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Activity

A Hands-On Approach to Understanding Electrochemistry for Middle and High School Students

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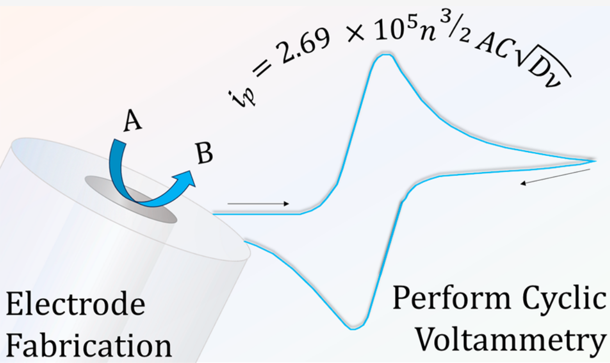
ABSTRACT: Electrochemistry plays a crucial role in our everyday lives. It allows us to power our phones and cars and allows for the real-time monitoring of blood glucose levels. While electrochemistry is of vital importance and is a long-established field of chemistry, it is often neglected in modern curricula for STEM courses. Herein, we show that hands-on electrochemical experimentation can be approachable and attainable for students as young as sixth grade and can help facilitate learning of redox chemistry. The proposed experiments allow students to better understand electron transfer and general redox chemistry as well as gain invaluable laboratory experience, which can also contextualize what electrochemistry in a research lab may look like. We propose a simple method for the fabrication of platinum electrodes, instruction on how to introduce electrochemistry and cyclic voltammetry and how to probe the parameters that affect an electrochemical measurement. Using this hands-on teaching approach, middle school aged students will be able to put together research-quality posters and demonstrate their understanding of the presented topics.



KEYWORDS: *Middle School Science, High School/Introductory Chemistry, First-Year Undergraduate/General, Analytical Chemistry, Demonstrations, Laboratory Instruction, Outreach, Hands-On Learning, Electrochemistry, Oxidation/Reduction*

■ INTRODUCTION

In the history of chemistry, electrochemical experiments surfaced early. At the turn of the 19th century, scientists became increasingly interested in electrochemical phenomena and began devising electrochemical experiments which also helped develop chemical theories. Volta's pile[1] and the electrolysis of water[2] in 1800, Humphry Davy's isolation of several new elements, including sodium, potassium, and chlorine,[3] and Michael Faraday's law relating the degree of electrolysis to the amount of electricity passed through solution[4] all demonstrate the early fascination and utility of electrochemical experiments. It may be surprising to realize that all of the aforementioned observations were made without the experimenters knowing that salts dissolved into charged ions (from Svante Arrhenius' work in the 1880s)[5] or that charged subatomic particles existed (from J. J. Thomson's cathode ray tube experiment in 1897).[6] If electrochemical experiments could be performed and understood such that new truths of nature could be learned without any of our modern chemical intuitions, then thoughtfully crafted lessons using electrochemistry could be perfect learning experiences for students who often lack background knowledge, as they are currently being introduced to the field of chemistry.

Despite electrochemistry's early and pervasive participation in chemistry's growth, the subdiscipline is often taught sparingly—if at all—in introductory high school and undergraduate general chemistry courses. In fact, concerted teaching of electrochemistry is usually reserved until a few lectures in an analytical chemistry course or an elective upper-level undergraduate or graduate level course. While reduction and oxidation reactions are introduced in the American education system as early as introductory high school chemistry classes, the College Board and the ACS have written several enduring understanding, learning objective, and essential knowledge statements concerning electrochemistry for the AP Chemistry exam[7] and General Chemistry Content Map,[8] respectively.

We argue here that hands-on electrochemical experiments provide an accessible and simple avenue for students to not only address these learning objectives and others but also gain experience performing research. Excellent introductory texts to electrochemistry and cyclic voltammetry exist in the literature,[9,10] including the most read article published in this *Journal* by Dempsey et al.[11] We have previously shown in this

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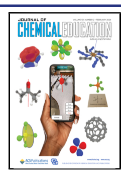

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Journal that upper-level undergraduate and graduate students could design and manufacture simple potentiostats that cost about $55 and can be used to perform electroanalytical techniques.[12] Additionally, we have also provided simple experiments for high schoolers or undergraduates to examine the role of dissolved oxygen in the voltaic pile, requiring only pennies, salt, cardboard, a jar, a candle, and a multimeter.[13] A number of other electrochemical experiments have also been proposed in this *Journal* and elsewhere.[14−16] Electrochemical experiments can be made so accessible that in this article we detail how middle school students could fabricate their own electrodes, collect their own cyclic voltammograms, and investigate additional physics and chemistry concepts. Students should be able to examine:

- How measured peak current can be used to quantify the concentration of redox molecules or how fast molecules can move in a solution.
- How manipulation of measurement parameters can affect the observed peak current.
- How blocking an electrode surface with different materials can give insight into the conductivity and electrocatalytic activity of that material.

We acknowledge that electrochemistry is not traditionally taught until students are in high school or undergraduate classrooms, and when it is taught in these contexts, pedagogical methods are frequently limited to direct instruction. The hands-on experiments presented here are geared toward middle school students and are intended to be supplemented by a combination of direct instruction of simplified electrochemical concepts, as well as guided hypothesis formation, data plotting and interpretation, and poster preparation. Since electrochemistry is vitally important to current issues including improving stability and performance of batteries, mitigating corrosion, converting greenhouse gases into fuels, and generating alternative, cleaner fuels (like hydrogen gas) to tackle environmental pollution, we feel these experiments can serve as an important introduction to the field. Electrochemistry pervades the lives of our students, and thus, it is advantageous to engage students in performing and understanding simple electrochemical measurements. The experiments we detail in this article, although proposed for middle school students, could easily be modified to reach more advanced learning objectives suitable for even graduate students.

■ HAZARDS

Proper protective eyewear and gloves should be worn when performing all experiments. Extra precautions should be taken when sealing the electrodes in the propane torch. Long hair should be tied back, and experimenters should be educated on how to hold the capillary in the torch to minimize the burn risk. All chemicals used herein have no associated hazards. However, it is imperative to discuss the dangers of contacting and ingesting any chemicals, especially with middle schoolers, who may have very limited experience with safe lab procedures.

■ MATERIALS AND METHODS

Fabrication of Platinum Electrodes

Borosilicate capillaries (Sutter Instruments) [O.D. 1.5 mm, I.D. 0.86 mm, 10 cm length] were threaded with approximately 3 cm of platinum wire (Alfa Aesar) [various diameters; herein we used d = 25 or 250 μm]. The platinum wire was sealed within the borosilicate capillary by exposing a majority of the portion of the capillary threaded with platinum wire to the flame of a propane torch (Benzomatic) for approximately 30 s. Students should be instructed to rotate the capillary slowly during the sealing process. A portion of the platinum wire was left unsealed in the capillary to make electrical connection. After the platinum wire was sealed within the capillary, the capillary was sanded down such that the electrode has an inlaid disk geometry (or the electrode was a flat sealed disk). Electrical wire (Pulsivo [purchased through Amazon]), which was cut to be slightly longer than the sealed capillary, was stripped and threaded into the capillary. The connection between the electrical and platinum wires was checked visually. The wire was secured in place with hot glue. Figure S1 shows images of each of the described steps. Supporting Information Video S1 also shows the process of Pt electrode fabrication. After the electrode was fabricated, the diameter of the platinum wire was determined with a digital microscope (AmScope) [50× to 500× USB Digital Hand-held Microscope].

Performing Cyclic Voltammetry for Determination of Electrode Area and Investigation of Scan Rate Dependence

All voltammetry was performed using a CH Instruments (CHI) potentiostat (Model 601D, 6012D, or 6284E). A solution of 10 mM potassium ferricyanide (Sigma-Aldrich) and 1 M KCl (Thermo Fisher) was prepared in ultrapure water (18.2 MΩ cm). Cyclic voltammetry was performed using a three-electrode cell with the fabricated platinum electrode as the working electrode (or in the case of scan rate experiments, a platinum macroelectrode (CHI) [d = 2 mm]), an Ag/AgCl reference electrode (CHI), and a graphite rod counter (Thermo Fisher) electrode. Voltammetry was taken from 0.5 to 0 V. For determination of the electrode surface area, a scan rate of 0.05 V/s was used. For determination of the effect of the scan rate on peak current, scan rates of 0.005, 0.01, 0.1, 0.5, 1, and 5 V/s were used.

Blocking Experiments

Voltammetry was taken in 10 mM ferricyanide and 1 M KCl as described above. Amperometry was then ran at 0.5 V for 600 s with a sample interval of 0.0167 s in approximately 20 mL of solution containing 1 mM ferrocenemethanol (Sigma-Aldrich) and 1 M KCl (Thermo Fisher) in ultrapure water (18.2 MΩ cm). 200 μL sample containing 5 mg/mL silicon dioxide (Sigma-Aldrich) [~99%, 0.5−10 μm] in ultrapure water (18.2 MΩ cm) was spiked into the ferrocenemethanol solution, and amperometry was run again using the same parameters mentioned previously. Afterward, the electrodes were moved back to the 10 mM ferricyanide solution, and cyclic voltammetry was taken again.

■ RESULTS AND DISCUSSION

The first step in any electrochemical experiment is to choose the electrodes that you will be using to perform your measurements. A natural first step in a lesson in electrochemistry is then the fabrication of electrodes. Herein, we present a simple method for the fabrication of platinum electrodes using a commercially available blow torch, glass capillaries, platinum wire, and commercially available electrical wire. Figure 1 shows the fabrication of these electrodes (Figure 1A), an image of a fabricated electrode (Figure 1B), and a digital microscope image of the fabricated electrode with the

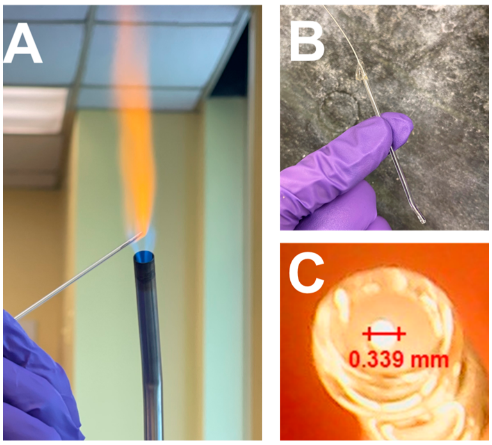

Figure 1. Platinum electrode fabrication. (A) Image of the sealing of a platinum wire into a borosilicate capillary with a commercially available propane torch. (B) Image of a fabricated platinum electrode. (C) Digital microscope image of a fabricated electrode with measured diameter.

measured electrode diameter (Figure 1C). A more detailed guide for the fabrication of these electrodes can be found in Figure S1 and a video guide can be found in Supporting Information Video S1. If the cost of purchasing pure platinum wire is prohibitive, other cheaper metal wires may be substituted, such as copper or other alloyed metals, but this may come at the cost of electrochemical performance and durability of the electrodes (i.e., potential windows will change on different materials, and less inert electrodes may oxidize and/or become passivated).[11,17] Also, the melting point of other metals must be considered when substitutions are made, and the heat used for the sealing of the borosilicate capillary should be adjusted accordingly.

Once the platinum electrode is prepared, it can be used as a working electrode in an electrochemical experiment. As shown in Figure 2, a typical three-electrode cell configuration (Figure 2A) used to perform cyclic voltammetry consists of a working electrode (which is where the fabricated electrode comes in), a reference electrode, and a counter electrode. Here we show a commercially available electrode as the working electrode (as opposed to the fabricated electrodes used by the students) as this is typical of electrochemical experiments performed in research laboratories. An example of voltammetry taken with this three-electrode cell can be seen in Figure 2B. Reference electrodes can also be fabricated for use in such a cell[18] and a discussion on reference electrodes can be added to expand the lesson for high school and undergraduate students.

While all experiments presented within this article were performed on a CHI potentiostat with the described three-electrode cell, it would be possible to perform all experiments presented with a two-electrode cell (a working electrode and a quasi-reference counter electrode on a simpler potentiostat. Our group has previously reported a simpler potentiostat, the SweepStat, which can be assembled for approximately \$55 and can run all electrochemical techniques reported within this manuscript.[12] For the two-electrode cell, the fabricated electrode can be used as a working electrode, and a simple piece of pencil lead can be used as a quasi-reference counter electrode, effectively reducing the cost of the experiments presented. However, it should be noted that when a two-electrode cell is used, the observed half-wave potential will be set by the species in solution, and so this set up may not be suitable for experiments where half-wave potential is determined.

After the voltammograms were obtained, the concepts of oxidation and reduction can be introduced (or reintroduced in the case of older students). When approaching the voltammogram, students can be instructed to think of the current as the rate of electron transfer across the working electrode and the potential as an applied energy to facilitate that electron transfer. Once these concepts are introduced, peaks for oxidation and reduction can be assigned based on the

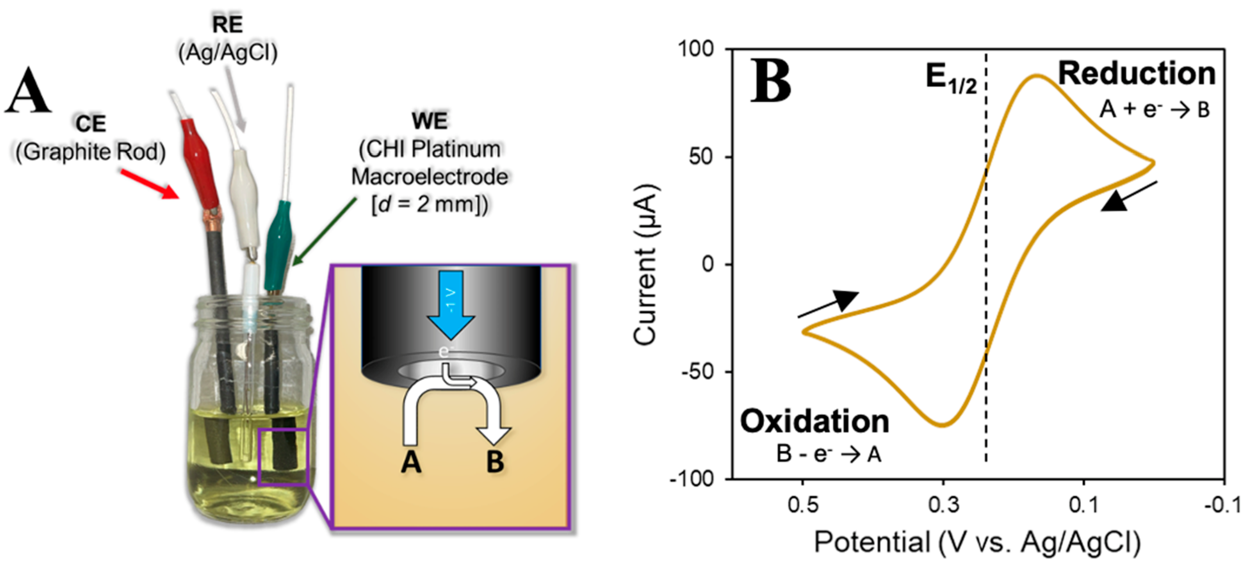

Figure 2. (A) Image of a typical three-electrode cell used for voltammetric measurements with an overlaid schematic of reduction occurring at the electrode interface (e.g., $A + e^- \rightarrow B$). For the experiments presented, a fabricated electrode was used in place of a CH Instruments (CHI) macroelectrode. (B) Voltammetry of 10 mM ferricyanide in 1 M KCl from 0.5 to 0 V taken with a fabricated electrode as the working electrode, a CHI Ag/AgCl reference electrode, and a graphite rod counter electrode at a scan rate of 0.05 V/s. Voltammetry is overlaid with the information that can be derived from the voltammogram (i.e., where reduction and oxidation occur and the half-wave potential [$E_{1/2}$]).

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convention used. It is important to note that the sign of the current will depend on the convention that the potentiostat software uses to plot the voltammogram. Herein, we present all voltammetry in the polarographic or U.S. convention, where cathodic [reductive] current is positive and anodic [oxidative] current is negative.[11] A simple explanation for the behavior of the voltammogram can be given as follows: as we scan toward more negative potentials, we increase the energy level of the electrons within the electrode so they can transfer to the mediator (or the chemical we added). This causes the mediator to be reduced, which is shown as a peak with a positive current because electrons leave the electrode. As we scan toward more positive potentials, the energy level of the electrons within the electrode decreases, and the electrode will want to accept electrons from the mediator. This leads to the oxidation of the mediator in solution, which is shown as a peak with a negative current because electrons go into the electrode. Even when the experiment originally does not have any reduced molecules in solution (as is the case in Figure 2, when only ferricyanide is present initially), negative (oxidative) current can still be seen because one is able to oxidize any molecules that were reduced previously in the experiment (e.g., during the negative sweep). The reason the voltammogram is "duck shaped" is because it takes time for molecules in solution to diffuse (or move) to and from the electrode. As a note: this explanation assumes a basic understanding of the atomic structure (i.e., students know what electrons are). If this lesson is to be given to younger students, then a brief introduction to atomic structure may be needed. If this lesson is to be adapted for older students (high school or undergraduate level), then the explanation of the shape of these voltammograms can become more complex. For example, students could be introduced to the concept of a diffusion layer and the different types of diffusion associated with different electrodes (linear diffusion for macroelectrodes and radial diffusion for ultra-microelectrodes). In general, students should be able to identify oxidation and reduction peaks and explain that an electron is either being accepted by or donated from the mediator in solution. Furthermore, students can be asked to calculate the half-wave potential from the potentials of the reduction and oxidation peaks using

$$E_{1/2} = \frac{E_{ox} + E_{red}}{2}$$

where $E_{1/2}$ is the half-wave potential, E_{ox} is the potential of the oxidation peak, and E_{red} is the potential of the reduction peak. One possible pedagogical extension for high school or undergraduate students could be to compare the reported standard reduction potentials to the experimental $E_{1/2}$ values measured in class. This discussion would necessitate detailing the conversion factor between different reference electrodes (e.g., between the experimental Ag|AgCl reference electrode used herein and the standard hydrogen electrode, against which E^0 values are commonly reported), discussion of the Nernst equation, and other concepts[9,11] depending on grade level. Middle-school students can still be asked to deduce what happens when we apply a potential greater (more positive) than the half-wave potential and what happens when we apply a potential that is lower (more negative) than the half-wave potential. At this point, students should understand how applying a potential can cause oxidation or reduction.

After students understand the basics of voltammetry and applied potential, they can then be introduced to the Randles–Sevcik equation, as shown in Figure 3A. With this equation introduced, the variables that directly affect peak current

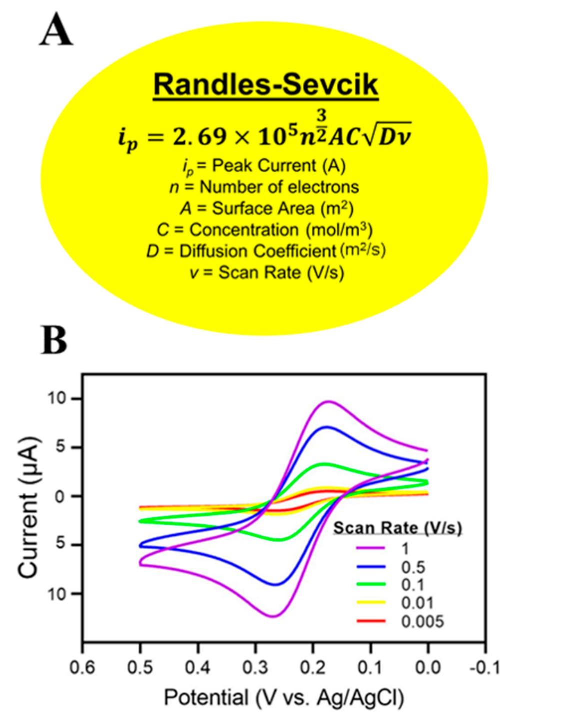

Figure 3. (A) The Randles–Sevcik equation with the defined variables and units. (B) Voltammetry of 10 mM ferricyanide in 1 M KCl taken at various scan rates.

magnitude become more obvious. First, students can use this equation to calculate the surface area of their fabricated electrode. A sample calculation of the electrode surface area from cathodic peak current can be seen in the Supporting Information (Section S2). This is a great starting point because students can compare the calculated value to the known values (which was determined via a video microscope in Figure 1C). Students can hypothesize why the calculated value may be different from the measured value. Factors such as a dirty electrode (partially blocked surface area) and error in the assumed values (e.g., error in the prepared solution concentration) can lead to inaccurate values. After understanding the factors which may affect the accuracy of values calculated from the Randles–Sevcik equation, students can be asked to directly probe these variables.

To probe the effect of the scan rate, students performed cyclic voltammetry measurements at various scan rates and overlaid the resulting voltammograms. An example of this data can be seen in Figure 3B. It can be seen from these data that with increasing scan rate, the peak current magnitude increases. This rather simple experiment, which just requires students to change one value in the potentiostat software, demonstrates the validity of the Randles–Sevcik equation. To take this one step further, students may be asked to plot the peak current versus the scan rate and versus the square root of scan rate as seen in Figure S2. With this plot, it becomes clear that the peak current scales linearly with the square root of the scan rate (and not just the scan rate) as predicted by the Randles–Sevcik equation. In addition, students can use this plot to calculate the diffusion coefficient of the chosen

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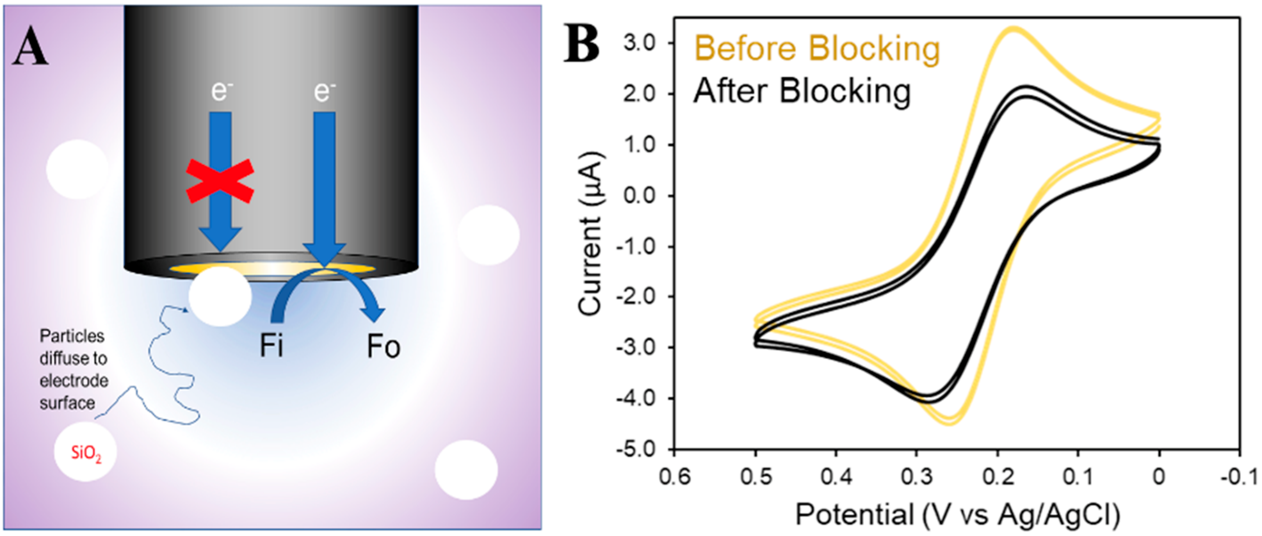

Figure 4. (A) Schematic of the blocking experiment. Silica nanoparticles in solution diffuse to the electrode surface and prevent electron transfer, effectively decreasing the active surface area. (B) Voltammetry of 10 mM ferricyanide in 1 M KCl taken before (gold trace) and after (black trace) blocking the electrode surface with silica nanoparticles. Voltammetry was performed with a fabricated electrode as the working electrode, a CHI Ag/AgCl reference electrode, and a graphite rod counter electrode at a scan rate of 0.05 V/s.

mediator as shown in the Supporting Information (Section S4).

Blocking experiments can be used to show how current changes with a changing surface area. A schematic showing a blocking experiment can be seen in Figure 4A. Briefly, insulating particles (in this case silica nanoparticles) can be added to solution and allowed to diffuse to the electrode surface. After these particles adsorb to the electrode surface, the electroactive area is reduced, because electron transfer cannot occur across an insulating particle. Following direct instruction, the students were able to describe that during this experiment, the nanoparticles diffused, or moved randomly, to the electrode surface and blocked electron transfer upon contact.

To perform this experiment, students can take a CV on a clean electrode in some mediator solution (in this case, 10 mM ferricyanide in 1 M KCl was used). They can be asked to calculate the area from the peak current, as described above. Students can then run amperometry at a suitable potential (for this experiment, they could choose the applied potential based on the calculated half-wave potential) before and after adding the insulating particles. An example of amperometry taken before and after the addition of insulating particles to solution can be seen in Figure S3. From these traces, it becomes apparent that the current decreases to a lower value after addition of the insulating particles. Students can then take a CV in the original solution and see how the peak current has changed. An example of CVs taken before and after the blocking experiment can be seen in Figure 4B. Students can easily perform this experiment without the associated amperometry, but if an ultramicroelectrode is used, it can become possible to resolve single blocking events.[19] After students obtain the CVs, they can be asked to calculate the difference in area before and after the blocking experiment. While the experiments performed here use nanoparticles ranging from 0.5 to 1 μm in diameter, which are too small to be resolved with microscopy (especially with the digital microscope used here), larger particles may be used for this experiment and the blocking of the electrode surface could be observed optically.

In addition to the presented experiments, students could also (or alternatively) be asked to investigate the effect of mediator concentration (which can easily be tested by either diluting or concentrating the tested solution by adding water or more mediator) or diffusion coefficient (by heating or cooling the tested solution as the diffusion coefficient is temperature dependent or by testing voltammetry in solutions of various viscosities [ethanol, water, glycerol, etc.]).

To conclude this activity, students could be asked to create a research poster as this provides an excellent opportunity to consolidate their learning and practice scientific communication. A sample poster can be found in Figure S4. Additionally, if time does not allow, an exit ticket is provided in Figure S5 to gauge their understanding. Future pedagogical strategies should include greater emphasis on applications, which can include corrosion, electrochemical (bio)sensing, batteries, and other energy conversion technologies, that may be more familiar to middle-school students. In total, it is expected that students should spent approximately 5 class hours to obtain all the described data (including lecture/instruction time) and 5 class hours assembling research posters. Since middle-school students are expected to start with no lab experience, the instruction time could likely be shortened with older students. Also, all solutions used herein were prepared by the instructor for the sake of time. To add more valuable lab experience, students could be asked to prepare solutions themselves (and potentially even test the accuracy of these solutions by calculating the concentration using Randles–Sevcik!).

We have supported classroom use for all of the procedures reported in this paper, in middle and high schools, as a way to ensure that implementation can be effective. In our experience, students are able to grasp rather complex concepts of electrochemistry such as those presented here. Further work to collect data on student experiences will be reported subsequently. In a world powered by electrochemistry, the presented experiments could provide an engaging and memorable introduction to this exciting and important field.

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■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.3c00939.

Electrode Fabrication Video (MP4)

Electrode fabrication guide, sample calculation of electrode area, understanding scan rate dependence, sample calculation of the diffusion coefficient of ferricyanide, amperometry before and after blocking, sample poster given to students, and electrochemistry exit ticket (PDF)

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Author Contributions

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Notes

The authors declare no competing financial interest.

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