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# Investigating Saltwater Desalination by Electrodialysis and Curriculum Extensions To Introduce Students to the Chemical Physics of Polymeric Ion-Exchange Membranes

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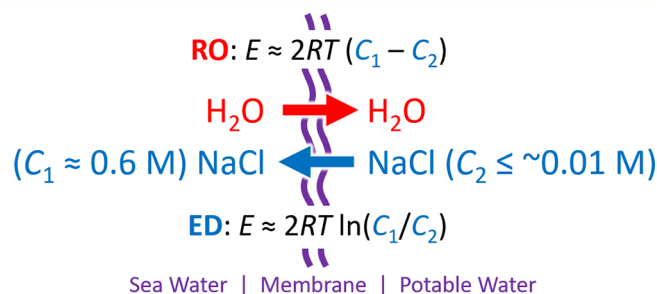
## S Supporting Information

**ABSTRACT:** Even though our planet contains large reservoirs of water as oceans, access to drinking water is a global concern because large amounts of salt are fatal to humans. While electrochemical desalination, that is, electrodialysis, is a process that is capable of generating potable water from salt water, most chemistry curricula do not teach this process. Therefore, we developed a curriculum and accompanying low-cost activity to expose students from middle school to undergraduate studies to the concept of electrodialysis and the importance of polymeric ion-exchange membranes in the electrodialysis process. The curriculum provides background by introducing the students to issues of water access, current state-of-the-art solutions and the scenarios where they are optimal, and the urgent need for alternative and innovative processes for clean, potable water generation. The concepts and techniques presented in this curriculum cover those relevant to desalination, which encompass several physical phenomena that span multiple disciplines. The supporting activity that accompanies this curriculum allows students to perform electrodialysis and monitor the progress of the reaction using common pH indicators, which inherently includes many concepts that are relevant to general chemistry. The scientific depth of this curriculum is easily adjusted to challenge students at various levels of expertise.

**KEYWORDS:** High School/Introductory Chemistry, First-Year Undergraduate/General, Environmental Chemistry, Conductivity, Electrochemistry, Ion Exchange, pH, Water/Water Chemistry, Polymer Chemistry, Acids/Bases

## INTRODUCTION

Scarcity of clean, potable water is a problem of immediate and enormous concern. It affects people in both developing and developed nations and is the root of numerous violent conflicts.<sup>1,2</sup> The United Nations has projected that in less than 15 years, nearly half of the global population will live in areas of water stress.<sup>3</sup> Moreover, most of these people will live in developing nations. In the United States, people in Southern California are experiencing a prolonged drought, which affects the entire nation because California is the major supplier of domestic produce.<sup>4</sup> While water is relatively abundant on Earth, more than 96% of it contains salt at concentrations that are unhealthy to humans and plants. Therefore, technologies to desalinate salt water and convert it to potable water are hugely important. In developed nations, with infrastructure to support an electric grid and the capital and labor to construct nearly billion-dollar desalination plants, reverse osmosis (RO) is the state-of-the-art commercial means used to desalinate seawater.<sup>5-7</sup> The RO process occurs through pressurization of a container of salt water, which forces water molecules through a semipermeable membrane that excludes most solutes, including salt ions, resulting in less salty water on the other side of the membrane (Figure 1). Desalination by RO occurs when the external applied *pressure* opposes and exceeds the natural osmotic pressure between the salt water and the desalinated water on the other side of the membrane. Electrodialysis (ED) is a technique that is comparable to RO in terms of energy



**Figure 1.** Diagram depicting two desalination processes: reverse osmosis and electrodialysis. The goal of RO is to transport water (red), and the energy ( $E$ ) requirement to do so is proportional to the difference in salt concentration across the membrane (purple wavy lines). The goal of ED is to transport salt (blue), and the  $E$  requirement to do so is proportional to the logarithm of the ratio of the salt concentrations on each side of the membrane. In each case, the constant of proportionality is  $2RT$ , where  $R$  is the ideal gas constant ( $8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ ) and  $T$  is temperature (in K).

requirements.<sup>8-13</sup> Desalination by ED occurs when an external potential is applied across several chambers containing water of

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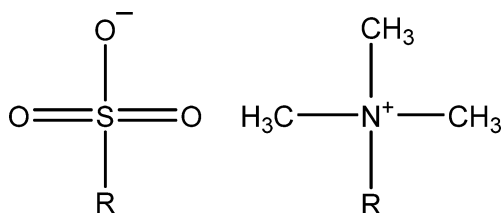
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differing salinity such that the *electric potential* opposes and exceeds the natural chemical potential difference between the charged species in the solutions. This forces salt ions through a series of permselective polymeric ion-exchange membranes, each of which predominantly transports ions of one charge type, resulting in net transport of ions away from the salty water, thus desalinating it (Figure 1).

The activity presented herein demonstrates desalination by ED as well as its relevance to concerns of global importance, such as desalination of salt water to potable water. It introduces the chemical physics and mechanisms of polymeric ion-exchange membranes and topics common to chemists and chemical engineers, notably pH, dialysis, and electrochemistry.

## TECHNICAL BACKGROUND

Two of the most important physical processes that dictate the performance of desalination by ED are mass transport of ions (i.e., rate) and membrane thermodynamics (i.e., energetics). Desalination constitutes a collection of processes that are common to plant-scale chemical engineering, including both RO and ED. A mathematical and physical description of basic transport phenomena is provided in the [Supporting Information](#). The thermodynamics of membrane processes for ED is primarily dictated by the net charge of the functional groups that are covalently bound (i.e., fixed) at the membrane surface. The most common charged groups are sulfonate anions for cation-exchange membranes (CEMs) and quaternary ammonium cations for anion-exchange membranes (AEMs) (Figure 2).<sup>14,15</sup> The presence of these fixed charges affords CEMs with



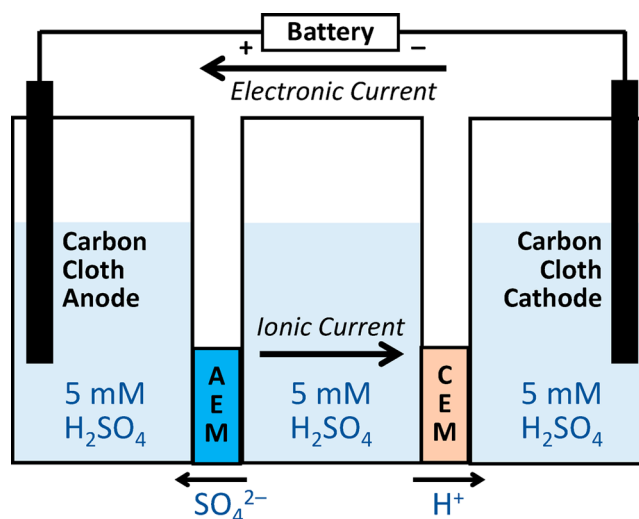
**Figure 2.** Most common fixed-charge groups found in ion-exchange membranes: (left) sulfonate ions in CEMs and (right) quaternary ammonium ions, e.g., trimethylammonium, in AEMs.

preferential transport of cations through their bulk and affords AEMs with preferential transport of anions through their bulk. A physical explanation of this basic membrane physics is presented in the [Supporting Information](#). During desalination by ED, CEMs predominantly transport Na<sup>+</sup>, but for the purpose of the activity associated with this curriculum, Na<sup>+</sup> can be replaced by H<sup>+</sup> because these ions have the same charge and the proton concentration is easier to detect using simple equipment.

## EXPERIMENTAL METHODS

### Electrochemical Setup

The experimental setup for the activity is shown in Figure 3 and consists of three chambers, each constructed from a standard disposable 4.5 mL square-bottom plastic cuvette. A hole (8 mm in diameter) is drilled near the bottom of one side of each of the two outer cuvettes, and two holes are drilled through opposite sides of the middle cuvette. The holes are then covered in Teflon tape by wrapping it around the walls of the cuvette twice, and then, using scissors, the tape covering each



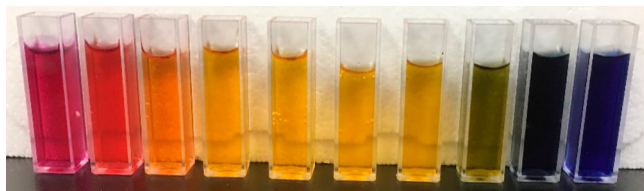
**Figure 3.** Diagram of the cell setup consisting of three cuvettes, two ion-exchange membranes (CEM and AEM), and two carbon cloth electrodes that are slid down the inside end faces of the outer cuvettes and connected to a battery using alligator clips and wires. Also shown are the directions of predominant ion (SO<sub>4</sub><sup>2-</sup>, H<sup>+</sup>) transport through the membranes and the net current flows during ED in solution (bottom) and through the electronic circuit (top).

hole is cut out and removed. The Teflon tape serves as a gasket to form a watertight seal with the membranes. Two commercial polymeric ion-exchange membranes are placed between pairs of the cuvettes, and the completed cell is then clamped together with a C-clamp to ensure watertight seals. Driving an anodic reaction (oxidation) in the chamber in contact with the AEM and a cathodic reaction (reduction) in the chamber in contact with the CEM forces ions in the center cuvette through the membranes on the basis of their charges such that cation transport occurs predominantly through the CEM and anion transport occurs predominantly through the AEM. This process desalinates (deionizes) the water in the central chamber via ED. From here on we refer to the outer chamber in contact with the AEM as the anode chamber and the outer chamber in contact with the CEM as the cathode chamber.

### Deionization with Acid

Desalination is a subset of a broader technique called deionization where instead of solely removing mineral salt ions, ions in general (e.g., protons and conjugate bases) are removed. Desalination and deionization obey the same physics that dictates the rate (mass transport of ions) and energetics (membrane thermodynamics) of these processes. Replacement of salt with acid is useful for a low-cost version of this activity because acid concentration is facile to measure precisely using a pH-sensitive colorimetric indicator, whereas salt concentration is not. In the activity described herein, aqueous H<sub>2</sub>SO<sub>4</sub> is used as the acid in place of NaCl, such that H<sup>+</sup> ions are transported by the CEM instead of Na<sup>+</sup> ions and SO<sub>4</sub><sup>2-</sup> ions are transported by the AEM instead of Cl<sup>-</sup> ions. Thymol blue is used as the colorimetric pH indicator. Thymol blue has two pK<sub>a</sub> values (1.6 and 8.9), and therefore, it exhibits two distinct color transitions (red-to-yellow and yellow-to-blue) depending on the activity of protons in the solution, i.e., pH (Figure 4).<sup>16–18</sup> The color changes occurring at low pH are important in this activity to monitor the deionization process of ~0.01 M acid (pH ≈ 2.0).

There is not a strict requirement on the exact type of material used for the current-carrying electrodes; however, in the activity



**Figure 4.** Thymol blue pH indicator at pH values of 1 to 10 in steps of 1 (from left to right).

described herein, carbon cloth electrodes were positioned inside the outer chambers and connected to a 9 V battery. ED occurred with the application of any potential beyond that required to drive two electrochemical redox half-reactions, which were likely  $4\text{H}^+ + 4\text{e}^- \rightarrow 2\text{H}_2$  and  $2\text{H}_2\text{O} \rightarrow \text{O}_2 + 4\text{H}^+ + 4\text{e}^-$  (water electrolysis at  $-1.23\text{ V}$ ) when  $\text{H}_2\text{SO}_4$  was used. Therefore, 9 V was a large excess of potential, but it helped facilitate rapid deionization; generally, the larger the potential, the faster the electrochemical reactions. It is critical that the anode of the ED cell be connected to the “+” terminal of the battery and the cathode of the ED cell be connected to the “-” terminal of the battery. If not, the central chamber will become enriched in ions instead of depleted of ions as current is passed. After 30 min, the battery was disconnected and thymol blue pH indicator was added to each chamber. Thymol blue should not be added while the battery is connected because it reacts at the electrodes and degrades.

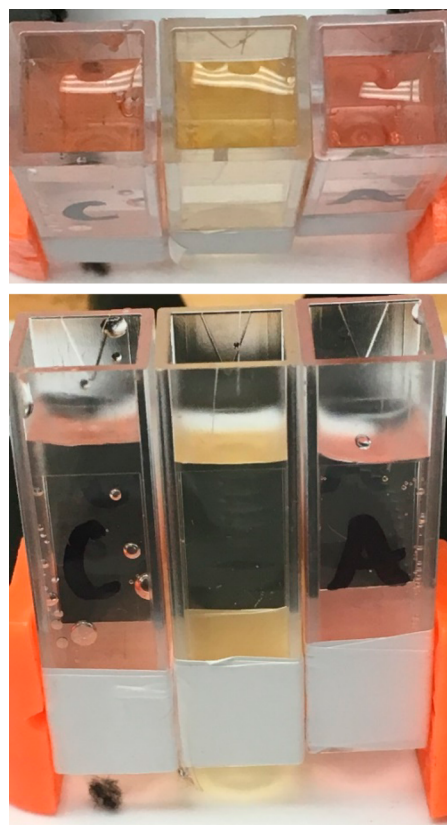
Several possible variations of the activity are discussed in the [Supporting Information](#), including the use of a different power source, type of electrode, pH indicator, type of acid and/or salt instead of acid and operation of the cell for a longer time.

### ■ SAFETY CONSIDERATIONS

This activity uses dilute but caustic solutions that are eye, skin, and respiratory irritants, and therefore, skin contact, eye contact, and inhalation should be avoided through use of proper personal protective equipment. Moreover, prior to initiating the experiments, a procedure for safe disposal of these solutions must be known. Another potential hazard is electrical shock from the 9 V battery; therefore, during ED the battery and cell should not be handled. Moreover, after the ED process is complete the leads should be disconnected from the battery terminals in order to prevent the possibility of forming an electrical shunt that can result in high currents being passed and large dissipation of heat. Additionally, if this activity is performed with younger children, they can assemble the dry electrochemical cell but it is advised that a supervisor add the acid solution to each chamber, immerse the electrodes in solution, attach the battery, and disconnect the battery at the end of the experiment.

### ■ RESULTS

After 30 min of electro dialysis using a 9 V battery and subsequent addition of thymol blue pH indicator to all three chambers, which originally contained transparent and colorless solutions, the outer chambers appeared red while the center chamber appeared orange as a result of successful deionization ([Figure 5](#)). The color directly relates to the pH of the solution and therefore to the number of free  $\text{H}^+$  ( $\text{H}_3\text{O}^+$ ) ions in solution. The differences in color are more (less) pronounced if the experiment is performed for more (less) than 30 min or a smaller (larger) volume of solution is used.



**Figure 5.** Digital photographs of the ED cell after a potential of 9 V was applied across the cell for 30 min and thymol blue pH indicator was added: (top) top-down view and (bottom) front view. Initially, each chamber contained only aqueous 5 mM  $\text{H}_2\text{SO}_4$  and was completely colorless.

### ■ DISCUSSION

This activity was performed with more than 25 middle-school students, high-school students, and undergraduate students majoring in chemistry. Formal survey feedback was obtained from the high-school students. Each student performed the activity, but students worked in pairs to assist in more delicate procedural steps. About half of the high-school students obtained the intended results. Those that did not either did not make large enough holes in their Teflon tape or attached their battery backward. More than one-third of the high-school students remarked that this activity was more enjoyable and a better learning experience than “Juice from Juice”, a well-established outreach activity that the students also performed on the same day. “Juice from Juice” is a dye-sensitized solar cell activity in which blackberry dyes are used to fabricate solar cells. It is based on the activity published by Smestad and Grätzel in 1998<sup>17</sup> and more recently popularized by the Solar Center for Chemical Innovation supported by the National Science Foundation. The most common feedback received from the high-school students was to increase the clarity of the instruction, including background information, steps in the procedure, names of the components used, and conclusions based on observed outcomes. Several students also suggested that technical names for each component be listed to assist in describing the processes involved in the overall function. Adjustments were made to the curriculum in response to this feedback. In particular, special emphasis is placed on the orientation of the battery connection.



## LEARNING OBJECTIVES

The following topics are related to the activity and can be taught in conjunction with the activity or at least indicated so that the participants are able to relate the activity to core chemical concepts:

- middle-school level
  - pH scale
  - dissociation of salts and acids in water
- high-school level
  - ion-selective membranes
  - the electrochemical cell as a circuit
  - solution resistivity/conductivity
- undergraduate level
  - redox chemistry at electrodes
  - benefits of various state-of-the-art desalination technologies
  - kinetic and mass transfer overpotentials
  - electrochemical techniques (e.g., chronopotentiometry, chronoamperometry, cyclic voltammetry)

The following inquiry questions may be asked of students to promote critical thinking and to assess learning outcomes:

- What are the advantages and disadvantages of ED versus RO?
- Why is there a pH change in the anode chamber over time? Is the reasoning similar for the pH change in the cathode chamber?
- If the difference in color is subtle, experimentally how can the color change be increased in terms of the power source, time, and concentration of acid initially used?
- Explain why a color change of thymol blue is, or is not, observed with weaker acids such as vinegar (acetic acid).
- Is a color change due to thymol blue expected if the solutions contain both acid and salt? Why or why not?

## CONCLUSIONS

A hands-on scientific inquiry activity and accompanying curriculum have been developed to promote the importance of desalination to future science leaders. This activity allows for a wide distribution of subject matter to be covered, including acid strength (pH), dependence of conductivity on the type and concentration of electrolyte salt solution, pH indicators, spectrophotometry, polymeric ion-selective membranes, ionic circuits, and osmotic pressure, among other topics. The flexibility in setup of this activity, which has many cost-effective options for implementation, allows for dissemination throughout many levels of science education and with a limited budget for supplies. We envision this to be a core component in advanced chemistry courses at the middle- and high-school levels and general chemistry courses at the college level.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: [10.1021/acs.jchemed.7b00021](https://doi.org/10.1021/acs.jchemed.7b00021).

Technical background; additional experimental methods; materials used; online resources (DOCX, PDF)  
Procedure diagrams (PDF)

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### Notes

The authors declare no competing financial interest.

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