JOURNAL OF CHEMICAL EDUCATION

Activity

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Electrolysis of Water in the Secondary School Science Laboratory with Inexpensive Microfluidics

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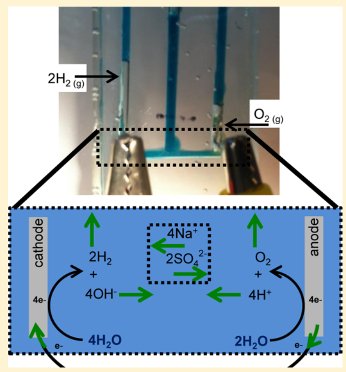
Supporting Information

ABSTRACT: This activity allows students to visualize the electrolysis of water in a microfluidic device in under 1 min. Instructional materials are provided to demonstrate how the activity meets West Virginia content standards and objectives. Electrolysis of water is a standard chemistry experiment, but the typical laboratory apparatus (e.g., Hoffman cell) is best suited for group presentations. With microfluidics, the cell volume is reduced from 100 mL to 100 μL, making the electrolysis safer and easier to view by an individual. A single device is reusable and assembled for \$5. This report describes the development of a microfluidic learning module that was implemented and assessed in the eighth-grade chemistry classroom.



KEYWORDS: *Electrolytic/Galvanic Cells/Potentials, Microscale Lab, High School/Introductory Chemistry, Elementary/Middle School Science, Stoichiometry*

■ INTRODUCTION

Electrolysis experiments are well-established demonstrations for introductory chemical education at the high school and postsecondary levels.[1−3] The electrolysis of water is useful to reinforce stoichiometry because the difference in the generated volume of hydrogen and oxygen (i.e., 2:1) is easily and quantitatively observable. The activity also supports a discussion of the conservation of energy and mass. However, a few factors must be considered when integrating this experiment in a teaching environment. Educators without access to glass blowing services must purchase Hoffman cells at an approximate cost of \$150 U.S. (see the Supporting Information). The hydrogen gas generated by the experiment is combustible. Although this is an adverse outcome, some instructors intentionally combust the generated hydrogen as further evidence to the student observers of the gas identity. Some of the different supporting electrolytes recommended are corrosive, which introduces additional safety considerations. For these reasons, electrolysis demonstrations are often limited to introductory chemistry courses at the college and, in some cases, high school level. Few educators have adapted electrolysis to earlier science education.[4−6]

The issues identified for benchtop electrolysis experiments are eliminated if the apparatus is miniaturized. Reducing the size of the electrolysis cell reduces the cost, eliminates safety considerations, and decreases the time required for an observable outcome (i.e., a 2:1 ratio of generated hydrogen vs oxygen). In fact, smaller electrolysis cells constructed from low cost materials such as plastic pipet bulbs[7] or filter paper and lithium batteries[6] have been reported. Laboratory instruction can enhance student learning when coupled with learning empowering technologies[8] and appropriate goals related to student learning outcomes. There is evidence that personalizing an experiment or demonstration reinforces learning.[9] The advantage of miniaturization through microfluidics is increasingly recognized in the teaching environment to demonstrate concepts ranging from clinical diagnostics to fluid dynamics.[10] The portable nature of these devices and reduced cost address several of the limitations of electrolysis equipment noted above. Glass,[11] poly(dimethylsiloxane) (PDMS),[12−15] poly(methyl methacrylate),[16] gelatin,[17] paper,[18] and low-cost commercially available thermoplastics[19] (i.e., shrinky dinks) have all been reported and utilized for classroom experiments. The goal of this report is to disseminate a microfluidics-based electrolysis demonstration so that the principles supported by this experiment can be implemented frequently and earlier in science education.

■ BENCH TOP ELECTROLYSIS

The experiment as completed with a traditional Hoffman Cell is archived as a video link (see the video file in the Supporting

dx.doi.org/10.1021/ed400757m | *J. Chem. Educ.* XXXX, XXX, XXX−XXX

Information (ed400757m_si_002.avi). The Hoffman apparatus (see Figure 1) is filled with 100 mL of the supporting

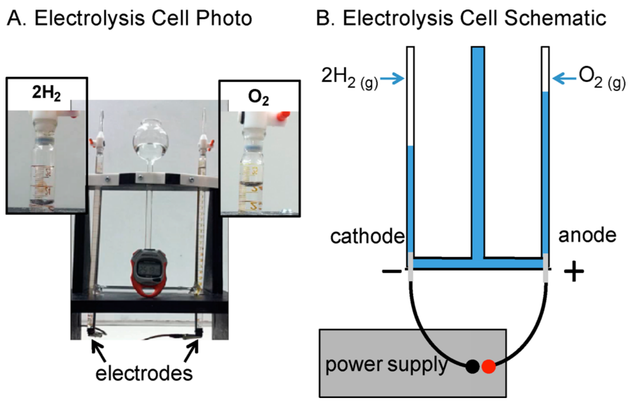


Figure 1. (A) Example of a traditional Hoffman cell used for electrolysis demonstrations. (B) Schematic of the instrument.

electrolyte. Once filled, the stopcocks are closed. A strategy has been reported to avoid errors in quantification due to the difference in gas solubility by saturating the liquid inside of the cell with hydrogen and oxygen gas.[3] Although the feasibility of different supporting electrolytes has been reported,[3] sodium sulfate is noncorrosive, inexpensive, nontoxic, and compatible with pH determinations.[20] Both the video and images shown in Figure 1 were performed with an aqueous solution of 1 M sodium sulfate as the supporting electrolyte, and a 13-V DC power supply. As shown in Figure 1, the gas collects in each glass reservoir at the top of the cell. A total gas volume of nearly 5 mL accumulated within 25 min.

■ ELECTROLYSIS IN A PDMS CHIP

The experiment has been translated into a chip using PDMS. The cured PDMS chip is reversibly sealed to a standard microscope glass slide. The protocols to construct a glass mold, to cast commercially available PDMS in the mold, and to assemble and operate the microfluidic electrolysis cell (see Figure 2A) are documented in the Supporting Information. Platinum wire electrodes or commercially available platinum plated needles are used to perform electrolysis (see the Supporting Information for proposed parts). However, the polymer chip can be fitted with any electrode with an outer diameter no larger than 400 μm that is inert and conductive (e.g., platinum, stainless steel, graphite). The 1 M sodium sulfate supporting electrolyte is introduced into the central channel with a plastic transfer pipet. Blue food coloring is added to the supporting electrolyte to improve visualization of the accumulating gas. The reaction is driven with power supplied by a disposable or rechargeable 9-V battery. Unlike the glass Hoffman Cell, which can be opened or closed with the stopcocks, the anodic and cathodic arms in the PDMS chip remain open. As shown in Figure 2B in the PDMS microfluidic device the gas collects in each reservoir at the electrode. This is due to the increased surface tension in the smaller diameter reservoir channels. In less than 1 min, approximately 1 cm of hydrogen and 0.5 cm of oxygen have accumulated. The reaction proceeds as long as liquid connects both electrodes, allowing current to pass through the system (see the video file in the Supporting Information (ed400757m_si_003.avi)).

■ ELECTROLYSIS IN A GELATIN CHIP

The microfluidic electrolysis was repeated using chips fabricated from gelatin as shown in Figure 2C. Although cured PDMS is nontoxic and commercially available for purchase, it has been noted that gelatin solutions are easier to acquire and can be used to fabricate microfluidic devices.[17] The gelatin preparation protocol (available as Supporting Information) is modified slightly from a previously reported Jell-O/Knox gelatin mix[17] to improve the mechanical stability and optical transparency of the gelatin microfluidic device. Once the gelatin is prepared, it is poured into the glass molds utilized to cast PDMS chips. After the gelatin has set, the electrodes are positioned in the device and the supporting electrolyte (1 M aqueous sodium sulfate combined with blue food coloring) is introduced into the central channel with a plastic transfer pipet. Again, the reaction is driven with power supplied from a disposable or rechargeable 9-V battery. The reaction proceeds in a manner similar to that of the PDMS electrolysis chip (see the video file in the Supporting Information (ed400757m_si_004.avi)).

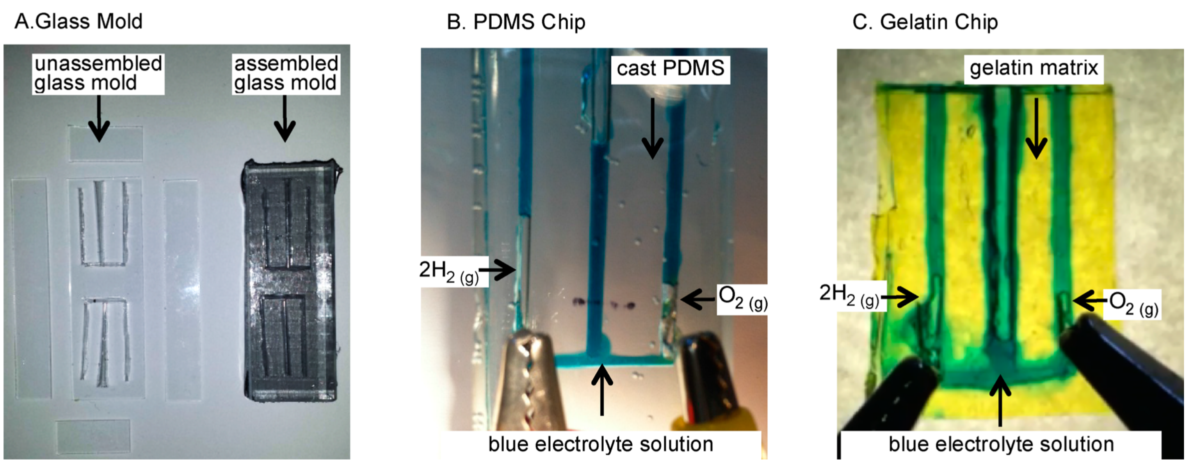


Figure 2. Depicts images relevant to electrolysis in microfluidic cells. The photograph in (A) shows the assembly of a glass mold used to cast the microfluidic chips. The mold is fabricated from common laboratory slides held together with duct tape. The photographs in (B) and (C) are of electrolysis in a microfluidic electrolysis cell constructed of PDMS and gelatin, respectively. For both electrolysis images, hydrogen and oxygen are generated in the left and right channels, respectively.

UTILIZING ELECTROLYSIS CHIPS IN THE CLASSROOM

The different characteristics of PDMS and gelatin microfluidics determine which material is employed in a specific classroom. Although a reusable PDMS chip can be fabricated after a 2-h cure period at 120 °C, the toxicity of uncured PDMS excludes students from participating in the fabrication process. For gelatin chips, the students may cast their own microfluidic device; however, the molds must be cured at ∼4 °C for 24 h. This requires the educator to have access to a refrigerator. The students may assemble the glass molds and cast the gelatin in a single class period and then assemble and test the device during the next class period. After the gelatin has set, the device may be used at room temperature for a limited time (∼2 h) during which the material remains gel-like and viable for use. Gelatin chips are more fragile than PDMS chips and are typically used only once. If gelatin microfluidics are to be used, it may be prudent for the educator to instruct students to cast 2−3 gelatin chips and to have some PDMS chips available for students who do not successfully cast gelatin chips.

TEACHING ELECTROLYSIS WITH CHIPS

The experiments were implemented in six sections of eighth grade basic science at Wildwood Middle School. Learning outcomes that meet West Virginia Department of Education content standards and objectives were documented (see Supporting Information). Students participated in synchronous video mentoring using FaceTime in order to view benchtop electrolysis prior to the lecture. The purpose of this synchronous visualization is to stimulate students to conceptualize the content prior to lecture with a technology tool used by adolescents outside of the classroom. Videos of benchtop electrolysis, such as the one available in the Supporting Information, are a suitable replacement in the absence of an available video mentor. Prior to conducting experiments, students received a lecture on fundamental concepts in chemistry including chemical reactions, stoichiometry, and conservation of energy and mass. An example of a lecture handout is included in the Supporting Information. Students were given the opportunity to passively observe electrolysis using a benchtop Hoffman cell. In one classroom, students were tasked with creating and narrating a video of the benchtop electrolysis experiment. All six science classes were allowed to independently conduct electrolysis experiments with chips. Students who accurately and effectively demonstrated a firm grasp of the experiment were given an opportunity to conduct a chip-based electrolysis experiment outside of the classroom if they agreed to complete a take home survey that required documentation of the effectiveness of the explanation of the chemistry concepts to others. These students were given the necessary equipment and shown how to use common Epsom salt (i.e., magnesium sulfate) as a substitute for sodium sulfate. Six different teachers integrated the kits into their middle school class rooms and offered students the opportunity to take kits home and complete the assessment. Three middle school teachers who used the kits completed surveys. Answers to questions ranked 1 (strongly disagree) to 5 (strongly agree) indicated confidence in teaching electrolysis, in student understanding of the material, and that the lesson is appropriate for the academic level of the student as 4.3 ± 0.6, 4.7 ± 0.6, 4.7 ± 0.6, respectively. A total of 175 surveys were returned by students. Answers to quantitative questions ranked student achievement from 0 (poor) to 5 (excellent), and responses indicated student proficiency. Scores for questions assessing the student's explanation of the process of electrolysis and of the student's interest in the unit were 4.5 ± 0.7 and 4.4 ± 0.9, respectively. Of the 172 survey respondents who offered comments, 69% communicated an understanding of electrolysis based on the experiment. In addition, 44% of respondents conveyed a positive response to the science exercise, whereas 6% expressed a negative response. Both the video choreography exercise and the take home microfluidic experiment are designed to enhance learning through teaching. There are many educational benefits to learners who function as teachers,[21,22] including increased knowledge retention and improved critical thinking.[23,24] In addition, science outreach to neighbors, siblings, parents, or friends increases the science literacy of the public.

BEYOND THE ELECTROLYSIS TEACHING EXERCISE

Through miniaturization (i.e., decreasing the volume from 100 mL to 100 μL) the experimental observation time is decreased and safety issues are eliminated. These changes render microfluidic electrolysis feasible in the secondary school classroom and personalize the experiment. To ensure that educators may perform the experiments independent of support, we have striven to keep the costs low and utilized materials that are commercially available. As outlined in the Supporting Information, approximately 30 individual microfluidic electrolysis cells can be fabricated and used repeatedly for the price of a single Hoffman electrolysis cell. A single batch of PDMS chips may be stored for extended periods (years). The electrolysis experiment may be performed during a single class period. Once students are familiar with microfluidic electrolysis, students may apply the device to additional experiments that are outlined in the teacher handout in the Supporting Information. These extended experiments involve changes to the supporting electrolyte designed to teach students the process of hypothesis testing and expand the course content to chemical concentration, additional chemical reactions, and reaction rate. The exercise is easily adapted to include experiments documented by others including colored indicators to demonstrate the change in pH associated with electrolysis.[20,25] As soon as students gain skill in fabricating gelatin chips, they may proceed to other published gelatin chip experiments.[17] Many barriers to incorporating electrolysis in the secondary school classroom are removed. This report provides teachers with lecture materials (handouts, videos, etc.), documented content standards and objectives, and protocols for device fabrication based on materials available to educators (PDMS, sodium sulfate) and students (gelatin, Epsom salt). The experiment was further disseminated to teachers within the state of West Virginia through a hands-on workshop at the annual West Virginia Science Teachers Association Conference. The development of accessible instrumentation supported by lecture content, documented instructions, and training provides educators new tools to enhance early science education. The electrolysis experiments outlined in this report were deployed in the middle school classroom but may also be adapted to high school and college level chemistry instruction.

■ ASSOCIATED CONTENT

Supporting Information

Extensive instructional materials about implementing the experiments are included. Ancillary data includes sample lecture materials, student handouts, documented content standards and objectives, and protocol to assemble glass molds and cast PDMS as well as gelatin chips. Videos of electrolysis in a traditional Hoffman cell, in a PDMS microfluidic chip, and in a gelatin microfluidic chip are provided.This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

This material is based upon work supported by the National Science Foundation under Grant No. CHE1212537. We acknowledge support for T.A.D. and C.L.C. from the National Science Foundation (Cooperative Agreement 1003907). S.L.A. acknowledges funding through the Teacher Research Experience for the Advancement of Knowledge (TREK) supported by an NSF Grant No. EPS-1003907. J.K.D. acknowledges the WVU-REU program supported by the National Science Foundation Divisions of Materials Research and Chemistry (DMR-1004431) with recreational activities funded by WVU Research Corporation and the WVU Eberly College of Arts and Sciences. The authors acknowledge and express sincere appreciation to Francine DeRhonda and Jennifer Moss (Wildwood Middle School) for suggestions and feedback about the unit lesson plan, as well as Harry Finklea (WVU, C. Eugene Bennett Department of Chemistry) for comments and advice about electrolysis and electrochemistry.

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