, is a membrane-free technique. In general, the energy extraction is based on potential differences developed as a result of double-layer (i.e., electrostatic) interactions between high-surface-area electrodes through which solutions of different NaCl concentrations flow. In principle, the exchange between two bulk solutions with different salinities that are in contact with two charged high-surface-area electrodes leads to changes in their capacitance and, consequently, in the potential of the electrochemical cell utilizing them:

$$\Delta V = \frac{Q}{\Delta C}$$  \hspace{1cm} \text{(1)}$$

where $\Delta V$ [V] is the electrodes-potential difference [i.e., the sum, in absolute values, of the electrical double layer (EDL)-related potentials of both the cation- and anion-capturing electrodes], $Q$ [C] is the total charge of the electrodes (equal but with opposite signs) and $\Delta C$ [F] is the change in capacitance resulting from the mixing. A decrease in cell potential (for a cell with a fixed constant charge) should result in an increase in the stored energy:

$$E = \frac{1}{2} Q \Delta V$$  \hspace{1cm} \text{(2)}$$

Capacitive mixing can be subdivided into three main categories: the conventional CAPMIX (or externally charged CAPMIX), Donnan-potential capacitive mixing (CDP) and “zero charging” CAPMIX.

Basically, the conventional capacitive mixing cycle consists of four steps. In all the approaches, there is a consecutive alternation between the solutions. First, the high-surface-area electrodes accumulate charge through an external load, up to a desirable potential. Next, the bulk solution is exchanged with a solution of a lower salinity, and the voltage drop across the double layer is increased. In the next step, the cell is discharged to a defined potential through an external load. In this phase, energy is extracted from the cell. In the last step, the electrodes are brought into contact with the high-salinity solution, bringing the electrodes back down to their initial potential. CDP involves the use of ion-selective membranes that are in conjugation to the porous carbon electrodes. The alternation between river water and seawater produces continuous power, and an external power supply is not required. Sustainable power can be generated by the presence of Donnan potentials between the two sides of each electrode, which, in turn, induces ionic currents that store charge into the electrodes by the formation of EDLs at the electrode/solution interfaces.

In “zero-charged CAPMIX,” the power can also be generated without the need for an external power supply. Energy extraction without the need for a power supply can be performed by choosing proper electrodes that are similar in their spontaneous potential but opposite in their potential response to concentration change. However, we address the conventional CAPMIX. An equivalent electrical circuit for the conventional CAPMIX cell consists of two capacitors connected by a series of in-line and in-parallel resistors, analogous to a solution resistance and charge-transfer resistance, respectively (Figure 1). Therefore, the contributions to the energy extraction for both the cation-capturing and anion-capturing electrodes can be calculated separately, according to

$$\Delta E = \left[ \int \varphi_{c} dQ \cdot \int \varphi_{a} dQ \right]_{\text{Low}} - \left[ \int \varphi_{a} dQ \cdot \varphi_{c} dQ \right]_{\text{High}}$$

$$= \left[ \int \varphi_{c} dQ_{\text{Low}} - \int \varphi_{a} dQ_{\text{High}} \right]_{\text{High}} - \left[ \int \varphi_{a} dQ_{\text{Low}} + \int \varphi_{c} dQ_{\text{High}} \right]_{\text{Low}}$$  \hspace{1cm} \text{(3)}$$

where $\varphi_{a}$ and $\varphi_{c}$, respectively, represent the potential drop from the electrode surface to the bulk solution for the cations and anions capturing electrodes (in both high and low salinity) with respect to a reference electrode. Q is the charge that is exchanged between the electrodes and $\Delta E$ is the energy which is extracted from the process. Consequently, the potential rise is highly important as a response to concentration change. The whole cell potential reported in most publications remains low, typically around 30–50 mV (15–25 mV for each electrode).

It has previously been demonstrated that a selective carbon electrode toward sodium can be fabricated considering a restricted pore-size distribution size function and oxidative surface treatment. By

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controlled activation/oxidation, a precise pore-size distribution can be obtained with activated carbons on the order of a few angstroms to a few nanometers. The surface of the carbon can be modified by oxidation, for instance, using concentrated aqueous HNO₃ solutions. It was shown that treating a pristine carbon with nitric acid (even at room temperature) should result with some incremental changes in the surface area, whereas treating a well-developed porous carbon should not induce significant changes at surface area. However, the reaction of carbon with nitric acid should result with the introduction of oxygen-containing functional groups (such as carboxylic, lactonic and phenolic groups). We previously exploited cation selective electrodes as an approach to achieve a maximum of charge efficiency in capacitive deionization (CDI) processes. Hatzell has demonstrated a negative potential rise of about −30 mV (with solution exchange between 0.5 and 0.011 M) for a carbon electrode originating from hardwood that has been under prolonged exposure to nitric acid. She attributes the negative drop in the potential to the transport of adsorbed chloride ions from the oxidized electrode surface into the less concentrated solution in order to reach stabilization by coordinating with more water molecules (driven by the absence of surrounding ions in the solution possessing lower ionic strength).

In this work we show a potential rise for activated carbon electrodes upon changing solution concentration, which is explained by virtue of the electrodes’ selectivity toward sodium ions within their porous structure. It is also shown herein that anion-exclusion carbon electrodes (accommodation of sodium and exclusion of chloride ions) can be easily fabricated by one activation/oxidation step.

Experimental

Analytical-grade NaCl (greater than 99%) was purchased from Frutarom, Israel. HCl (36%) and HNO₃ (70%) aqueous solutions were obtained from Sigma-Aldrich. The precursor for the carbon material was cellulose filter paper (Whatman Inc.). The carbon papers were produced in the same manner as that reported in previous publications. The temperature program for carbonization under argon flow includes a slow ramp of 0.58 °C min⁻¹ in the 150–400°C range, and then an increase of 18°C min⁻¹ from 400 to 600°C. Activation of the carbon paper was achieved with an aqueous HNO₃ solution at room temperature by dipping carbon-paper samples into the HNO₃ (70%) solution for 25 minutes.

The electrochemical setup was a simple three-electrode cell. The electrode investigated was attached to a platinum current collector. The reference electrode, placed adjacent to the working electrode, was a standard saturated-calomel electrode (SCE). The three electrodes were dipped in a chamber that enables solution exchange by pumping solutions from a reservoir via an entrance and drain holes. The reservoir’s solutions used contained 10⁻⁸, 10⁻¹, 10⁻² and 10⁻³ molar concentrations of NaCl. The setup resembles that reported by Brogioli et al., but the electrodes were connected to a potentiostat instead of an external load.

Electrochemical measurements, cyclic voltammetry (CV) and galvanostatic charging, were conducted with a PGSTAT Autolab electrochemical measuring system from Ecco Chemie, Inc. The specific capacitance was calculated from the current values of the voltammograms according to \( C = I/dV/dt \), where \( I \) is measured in amperes per gram of electrode material, \( V \) is the electrode potential in volts, \( t \) is the time (in seconds) and \( C \) is the capacity magnitude in farads per gram.

HRSEM images of the treated carbon paper were obtained using JEOL-JSM-7000F electron microscopes equipped with an EDX microanalysis system. The specific surface area of treated carbon paper was measured by the Brunauer–Emmett–Teller method using a Micromeritics Gemini 2375 analyzer with nitrogen adsorbate at 77 K.

Results and Discussion

Figure 2 shows a steady-state multiple cyclic voltammetry curves of a carbon paper electrode that was exposed to a concentrated HNO₃ solution at room temperature during 25 minutes in 10⁻⁸, 10⁻¹ and 10⁻³ M NaCl solutions. At relative negative potentials, the differential capacitance is significantly higher than that at the positive potentials. This result is in agreement with our previous research, where we showed that very short exposure of pristine carbon paper to HNO₃ (less than 15 minutes at room temperature) is insufficient for the development of an accessible porous structure toward sodium electro-adsorption whereas, after 30 minutes of exposure, the porous structure enables the accommodation of both sodium and chloride ions (but at different ratios). Here, an intermediate exposure time was used.
The electrostatic environment created by the fixed charges at the pores entrances leads to a selective differentiation between adsorption of anions or cations. In most cases, the pores are selective toward ions carrying charges opposite to their own.\textsuperscript{21} This behavior is, most likely, due to the negatively charged carboxyl groups present at the carbon-pore entrances.

High-resolution scanning electron microscopy (HRSEM) with energy-dispersive X-ray (EDX) analysis as an oxygen- and carbon-mapping technique was employed in order to examine the surface morphology of the carbon paper after treatment. After treatment by nitric acid solution a dispersion of small cavities (in the range of 4–20 nm) on the carbon surface can be observed (Figure 3a). According to the EDX results, the HNO\textsubscript{3}-treated carbon paper exhibits around 10% (atomic) oxygen on the carbon surface. This confirms the presence of oxygen-containing surface groups on the HNO\textsubscript{3}-treated carbon paper. Figure 3b exhibits the pore-size distribution of HNO\textsubscript{3}-treated carbon according to the Barrett–Joyner–Halenda (BJH) model (the inset shows the adsorption–desorption isotherm). According to BET model, the specific surface area of HNO\textsubscript{3}-treated carbon is 41 m\textsuperscript{2}/g.

The asymmetry at the cyclic voltammetry is attributed to a combination of the molecular-sieving effect of the electrode and ion-charge interaction.\textsuperscript{16} For instance, even when the average pore size is larger than the chloride-ions dimension, there is a repulsion of anions due to the negatively charged carboxylic surface groups. Thereby, the pores can accommodate sodium ions (they are large enough for that) but repel chloride ions. Anyhow, the mechanism for sodium ions adsorption and chloride ions exclusion within the porous carbon matrix, cannot be explained solely by the relation of pore-size distribution to the size of the ions.

Recently, the modified Donnan (mD) model\textsuperscript{22} proposed by Biesheuvel and Bazant\textsuperscript{23} has been suggested to better describe the EDL within the micropores of activated carbon electrodes. The classical Gouy-Chapman-Stern (GCS) model is used to describe equilibrium adsorption of salt and charge in electrodes as a function of cell voltage and ionic strength, but, however, with porous electrodes, the diffuse layer has a typical extension into the free solution of the order of ∼3 nm (10 mM) and overlapping of the diffuse layer within the porous carbon exists. Analogous to charged gels, it is assumed that within the carbon particles the pores space has a constant electrostatic potential and the ions within the pores of the carbon are homogeneously distributed without gradients. Consequently, it is assumed that porous carbon electrodes display two types of porosities, macroporosity and microporosity. Macropores are simply inter-particles spaces, where the transport of ions passes the electrode takes place. Micropores are the zone where the counter-ions are preferentially stored when the electrode is polarized and where the EDL is actually formed. The “modification” is the inclusion of charge-free Stern layer and considering an additional attractive force for the ions within the carbon particles.

According to the model, the potential of an individual electrode, ϕ\textsubscript{i,j}, (ϕ is the potential drop from the electrode surface to the bulk solution) is given by

\[ \phi = \Delta \phi_i + \Delta \phi_j, \]

where Δϕ\textsubscript{i} and Δϕ\textsubscript{j} are the potential drops over the Stern layer and the Donnan potential between the micropores and macropores, respectively.

The relation between the ions concentration in the micropores and the ions in the macropores is given by

\[ C_{j,\text{micro}} = C_{j,\text{macro}} e^{-Fz_1/RT}, \]

where C\textsubscript{j,macro} and C\textsubscript{j,micro} are the macropore and micropore concentration of a specific type of ions, respectively, z\textsubscript{i} is the charge number of the ions, F is the Faraday constant (C/mol), R is the gas constant (J mol\textsuperscript{−1} K\textsuperscript{−1}), T is the temperature (K) and μ (J) is the attractive excess chemical potential related to the chemical attraction between the ions and electrode surface.

In this work (for simplification purposes) we consider the classical modified Donnan model of the EDL. It is worth mentioning, that recently, a better Donnan model (the amphoteric Donnan model (“amp-D”))\textsuperscript{22,25} was suggested. Instead of using the attraction chemical term, “surface charge” is included. This model can address, for instance, other CDI architecture, inverted -CDI, where expulsion of ions are dominant (instead of adsorption) upon voltage application. However, in the capacitive mixing mode of operation, upon the solution exchange step, the charge (whatever the source of the charge is) on

\[ I_{\text{cap}} = \frac{Fz_1 C_{j,\text{macro}} A}{D_j RT}, \]

where F is the Faraday constant (C/mol), z\textsubscript{i} is the charge number of the ions, C\textsubscript{j,macro} is the ion concentration in the macropore solution, A is the geometric area of the carbon paper electrode, D\textsubscript{j} is the ion diffusion coefficient in the macropore solution, and RT is the thermal energy.

\[ I_{\text{cap}} = \frac{Fz_1 C_{j,\text{macro}} A}{D_j RT} \]

Figure 2. Multiple steady-state cyclic voltammetry curves of a representative carbon-paper electrode that was etched in a concentrated nitric acid solution at room temperature for 25 minutes in 10\textsuperscript{6}, 10\textsuperscript{5} and 10\textsuperscript{4} M NaCl solutions. The scanning rate was 1 mV/s. The differences of the capacitance of the electrode (the differential capacitance is Cd = d\textsubscript{I} /d\textsubscript{E}) with respect to the concentration of the solution, are significant.

Figure 3. a - HRSEM micrographs of HNO\textsubscript{3}-treated carbon; Scale bars 300 nm; b - Pore-size-distribution of HNO\textsubscript{3}-treated carbon paper, according to the BJH model (in the inset, nitrogen adsorption–desorption isotherm (at 77 K)).
the electrode is assumed to be constant, thus the classical m-Donnan model can be employed.

However, in our case, only small portion of the pores’ volume, in the carbon matrix can actually accommodate anions within its microporous structure. Analogous to the potential of biological membranes in living cells, the equilibrium potential across the cells’ membranes (or the boundary potential between the micro and macro pores) should be calculated taking into account the selectivity of membrane’s permeability toward each particular ion. Therefore, Equation 5 can be extended to take the form of the Goldman–Hodgkin–Katz (GHK) equation,\textsuperscript{26} except that the permeability (in meters per seconds) should be regarded as a relative portion of the pores’ volume that can accommodate a particular ion and considering a zero net flux of permeable ions at equilibrium. In this way, we can calculate the potential difference between the inside (micropores) and outside (macropores) of the activated carbon electrodes, from the relative contribution of permeable ions at equilibrium:

\[
(\mu + \Delta \phi_d) = \frac{RT}{F} \ln \left[ \frac{p_{Na^+}[Na^+]_{macro} + p_{Cl^-}[Cl^-]_{micro}}{p_{Na^+}[Na^+]_{micro} + p_{Cl^-}[Cl^-]_{macro}} \right].
\]  

Or

\[
(\mu + \Delta \phi_d) = \frac{RT}{F} \ln[p_{Na^+}[Na^+]_{macro} + p_{Cl^-}[Cl^-]_{micro}]
- \frac{RT}{F} \ln[p_{Na^+}[Na^+]_{micro} + p_{Cl^-}[Cl^-]_{macro}],
\]

Where \( p_{Na^+}[Cl^-] \) is the permeability of each ion, across the entries of the carbon microporous structure.

Due to electro-neutrality constraints within the micropores, the charge (volumetric charge density, \( \sigma_{micro} \)) should obey

\[
\sigma_{micro} = \sum_j z_j C_{j,micro}.
\]

For an electrolyte containing only Na\(^+\) and Cl\(^-\) ions, Equation 8 can be rewritten:

\[
\sigma_{micro} = [Na^+]_{micro} + [Cl^-]_{micro}.
\]

In the case where \( p_{Na^+} \gg p_{Cl^-} \), \( p_{Na^+} \) can be omitted and Equation 9 reduced to

\[
\sigma_{micro} = [Na^+]_{micro}.
\]

The sodium-ion concentration within the micropores can be replaced with the micropore volume charge density and be therefore regarded as a constant, and Equation 7 converges to

\[
(\mu + \Delta \phi_d) = \frac{RT}{F} \ln [Na^+]_{macro} - \frac{RT}{F} \ln [\sigma]_{micro}.
\]

The Stern potential is given by

\[
\Delta \phi_{St} = \frac{RT}{F} \frac{\sigma_{micro}}{C_{st}}.
\]

Where \( C_{st} \) is a volumetric Stern layer capacity in farads per cubic meter. For \( C_{st} \), the following expression\textsuperscript{12} can be used:

\[
C_{st} = C_{st,0} + \alpha \cdot \sigma^2,
\]

Where \( \alpha \) is an empirical constant.

The attractive excess chemical potential \( \mu \) for each ion in the micropores has been shown to be inversely proportional to the micropore total ion concentration.\textsuperscript{23} In addition, the accumulated charge in the capacitive mixing operation cycle is believed to be constant and, therefore, for the differentiation of \( \psi \) with respect to \( \log[Na^+]_{macro} \), the terms \( \mu \) and \( \Delta \phi_d \) can be excluded and the derivative reaches its maximal value as follows:

\[
\frac{d \psi}{d \log[Na^+]_{macro}} = \frac{d \Delta \phi_d}{d \log[Na^+]_{macro}} = \frac{2.3RT}{F}.
\]

Since the micropores can accommodate mostly sodium ions within their structure, the electronic charge is compensated only by the sodium ions. Upon solution exchange, the sodium ions are prohibited from leaving the pores and the only variable is the sodium ions concentration in the macropores leading to a maximal potential change as a function of a solution concentration change, while the electrode is held with a fixed charge. Prior to solution exchange, the electrodes were brought to a potential of +14 mV vs SCE after a constant charging at a current corresponding to −5 mA g\(^{-1}\). The current was set to relatively low values in order to avoid a mistaken interpretation of a possible voltage increase as the cause of the charge re-distribution in the carbon micropores.\textsuperscript{28,29} Figure 4a shows the variations in the potential of these electrodes (vs SCE) as a function of solution exchange (i.e. Na ions concentration outside the electrodes). When these selective electrodes were flushed with solutions of different concentration, their potential became lower as the solution was more diluted. In accordance to Eq. 11, it is expected that negatively charged electrodes should experience a negative increase in their potential upon changing the solution to a solution with a lower concentration. When these electrodes were flushed with 10\(^{-3}\) M NaCl solution, their potential went down to too low values that could enable reduction of dissolved oxygen on them.\textsuperscript{30,31} Thereby, we address the potential changes of these electrodes with solutions which concentration varies over 3 orders of magnitude only (1, 10\(^{-1}\) and 10\(^{-2}\) molar of NaCl).

In a previous work,\textsuperscript{36} we explored the role of dissolved oxygen reduction reaction on the self-discharge mechanism of the cation capturing electrodes in CDI processes. It was demonstrated that the main cause for charge leakage is the presence of oxygen in the feed water. In Figure 3b, the self-discharge of the carbon-paper electrode is provided. At potentials lower than −150 mV (vs SCE), the leakage, in terms of potential drop, namely, \( d\psi/dt \), can be as high as 300 µV/s. However, at the potential range of −50 to −150 mV, the average leakage current is about 40 µV/s.
Since the electrode undergoes self discharge, equilibrium with its potential is never reached at any point. However, we address the potential change after about 150 seconds, since the solutions exchange. The high potential rise (~51 mV, Figure 4a) can be explained by considering that the dominant change in the potential upon solution exchange is attributed to the changes in the electrical double layer component, in accordance to the mD model.

For the evaluation of the specific energy (J g\(^{-1}\)) that can be extracted from the fabricated cation-capturing electrodes, the electrodes potential versus the stored charge is provided in Figure 4, which exhibits exchanged charge-voltage curves for solutions of 10\(^{-1}\) and 10\(^{-2}\) M NaCl. The curves related to each solution in Figure 5, voltage profiles vs. accumulative exchange charge, were obtained by applying constant discharging current (corresponding to ~5 mA g\(^{-1}\)) to the activated carbon paper electrodes in two NaCl solutions of different concentration. The operation cycle for the (anion exclusion) cation capturing electrode, is as follows: A- is the initial point: a positively charged electrode in high salinity solution. From A to B the electrode is cathodically charged by an external power source in the high NaCl concentration solution. B- flow of low-salinity solution, the potential drops to C. From C to D the electrode is cathodically charged by an external power source in the high NaCl concentration solution. When the electrode reaches point B, a flow of low-salinity solution takes place, the potential drops to C. From C to D the electrode undergoes discharging, capacity and energy are extracted. When the electrode reaches point D, a flow of high-salinity solution takes place, the potential rises and the electrode gets back to its initial point - A. The areas that are enclosed in red and blue dashed lines represent the upper limit of the energy that can be extracted, where the exchanged charge corresponds to the exchange of 1 and 5 C g\(^{-1}\), respectively.

The area enclosed by the cycles is the upper limit of energy extraction for the cation-capturing electrodes. In accordance with other research published in this field, the specific energy was calculated according to the exchanged charge corresponding to 1 and 5 C g\(^{-1}\). The value for zero charge was arbitrarily set to the spontaneous potential. The spontaneous potential reflects a mix potential that is affected by active faradaic redox reactions such as quinone-hydroquinone, oxygen reduction reaction and protonated surface groups rather than the real potential of zero charge. However, for the purpose of estimating the specific energy extraction, which is provided by the integration of the electrode voltage with the exchanged charge, setting zero charge to the spontaneous potential is sufficient. The specific energy of such an electrode is calculated to be 0.288 J g\(^{-1}\) (exchanged charge corresponding to 5 C g\(^{-1}\)).

The specific energy extraction of the cation-capturing electrode is a function of both the potential variation as a response to concentration change and the specific capacitance of the electrodes (Equations 1 and 2). The specific energy of the cation-capturing electrode can be maximized by using a relatively wider open porous structure, which, in turn, provides higher specific capacitance, but with similar anion-exclusion properties. One way to achieve this is, for instance, to adjust the pore opening of a high-capacitance carbon electrode by the chemical vapor deposition (CVD) technique, following adequate surface treatment. In this way, the anion-exclusion property can be realized together with the high surface area of the electrode and hence, high capacity. Here, however, we stress the point that the potential increment of electrodes with a fixed charge, as a response to a concentration change, can be optimized by the design of electrodes with anion- or cation-exclusion properties.

Conclusions
A carbon with anion-exclusion properties was fabricated by carbonization of cellulose filter paper followed by short exposure to concentrated nitric acid solution at room temperature. The anion-exclusion properties (the ions present are sodium and chloride) were confirmed by cyclic voltammetry measurements. Following our calculations, based on the modified Donnan model of the electrical double layer, carbon electrodes as those described and discussed herein, should display high potential change as a response to concentration change. In practice, the fabricated carbon electrodes we used, showed indeed a high potential change of about 51 mV when the solution concentration was changed from 1 M to 10\(^{-1}\) M and (10\(^{-1}\) M to 10\(^{-2}\) M), what may corresponds to a specific energy of ~0.3 J/g (with accumulated charge corresponding to 5 C/g). These preliminary findings may set a basis for further development of energy storage and conversion devices, based on selective activated carbon electrodes.

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