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Laboratory Experiment

Teaching Heterogeneous Electrocatalytic Water Oxidation with Nickel- and Cobalt-Based Catalysts Using Cyclic Voltammetry and Python Simulation

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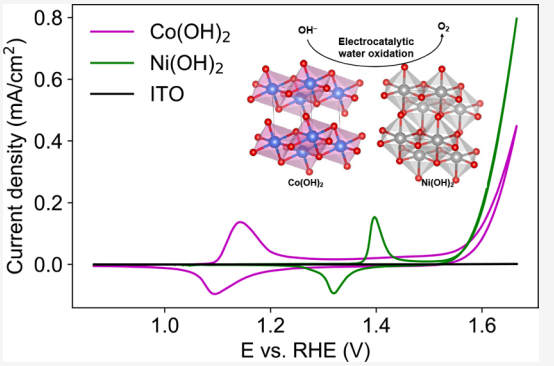
ABSTRACT: An integrated inorganic chemistry laboratory experience focusing on heterogeneous electrocatalysis with nickel (Ni)- and cobalt (Co)-based electrocatalysts is designed for upper-division, major-level chemistry students. In this laboratory, students will be guided through the fabrication of an indium tin oxide (ITO)-coated glass working electrode, electrodeposition of the nickel hydroxide ($Ni(OH)_2$) and cobalt hydroxide ($Co(OH)_2$) electrocatalysts on the ITO working electrodes, and the electrochemical characterization of the electrocatalysts. Students will investigate the electrocatalytic oxygen evolution reaction (OER) with electrocatalysts $Ni(OH)_2$ and $Co(OH)_2$ in an alkaline electrolyte. Cyclic voltammetry (CV) is the major experimental technique students will apply to measure the performances of different electrocatalysts on the OER activities. Additionally, a Python-based simulation of the cyclic voltammograms will be applied to help students gain more insights about the interpretation of the cyclic voltammograms of reversible one-electron redox reactions.



KEYWORDS: *Upper-Division Undergraduate, Inorganic Chemistry, Electrodeposition, Electrocatalysis, Water Electrolysis, Cyclic Voltammetry, Python Simulation*

INTRODUCTION

Undergraduate students are motivated and engaged to persist in STEM by learning skills to combat climate change and reduce carbon dioxide (CO_2) generation. To replace fossil fuels with sustainable, environmentally friendly, and affordable energy sources, hydrogen (H_2) is considered as the most promising energy carrier.[1] The mass production of high-purity H_2 could be achieved with water electrolysis using renewable electricity produced by solar cells and other energies such as wind power with minimal carbon footprint.[1] Water electrolysis is a leading H_2 production pathway to achieve the goal of reducing the cost of clean H_2 by 80% to $1 per 1 kg in 1 decade.[2] From an educational point of view, water electrolysis is also a classical model system in the undergraduate electrochemistry education.[3−8]

The topics related to electrochemistry are often introduced in the undergraduate chemistry curriculum in different contexts. Although different concepts of electrochemistry are taught in courses such as general chemistry, inorganic chemistry, analytical chemistry, and physical chemistry, experimental techniques of electrochemistry are not often applied in laboratory courses.[6] Since it is challenging to provide students a comprehensive understanding of electrochemistry solely with lectures, more and more laboratory experiments and simulation tools have been developed in the past few years to promote hands-on experience in electrochemistry education.[6,8−19] Water electrolysis has recently been incorporated into the teaching laboratory curriculum as an introduction module to electrochemistry.[6] Water electrolysis also serves as a model system for comprehending its side reactions,[8] its implementation via photoelectrochemical (PEC) process[11] as well as the spectroelectrochemistry technique.[9] Several activities were also developed to teach the important fundamental concepts in electrochemistry such as electrode kinetics and mass transfer to undergraduate students.[20−22] To help students gain hands-on experience and fundamental knowledge of electrocatalysis for energy storage, we apply a "three-pronged approach"[23] to teaching electrochemistry by integrating mini lectures with an integrated laboratory, both experimental and simulation, to study heterogeneous electrocatalysis of the anodic water oxidation with electrodeposited catalysts for upper-division undergraduate students.

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Here we report a teaching laboratory experience to help students understand the oxygen evolution reaction (OER) using Ni- and Co-based electrocatalysts with cyclic voltammetry (CV) and Python-based simulations. Students were equipped with foundational knowledge of water electrolysis and the OER from pre-lab reading and a mini lecture and subsequently guided through the fabrication of working electrodes and the electrodeposition of metal hydroxide electrocatalysts. They were engaged in conducting the CV measurements, simulations, and data interpretation of their electrocatalysts. To enhance students' understanding of water electrolysis and the OER, each student delivered a 10 min presentation on a recent research article centered on water electrolysis or the OER at the end of the semester. As one of the most popular electrochemical techniques,[24] CV is suitable for undergraduate students to study the solid-phase Ni- and Co-based electrocatalysts. To understand the somewhat abstract microscopic processes in the electrocatalysis, simulations represent an effective and safe tool to support multidimensional learning.[25,26] Python-based simulations of cyclic voltammograms are used to help students interpret their experimental results. Python is chosen because it is an open and free coding source which can run on a web-browser. The combination of mini lectures, experimental work, and simulation practice is anticipated to foster a good understanding of essential concepts in electrochemistry for the students.

Course materials, including the instructional notes, detailed experimental procedures, pre- and post-lab questions, the organization of student presentations, a list of necessary equipment, chemicals and materials, and Python codes for CV simulations, are provided in the Supporting Information (SI).

STUDENT LEARNING OUTCOMES

This laboratory experience highlights the use of mini lectures, experimental CV measurements, and simulation of cyclic voltammograms using Python for studying the heterogeneous electrocatalytic OER process. These activities are designed to facilitate the achievement of the following student learning outcomes:

1. Students will describe the background knowledge of water electrolysis in alkaline electrolytes, the anodic and cathodic reactions, and understand *the role of electrocatalysts*.
2. Students will fabricate working electrodes, perform electrodeposition, and conduct CV measurements to *gain hands-on experience*.
3. Students will correlate the current signals in cyclic voltammograms with the redox transformation of the electrocatalysts and the OER to understand the concepts of *chemical reversibility*.
4. Students will analyze the separation of anodic and cathodic peak potentials of the electrocatalysts in the cyclic voltammograms collected experimentally and relate the peak separations to *electrochemical reversibility*.
5. Students will use simulations to understand the *kinetics of heterogeneous charge transfer* by varying the values of standard rate constant (k^0) of charge transfer and correlate the changes of k^0 with the changes in peak separations in the cyclic voltammograms.
6. Students will explore the impact of scan rate (v) on the current signals in the cyclic voltammograms with both experimental and simulation data, and be able to describe the impact of *mass transfer* on the rates of electrochemical reactions.

EDUCATIONAL CONTEXT

The experimental module described here has been introduced to CHEM 426, an undergraduate advanced inorganic chemistry laboratory course at San Francisco State University. Up to 12 senior-level chemistry major undergraduate students are enrolled in CHEM 426 each spring semester. The students meet for two 3 h long laboratory sessions each week. The experimental components of the electrocatalytic OER were implemented in the semesters of springs 2022 and 2023, while the CV simulations were conducted only in spring 2023. In CHEM 426, students usually collaborate in small groups of 3−4 members for laboratory work. They engage in hands-on experimentation collectively and analyze the data independently when preparing individual lab reports. Furthermore, the experimental module was incorporated as a training component in electrocatalysis research for 11 undergraduate research students from different majors such as chemistry, biochemistry, and biology from 2019 to 2023. This took place in the course CHEM 699, which provides an independent research experience for undergraduate students at San Francisco State University. In the summer of 2022, two high school students from the ACS SEED program also participated in the experimental module under the guidance of an undergraduate mentor.

EXPERIMENTAL OVERVIEW

Electrodeposition of Electrocatalysts

The instructor in this laboratory prepared clean indium tin oxide (ITO)-coated glass substrates (Delta Technology) in the dimensions of approximately 1.5 cm × 1.5 cm for making working electrodes (details shown in the SI). Students then fabricated working electrodes from the ITO substrates with stable electrical contacts and well-defined geometric surface areas following the experimental procedures shown in the SI. It is important to note that the dimension of the substrates is flexible and the desired geometric surface area can be defined with the insulating epoxy. In situations where time is limited, the instructor can cut the ITO substrates into 1 cm × 2 cm dimensions and directly use them as working electrodes by partially immersing them into electrolytes. Students performed cathodic electrodeposition to grow the $Ni(OH)_2$ and $Co(OH)_2$ electrocatalysts on the ITO working electrodes using 0.1 mol/L nickel nitrate ($Ni(NO_3)_2$) and cobalt nitrate ($Co(NO_3)_2$) solutions as the precursors, respectively. The electrodeposition was conducted using a two-electrode electrochemical cell in which ITO was the working electrode, and Ag/AgCl (filled with 3 M KCl) was the other electrode with a potentiostat (Figure S3). The electrodeposition was controlled with a chronopotentiometry (CP) experiment at a constant current density of −0.1 mA/cm^2 for 2 min following a previous report,[27] which usually results in a thin film of approximately 40 nm. The deposition time can be adjusted to vary the film thicknesses, and the surface morphologies of the electrodeposited catalysts are shown in Figure S4.

CV Measurements

The experimental CV analysis of the electrocatalysts was performed by the students with a three-electrode setup using a

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J. Chem. Educ. XXXX, XXX, XXX−XXX

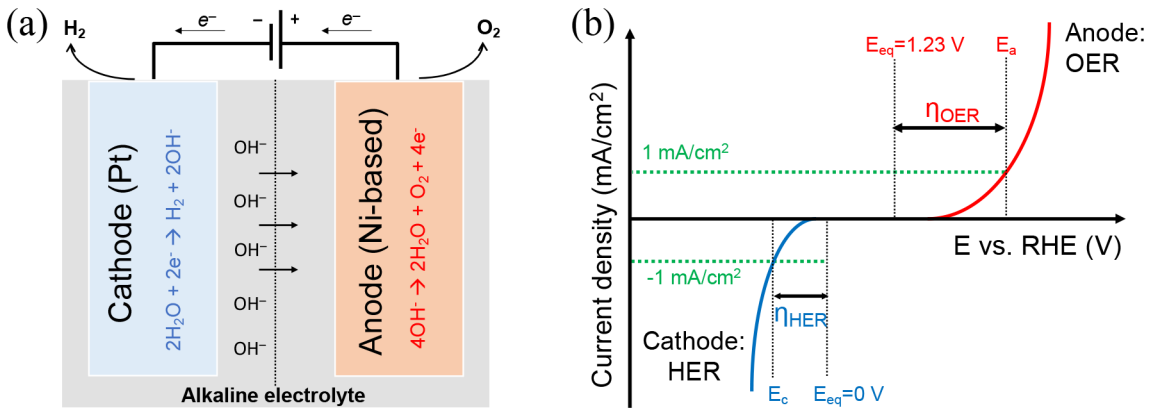

Figure 1. Schemes of (a) an AWE and (b) the current density−potential relationship for both the OER and HER in the same graph to compare the differences in the magnitude of the overpotential (η) when the magnitude of the current density is 1 mA/cm^2. The potential is referenced to the reversible hydrogen electrode (RHE) for better comparison with literature results. E_{eq} represents equilibrium potential, E_a is the applied potential on the anode, and E_c is the applied potential on the cathode. η_{OER} ($|E_a - 1.23\ V|$) is the overpotential of the OER and η_{HER} ($|E_c - 0\ V|$) is the overpotential of the HER.

potentiostat (Figure S3). The glass electrochemical cell was filled with 0.1 mol/L potassium hydroxide (KOH), and the working, counter, and reference electrodes were ITO working electrode with the electrocatalysts, a Pt wire, and a Ag/AgCl (filled with 3 M KCl) reference electrode, respectively. If Hg/HgO reference electrode is available, it is recommended to replace the Ag/AgCl to reduce the junction potential in the alkaline electrolyte.[28] To investigate the impact of scan rate on CV data, students collected the cyclic voltammograms at three different scan rates of 10, 20, and 50 mV/s, respectively. The choice of 0.1 mol/L KOH as the electrolyte aimed to minimize the usage of chemicals, but it is recommended to use 1 mol/L KOH as the electrolyte if more efficient electrocatalytic performances of the $Ni(OH)_2$ and $Co(OH)_2$ electrocatalysts are needed.

Simulation of Cyclic Voltammograms

Students were asked to run the simulation of cyclic voltammograms by using the Python codes provided by the instructor on Colab through a link without the requirements of any prior knowledge of Python. Colab is a free platform provided by Google to write and execute Python codes through the web-browser. The instructions for using Colab to run the CV simulations are included in the SI. The students adjusted the standard rate constant (k^0) of a one-step and one-electron redox reaction ($R \rightleftharpoons O + e^-$) and the scan rate ($v$) in the CV simulations.

HAZARDS

Personal protective equipment (PPE) including safety goggles, gloves, lab coats, long trousers, and closed-toed shoes is required for this lab. The KOH solution is corrosive and can produce serious skin burns. $Ni(NO_3)_2$ and $Co(NO_3)_2$ are oxidants. Hg and its salts in the Hg/HgO reference electrode are highly toxic. Methyl iso-butylketone in the conductive silver paints is a flammable liquid and a fire hazard and should be handled in the hood with caution.

RESULTS AND DISCUSSION

Students read the background knowledge of water electrolysis and electrolyzers (shown in the SI) before the laboratory work and completed the pre-lab knowledge-check questions. The instructor discussed the alkaline water electrolyzer (AWE) and the OER with students in a 20 min mini lecture before the laboratory to highlight the basic concepts of the thermodynamics, kinetics, and mass transfer of water electrolysis under alkaline conditions. The discussion centered on the two schemes shown in Figure 1 highlighting the following concepts:

- The electrolyte plays an important role in determining the half reactions that occur in water electrolysis, the selection of appropriate electrocatalysts, and the movement of ions between the two electrodes.
- While the thermodynamic potential difference required to drive water electrolysis is theoretically 1.23 V, significant overpotentials (η) are required to achieve a desired current density on both electrodes kinetically.
- In water electrolysis, the OER is responsible for the majority of the kinetic overpotential. This implies that the OER has a high activation energy, which makes it the rate-limiting half reaction, and electrocatalysts are important to lower the activation energy and thus the overpotential of this half reaction.
- In addition to charge transfer, the mass transfer of ions such as hydroxide anions (OH^-) also plays a crucial role in influencing the kinetics of electrochemical reactions.

After the fabrication of ITO working electrodes and the electrodeposition of $Ni(OH)_2$ and $Co(OH)_2$ electrocatalysts, students first collected their cyclic voltammograms in the electrolyte of 0.1 mol/L KOH at a scan rate of 10 mV/s and a bare ITO electrode was used as a control sample. The IUPAC convention was adopted in this teaching laboratory to better compare the results with recent literature on electrocatalysis. Students were asked to collect at least three cycles in each CV experiment to observe the electrochemical evolution of the electrocatalysts in terms of the redox waves.

Ni- and Co-based electrocatalysts are considered state-of-the-art OER electrocatalysts under alkaline conditions with metal doping.[27] This laboratory does not pursue the goal of achieving the best-performing electrocatalysts but instead uses these electrocatalysts as model systems to teach a set of important concepts in electrochemistry and electrocatalysis. Typical cyclic voltammograms of both $Ni(OH)_2$ and $Co(OH)_2$ catalysts are shown in Figure 2 referenced to the RHE. The conversion between the experimental Ag/AgCl reference

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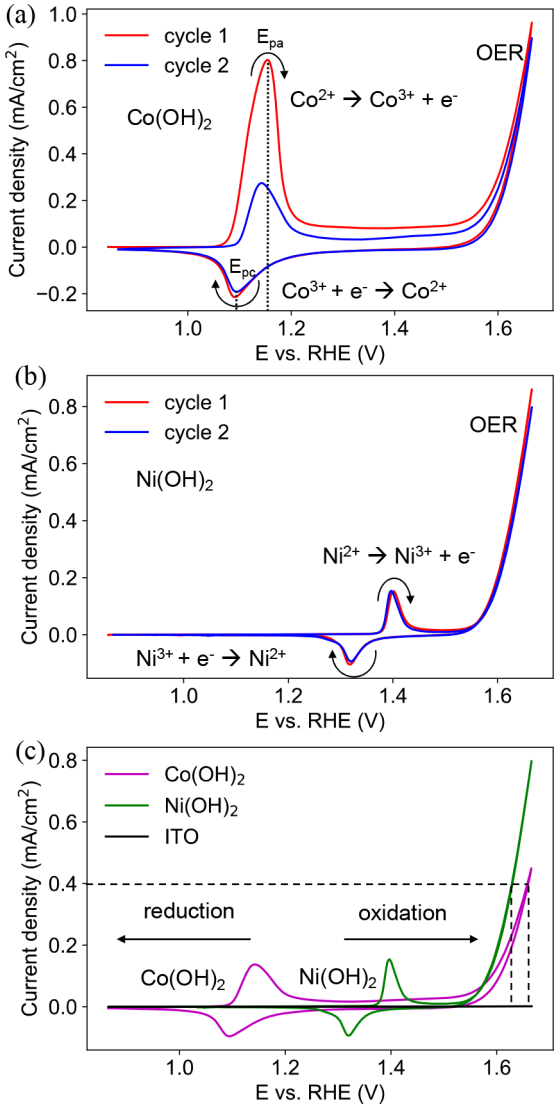

Figure 2. Typical cyclic voltammograms of (a) the $Co(OH)_2$ electrocatalyst and (b) the $Ni(OH)_2$ electrocatalyst including cycle 1 and cycle 2. (c) Comparison of the cyclic voltammograms of a bare ITO electrode (black), an ITO electrode deposited with $Co(OH)_2$ (purple), and an ITO electrode deposited with $Ni(OH)_2$ (green). The dashed lines in (c) show the different potentials of these two electrocatalysts when the same current density of 0.4 mA/cm^2 is reached. All the scan rates are 10 mV/s. The electrolyte is 0.1 mol/L KOH.

electrode and the RHE is shown in the SI. Students were able to collect comparable cyclic voltammograms for both electrocatalysts with small variations in the peak positions of the metal cation redox waves and the OER performances. Upon collecting the cyclic voltammograms, students were provided guidance on analyzing the current signals and their related electrochemical reactions (eqs 1−3) in their cyclic voltammograms. Using the first cycle of the $Co(OH)_2$ cyclic voltammogram as an example, students were able to understand the relationship between the peaks and the associated redox events. The anodic peak with a maximum potential (E_{pa}) at 1.15 V vs RHE (Figure 2b) is attributed to the Co^{2+} cations in $Co(OH)_2$ oxidized to Co^{3+} in CoOOH (eq 1) in the forward anodic scan, and the cathodic peak with a maximum potential (E_{pc}) at 1.1 V vs RHE is associated with the reduction of Co^{3+} to Co^{2+} in the reverse cathodic scan. The canonical "duck shape" of the collected cyclic voltammograms of the $Co(OH)_2$ and $Ni(OH)_2$ electrocatalysts demonstrates the reversible oxidation and reduction of the Co^{2+}/Co^{3+} and Ni^{2+}/Ni^{3+} redox couples within the solid electrocatalysts and provides evidence of their chemical reversibility. The chemical reversibility is important for the electrocatalysts to play the role of catalysis. It is noted that the first and second cycles of the cyclic voltammograms of the $Co(OH)_2$ electrocatalyst present a huge difference in the sizes of the oxidation peak, and this could be attributed to the possible charge trapping.[27] Students were then guided to integrate the anodic and cathodic peaks of the two electrocatalysts to perform a more quantitative analysis of charges (in the units of Coulomb) associated with each redox event with the software EC-Lab of the potentiostat.

Following that, students were challenged to locate the current response of the OER process (eq 3), which occurs at a potential more positive than the oxidation potentials of the electrocatalysts. The potential at which the OER current starts to become positive is often defined as the onset potential of the OER. The onset potential of the OER with the $Ni(OH)_2$ electrocatalyst (Figure 2b) is 1.56 V vs RHE, and the current signals overlap in the forward and reverse scans instead of showing a "duck shape". With the product O_2 escaping the system, the OER is chemically irreversible compared to the chemically reversible transformation of the electrocatalysts. Students were able to correlate the current responses of the two different types of reactions with the instructor's guidance. Students were then asked to compare (1) the onset potentials of the OER currents on the ITO electrodes with and without electrocatalysts, and (2) the overpotentials of the OER catalyzed by the two electrocatalysts. These comparisons provided them insights of the kinetics of the OER process in the presence of the electrocatalysts. As shown in Figure 2c, significant currents were observed beyond 1.6 V vs RHE when electrocatalysts were deposited onto the ITO electrodes. $Ni(OH)_2$ exhibited a higher catalytic efficiency for the OER compared to $Co(OH)_2$ because $Ni(OH)_2$ required a less positive potential to reach the same current density such as 0.4 mA/cm^2:

$$Co(OH)_2(s) + OH^-(aq) \rightleftharpoons CoOOH(s) + H_2O(l) + e^- \quad (1)$$

$$Ni(OH)_2(s) + OH^-(aq) \rightleftharpoons NiOOH(s) + H_2O(l) + e^- \quad (2)$$

$$4OH^-(aq) \rightarrow O_2(g) + 2H_2O(l) + 4e^- \quad (3)$$

To address the potential confusion related to the term "reversible" in electrochemistry, the concept of electrochemical reversibility was introduced and compared with chemical reversibility. Different from chemical reversibility, the electrochemical reversibility is defined as the ratio (Λ) of standard rate constant (k^0) to mass transfer ($(DFv/RT)^{1/2}$).[29] In this definition, D is the diffusion coefficients for the redox species, v is the scan rate, F is the Faraday constant, R is the ideal gas constant, and T is the temperature in the units of Kelvin. An electrochemical system in which the charge transfer interface is always at equilibrium is called an electrochemically reversible or Nernstian system.[29] A system may show reversible, quasi-reversible, or irreversible behavior depending on the value of Λ, or experimentally, the scan rate employed (more details

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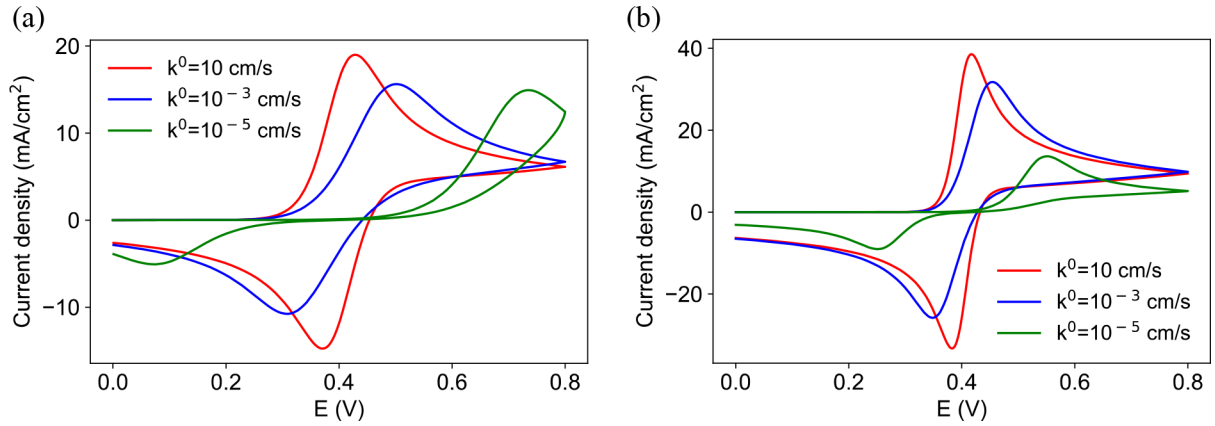

Figure 3. Simulated cyclic voltammograms with the standard rate constant k^0 varied at three different values by (a) EC-Lab software and (b) Python codes. The diffusion coefficients are 5×10^{-5} cm^2/s for both the oxidized and reduced species O and R, which is the calculated diffusion coefficient value for the OH^- anions.[30] Formal potential E^0 is 0.4 V for the redox couple, which is the average of the anodic peak and cathodic peak maximum potentials. The transfer coefficient α is 0.5, the initial concentration of R is 0.1 mol/L, and the initial concentration of O is 0 mol/L. The scan rate is 10 mV/s.

shown in the SI).[29] In general, the larger the Λ, the more electrochemically reversible is the system:

$$\Lambda = \frac{k^0}{(DFv/RT)^{1/2}} = \left(\frac{RT}{F}\right)^{1/2} \times \frac{k^0}{(Dv)^{1/2}} \quad (4)$$

From an experimental data point of view, electrochemical reversibility is reflected in the peak separation (ΔE) of the anodic peak maximum potential (E_{pa}) and the cathodic peak maximum potential (E_{pc}) in the cyclic voltammogram. The difference between the anodic and the cathodic peak potentials should be 57 mV for a one-electron redox couple that is chemically and electrochemically reversible at 25 °C.[24] Students were introduced to this background knowledge in the pre-lab reading and were asked to identify the electrochemical reversibility of the electrocatalysts based on the experimental data of peak separation ΔE. While both $Co(OH)_2$ and $Ni(OH)_2$ electrocatalysts show chemical reversibility in the cyclic voltammograms, the degree of electrochemical reversibility is different based on the values of their peak separations. The peak separation in the cyclic voltammogram of $Co(OH)_2$ is approximately 50 mV, and the peak separation in the cyclic voltammogram of $Ni(OH)_2$ is approximately 80 mV at a scan rate of 10 mV/s (Figure 2). From a microscopic point of view, electrochemical reversibility is related to both the charge- and mass-transfer kinetics of the electrochemical systems. The integration of experimental work and simulations were employed to enable students to understand the impact of these two factors on the experimentally observed cyclic voltammograms.

Students investigated the impact of charge transfer kinetics on peak separations in the cyclic voltammograms via simulations. The standard rate constant k^0 in eq 4 is a measure of the intrinsic heterogeneous kinetic facility of a redox couple and is a suitable microscopic kinetic descriptor for students to investigate in simulations. A system with a large k^0 can achieve equilibrium quickly, but a system with a small k^0 is sluggish. The one-electron redox reaction $R \rightleftharpoons O + e^-$ was applied as the simulation model to understand the one-electron redox reactions of the electrocatalysts (eqs 1 and 2) and the simulation was based on the Butler−Volmer equation assuming a linear diffusion model;[29] more details of the simulations can be found in the SI. Students ran the CV simulations with Python by varying the values of k^0 from 10 cm/s, 10^{-3} cm/s, to 10^{-5} cm/s at a scan rate of 10 mV/s, which represent fast, intermediate, and slow kinetics of charge transfer (more details on how to perform the simulations are shown in the SI). As shown in Figure 3, slower electron transfer with smaller values of k^0 leads to a lower current density and larger peak separations in the simulated cyclic voltammograms of the one-electron redox couple. The simulations run on Python were able to capture the key differences in cyclic voltammograms identical to the simulations performed with the commercial software EC-Lab. This simulation practice provided students the correlation between the microscopic charge transfer kinetics and the experimentally observed cyclic voltammograms and insights on the slow kinetics of the multistep and multielectron OER which has a small value of k^0.

In addition to investigating the charge transfer kinetics, the role of mass transfer in electrochemical reversibility was introduced with scan-rate-dependent CV measurements and simulations followed by a 20 min mini lecture. The solid-state $Co(OH)_2$ and $Ni(OH)_2$ electrocatalysts allow ions such as OH^- to permeate and participate in the redox reactions of the electrocatalysts and subsequently the OER. The OH^- ions need to diffuse from the bulk solution to the electrode surface and the concentration gradient of OH^- near the electrode surface in the diffusion layer is the key concept to help students understand the mass transfer in CV experiments. The diffusion coefficient of OH^- ions ($D = 5 \times 10^{-5}$ cm^2/s)[30] was used as the diffusion coefficients of both R and O in the simulations. The scan rate (v) of the CV experiments controls how fast the applied potential is scanned and the size of the diffusion layer (more explanations are included in the SI). At small scan rate (or long times), systems may yield reversible waves, while at large scan rate (or short times), irreversible behavior could be observed.[29] This is because large scan rates lead to a decrease in the size of the diffusion layer, redox species reach the surface of the electrode more quickly and higher currents are thus observed.[24]

Students adjusted the scan rates from 10 to 20 and 50 mV/s to perform CV measurements of their electrocatalysts and CV simulations of the one-electron redox couple. The majority of the students observed a consistent trend of scan-rate-

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J. Chem. Educ. XXXX, XXX, XXX−XXX

dependent currents in both the experimental and simulated cyclic voltammograms, identical to the typical data shown in Figure 4. However, one group of students experienced an inconsistent trend and was able to validate their experimental data with the simulated one. The inconsistency they observed could be attributed to the instability of their electrode. After students collected the scan-rate-dependent data and validated the trend of scan-rate-dependent current with simulations, the instructor presented a mini lecture focusing on the conceptual understanding of the diffusion layer, and the concentration gradient of OH^- ions at different scan rates with the materials from a developed activity.[20] The impacts of both charge transfer and mass transfer on the cyclic voltammograms and the electrochemical reversibility are also summarized in this mini lecture. The discussion materials are shown in the SI. In the post-lab data analysis, the students were asked to perform a more quantitative analysis of the anodic/cathodic peak maxima of their electrocatalysts with the scan rates in their cyclic voltammograms and explain how scan rates determine their experimentally observed currents.

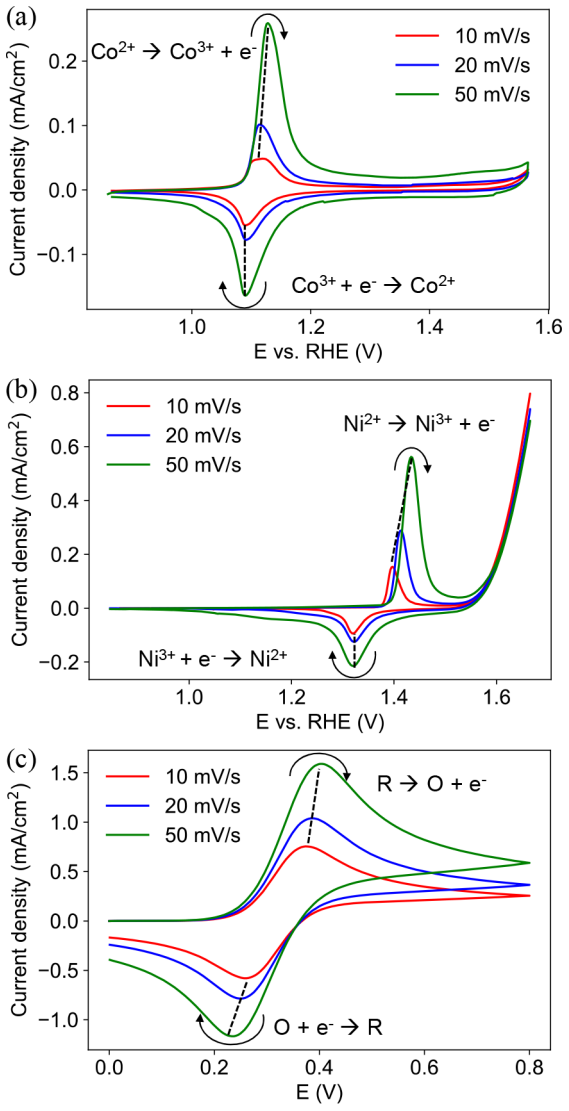

Figure 4. Typical cyclic voltammograms of (a) the $Co(OH)_2$ electrocatalyst and (b) the $Ni(OH)_2$ electrocatalyst on ITO electrodes in 0.1 mol/L KOH with three different scan rates of 10, 20, and 50 mV/s. (c) Python-simulated cyclic voltammograms with different scan rates. The diffusion coefficients are 5×10^{-5} cm^2/s for both the oxidized and reduced species, O and R. Formal potential E^0 of the redox couple is 0.4 V. The transfer coefficient α is 0.5, the initial concentration of R is 0.1 mol/L and the initial concentration of O is 0 mol/L. The standard rate constant k^0 is 10^{-3} cm/s.

Taken together, the experimental and simulation work are anticipated to provide students a comprehensive understanding of how the charge transfer and mass transfer affect the electrochemical reversibility (eq 4) by investigating the changes of k^0 and v on the cyclic voltammograms and better interpretation of the electrochemical properties of the electrocatalysts and the OER measured by the CV.

STUDENT LEARNING

This laboratory experience focuses on conceptual understanding of the role of electrocatalysts in boosting the efficiency of the OER, the electrochemical transformations of the electrocatalysts, and the associated charge and mass transfer processes. While this experiment module has been conducted in both teaching and research laboratory courses, the analysis of student learning primarily focuses on the teaching laboratory course CHEM 426. Student learning outcomes were assessed via the pre-lab questions, in-lab discussions, and post-lab data analysis. To help students better analyze and interpret the data, the instructor also provided help after the laboratory.

Students demonstrated a strong understanding of the half reactions in water electrolysis and the role of electrocatalysts and the ability to correlate the currents in the cyclic voltammograms to different types of electrochemical reactions studied in this laboratory. They were able to distinguish the concepts of chemical reversibility and electrochemical reversibility using the redox reactions of the $Ni(OH)_2$/$Co(OH)_2$ electrocatalysts and the OER as examples. Students' understanding of the microscopic processes related to charge and mass transfer was weak in the spring 2022 semester. The simulation practice and the mini lectures added in the spring 2023 semester enhanced student learning of these concepts.

Students highly valued the ability to make electrodes and electrodeposit catalysts to drive water oxidation and gain hands-on experience with electrochemical experiments. Additionally, students appreciated the opportunity to discuss research articles centered on water electrolysis in their presentations. This served as a platform for students to enhance their understanding of data interpretation in electrocatalysis, learn state-of-the-art water electrolyzer technologies, and learn related career opportunities in the field of renewable energy.

CONCLUSIONS

As was demonstrated in these experiments, the combination of experimental CV measurements and Python-based simulations along with mini lectures is a useful tool for students to understand the electrochemical reactions of electrocatalysts for the OER. This laboratory experience allows the students to understand the concepts of chemical reversibility, electrochemical reversibility, and charge- and mass-transfer kinetics in electrochemistry. Because the electrocatalysts are facile to prepare via electrodeposition, this experiment is suitable for undergraduate students as an introduction to heterogeneous electrocatalysis. This experiment could also be adopted in

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J. Chem. Educ. XXXX, XXX, XXX−XXX

course-based undergraduate research experiences (CUREs) in which students explore the dependence of the electrocatalyst compositions on their catalytic performance and investigate the more quantitative aspects of electrocatalysis.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.3c00176.

Notes for instructors; detailed experimental procedures; pre-lab and post-lab questions (PDF, DOCX)
Python codes for simulation (PDF, DOCX)
Python codes for exporting data (PDF, DOCX)

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Notes

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J. Chem. Educ. XXXX, XXX, XXX−XXX